



‘Paula’s Papers’: Salts in Porous Construction and Building Materials

Paper 3 – Salts Analysis: Diagnosis, Assessment and Treatments

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1. HOW CAN WE MEASURE THE TYPE AND AMOUNT OF SALTS?

1.1. Salt diagnosis in buildings

Samples of salts can be collected from buildings and be analysed with simple tools to obtain a useful rough general assessment. These samples can also be analysed in the lab to get precise results by means of more accurate analytical techniques.

When the source of the salts of an affected building is not clear, analyses of tap water, building materials, and the soil under or around the building is a useful procedure for understanding the origin of the problem. The lack of, or failure by bridging of a damp-proof course (DPC), can give a source of salts during rising damp. This might bring the consequent mixture of damp moisture within building materials producing hygroscopic salts seen as damp patches. Therefore, is important to know if the rising damp is active or if we just have hygroscopic salt residues from the past. The moisture content should be higher and increasing with depth at the base of the wall compared to higher heights with more superficial moisture.

We know that electrical moisture meters can provide misleading results on moisture equivalent measurements (WME%, in materials others than wood) by increasing the moisture level readings when salts are present. Recent studies have shown that resistivity-type meters (i.e. measuring conductivity with pins) seem to be more affected by NaCl salt. Whereas capacitance meters (radio frequency or 'search mode' over flat surfaces) appears more sensitive to variations in moisture. For the diagnosis of rising damp there are some old traditional known ways to evaluate this, such as the gravimetric analysis methodology developed by the UK British Research Establishment (BRE DG245).

Although salt content identification is not the objective of the measurements, the hygroscopic moisture content (HMC) is a simple laboratory technique that can be used to assess the sources of moisture from soluble salts in building materials. It evaluates to what extent the actual moisture content (MC) of collected samples could be due to hygroscopicity rather than to water coming from the ground i.e. 'true' rising damp. The method consists of extracting samples by drilling the material while collecting the powder and, after drying, subjecting the samples to a determined relative humidity (RH) level until reaching a constant weight. Water soluble salts have a characteristic hygroscopic behaviour and adsorb moisture from the air above their equilibrium RH (RHeq). If there are salts present in the samples, an increase of weight is registered, which depends on the type and amount of salt.

When collecting samples or building materials affected by salts (e.g. efflorescence, crust, flakes or powder of decayed materials) is necessary, preserving them in well-sealed containers to prevent phase changes and hydration-dehydration reactions is important. If the source of salts is the original composition of the building materials, to identify which ones are supplying the soluble salts is essential before deciding if removing and substituting those materials is the best way to proceed (**Figure 1**).



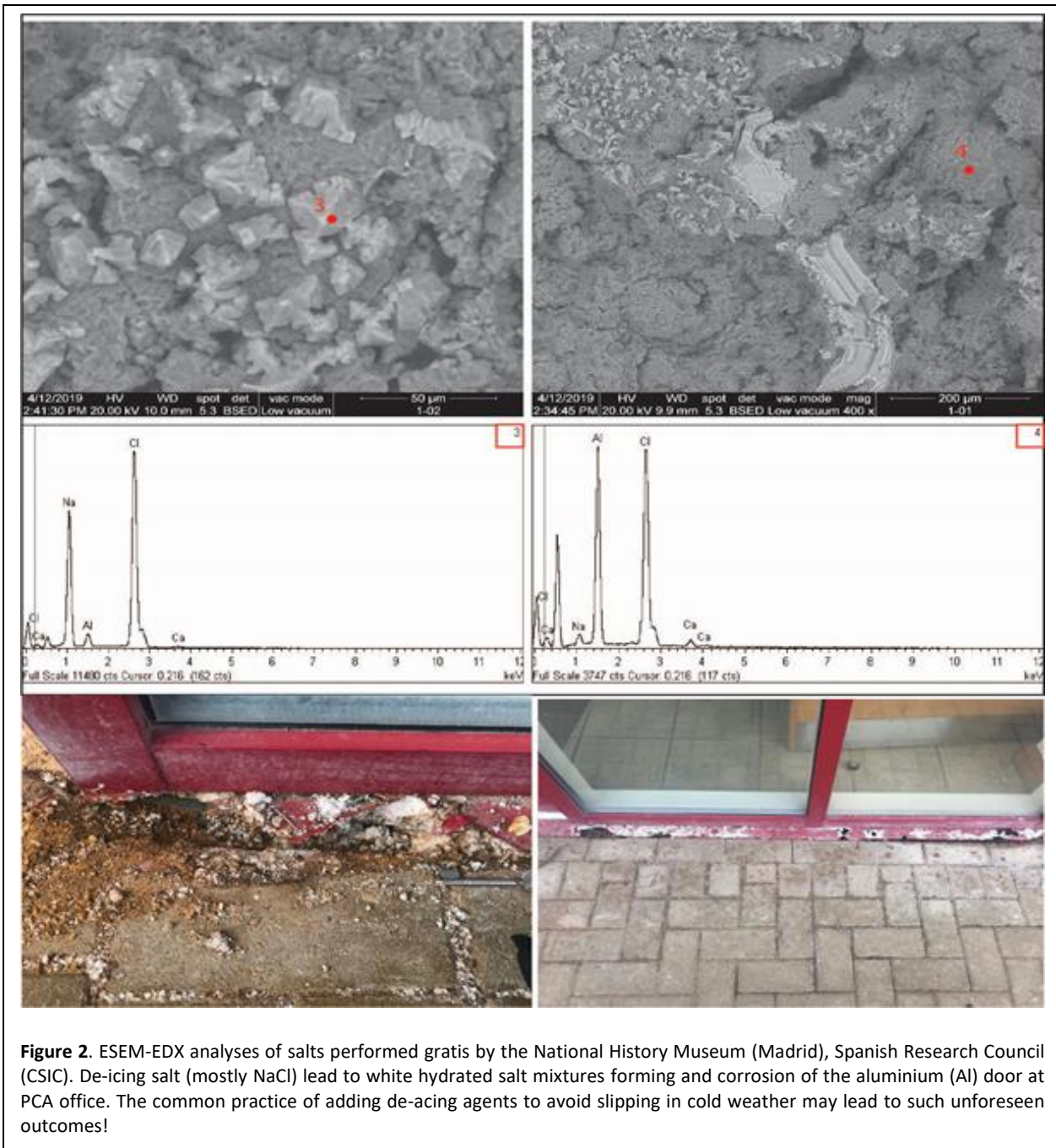
Figure 1. Holy Ghost church (Exmouth) built in 1912. *Bath stone* (a Jurassic oolitic limestone from SW England) was used surrounding windows and it is suffering very rapid decay. It is known that this English limestone, is not resistant to salt weathering. Its pore properties allow more moisture movement to take place inside the rock, being prone therefore to faster salt migration and more rapid degradation.

1.2. Salt analysis: On site and in lab

Salt analysis supports the identification of the source of damaging salts in a building, which is essential in order to find the best solution to solve it.

Analysis of salt efflorescence on historic buildings can be performed on site by means of portable and non-destructive analytical techniques. These high-tech tools have been mainly used for research purposes in order to get a high level of detail on the type of salts present on historic buildings or valuable artefacts such as historic paintings. Micro-Raman or X-Ray fluorescence analysis allow us to determine the chemical composition of many salts. Portable X-Ray diffraction analysis is also possible to determine mineral species with their corresponding state of hydration. All these techniques are also available in the lab, together with others used for more detailed analysis of salts, such as Environmental scanning electron microscopy coupled with Energy dispersive x-ray analysis, ESEM-EDX (**Figure 2**).

Ion chromatography (IC) provides very precise quantitative and qualitative analysis. The amount and type of ions (cations and anions) present in collected drilled samples from walls at different heights and depths can be accurately determined. This is also a common precise technique to analyse poultices used for desalination for assessing the efficacy of the salt extraction. However, conductivity measurements can be performed to assess the reduction of salts from affected walls after each poultice application. This method allows semi-quantitative measurement of the amount of water-soluble salt present in samples. It reduces the costs of the salt extraction procedure and assessment, by placing a known amount of sample in deionised water and recording the electrical conductivity of the suspension. Other qualitative measurements performed onsite enable us to identify the type of soluble salts using salt strip tests. These allow the presence of chlorides, sulphates and nitrates to be determined while some kits are also able to perform quantitative measurements of these salts in the field.



2. DURABILITY TESTS: SIMULATION OF SALT DAMAGE IN THE LABORATORY

Accelerated aging cycles are trials where severe or extreme environments (e.g. heat, moisture, sunlight, pollution, etc.) can be applied to simulate decay, or to accelerate the regular processes of weathering. The durability of materials can be assessed in this way over a longer term. The changes produced in the petrophysical properties of stone materials can be also evaluated before, during, and after the application of preservative, consolidating or water repellents products for choosing the most appropriate one for each specific type of substrate.

This allows us to assess whether or not the applied treatments are suitable in terms of compatibility and durability as a function of slowing or acceleration of decay. These tests also allow an assessment of treatment costs, economics, time, difficulty of application, etc.

There are numerous types of tests, most of them standardised, where the appropriate selection of one or another test depends on the extrinsic agents related to the environment and surroundings where the materials under study or treatment are or must be located and placed. Therefore, performing a laboratory test will provide a way to assess decay and durability of materials and products or methods to avoid salt crystallisation. Also, this is a way for testing new methods in the lab and to evaluate the efficacy of treatments prior to applying them in the field, reducing risks of possible failures.

Standard tests on stone and related materials are established to perform reproducible tests, being able to provide good quality data, following a unified methodology, and obtaining comparable results. The standardization activity has a goal to develop techniques that will be willingly used, as a reference to improve the quality and safety of any technological activity, science, or service. Salt damage to building materials can be simulated in the laboratory through different accelerated ageing tests. We mentioned in previous papers that the type and amount of salt ingress will condition their location in the materials, whereas the solubility of the salts will control their height and depth inside the material.

2.1. Factors to consider when performing salt crystallisation tests

It is important to consider some issues when performing an accelerated ageing test in the lab:

- Simulate the context where the building materials are located or will be settled as a function of the original or potential source of salts (i.e. marine spray, polluted atmosphere, rising damp, etc.).
- Depending on the source of the exposure environment select how the input of salts will be applied (e.g. spray, absorption, etc.).
- Calculate the concentration of the solution according to the standards or real analysis.
- Register the environmental conditions of the lab, or control these in climatic chambers or enclosed containers with supersaturated salt solutions. These will control the evaporation rates, the dissolution-crystallisation cycles and eventually the final decay.

2.2. Specimens subjected to salt solution absorption

There are standard tests, such as the British-adopted European Standard BS EN12370:1999 (Natural stone test methods. Determination of resistance to salt crystallisation) to perform salt crystallisation cycles to accelerate stone decay (recently updated to BS EN12370:2020). The test is performed using sodium sulphate salt due to its highly deteriorating action. Salt solution immersion followed by drying cycles in the stove are applied to stone specimens. Other non-standardised variations to this salt crystallisation test are performed by capillary absorption, sealing with wax the air-salt solution interface where long stone specimens are submerged (**Figure 3**).

As we mentioned before, saturation degree will condition the aggressivity of the salt and will depend on the concentration and the environmental conditions. Evaporation will cause the saturation of the salt solution and this will depend on the temperature and the relative humidity (RH) of the environment and the chemical composition of the solution. Therefore, is very important to register and control all environmental conditions in the lab for a better knowledge of the behaviour of materials affected by salts.

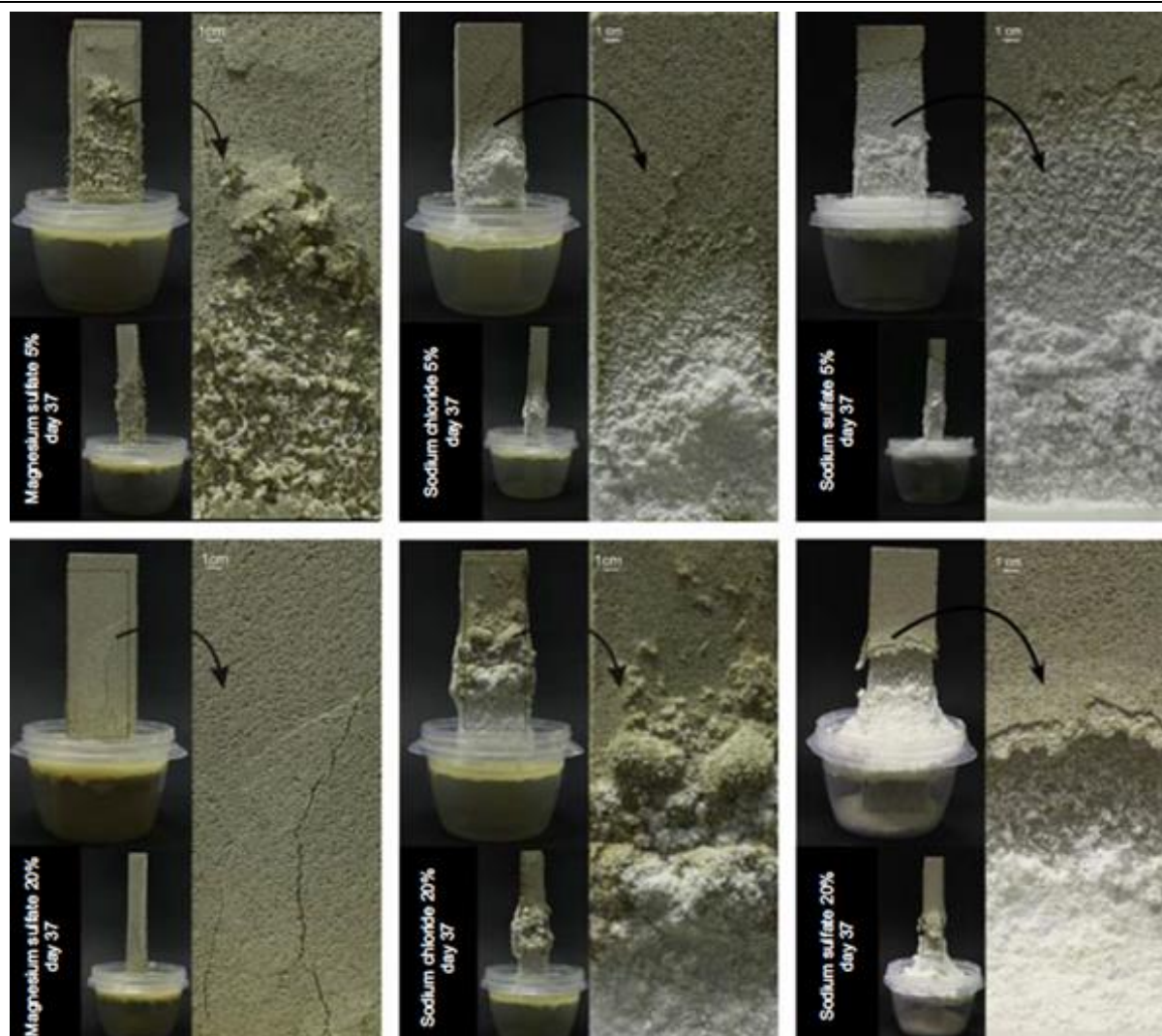


Figure 3. Result of saline solution absorption of *Bateig stone* specimens (from Alicante, Eastern Spain), typically used in historical and present-day buildings. Magnesium sulphate, sodium chloride, and sodium sulphate (5% and 20% concentration) after 37 days (with evaporation prevented using wax at solution:air interface).

2.3. Specimens subjected to salt solutions by salt mist

There are other accelerated tests involving cyclic exposure to salt mist, dry and wet conditions to simulate salt decay in paints and varnishes or corrosion of metals and alloys, such as BS EN ISO 11997-1:2017 and BS ISO 14993:2018, respectively.

Another standard test related to salt crystallisation of stone materials by the action of marine spray is the saline fog test. This can be performed in a continuous mode (e.g., ASTM B117 (2016); DIN 50021 (1988); UNE-EN 60068-2-11) or by means of cycles where the stone samples are exposed under a sodium chloride saline solution together with drying cycles (e.g. BS EN 14147:2003) (**Figure 4 and Figure 5**).



Figure 4. Marine aerosol (sodium chloride) accelerated aging test. (a) Salt Spray Corrosion Test Chamber; (b) Drying process without ventilation; (c) Drying process accelerated by ventilation (fan).

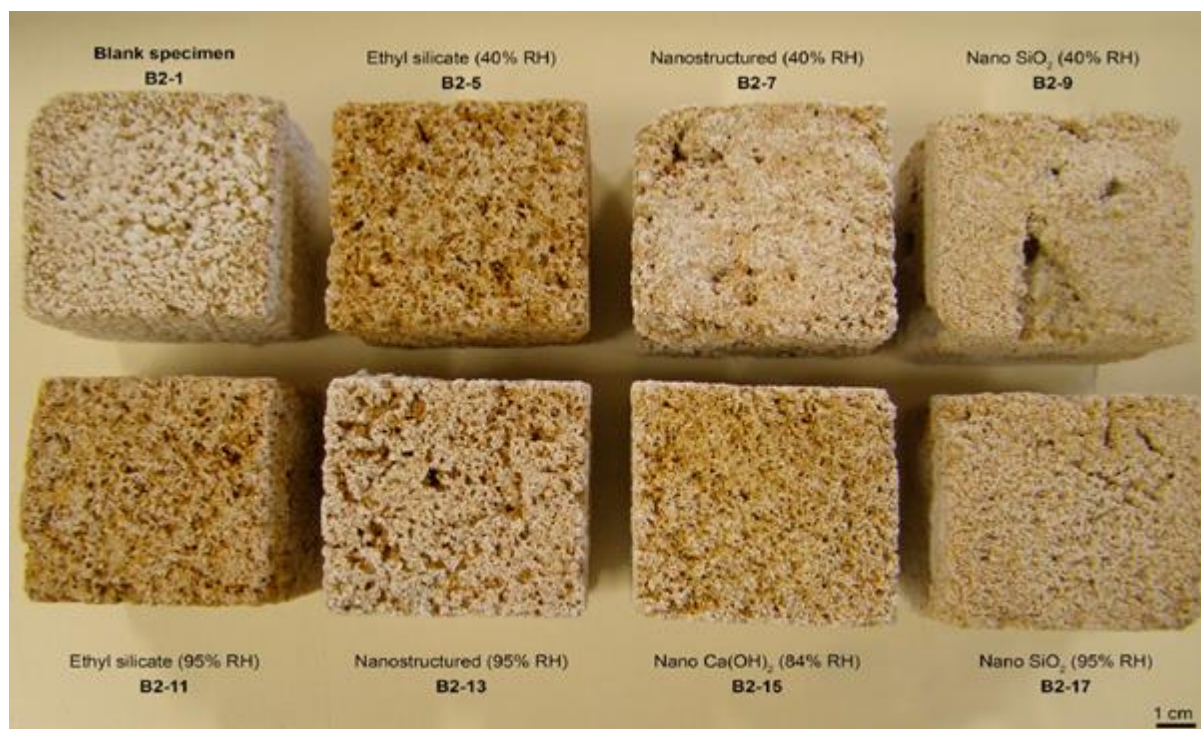


Figure 5. Calcarene stone cubic specimens after treatment with different consolidating products exposed to different RH (dry (40% RH) and very humid (84–95% RH) environments) after marine aerosol aging test and before cleaning with water.

There is also an acidic fog test which can simulate the resistance of materials under polluted atmospheres. Climatic chambers with humid and SO₂ atmospheres can be used to perform the test (DIN 50018 (2013) or alternatively by means of H₂SO₃ solution immersion and drying cycles (BS EN 13919:2002) or with nitric acid to simulate a NO_x atmosphere.

3. TYPES OF TREATMENTS AND MEASURES TO AVOID SALT CRYSTALLISATION

The first step to find the most effective solution for salt damaged materials is knowing the source of salts. Then the next step should be to cut or reduce as much as possible the input of water ingress without generating unintended side effects (e.g. restricting normal evaporation pathways). Complementary techniques can also be used for the extraction of accumulated salts from the substrates using different desalination procedures. A stone being fed constantly with moisture from the soil, will constantly accumulate salts at the point of evaporation. So, the first obvious action to take is avoiding rising damp. For highly valuable architectural heritage conservation, the application of poultices can provide a complementary measure to extract the salts from the substrate, once the input of salts has been stopped. Poultices might be useful if these are applied properly, considering different aspects regarding the substrate, source, input and location of salts.

If a conservation treatment, such as consolidating or damp proofing product is applied to salt-containing masonry, the distribution of the salts can be significantly affected. Salts accumulated at or behind waterproofing areas may be problematic. Sub-efflorescence can be developed at the interface between treated and untreated stone leading to subsequent decay.

3.1. *Inhibitors or modifiers of salt crystallisation*

No ultimate solution exists to limit salt crystallisation damage, unless by removing either the salt or the moisture. Inhibiting nucleation, or the modification of the shape and size of salt crystals, can be done by using trace amounts of chemicals commonly used to inhibit mineral scaling on pipes in industrial applications. In some ways it is a risky strategy and the application of inhibitors (e.g. sodium-ferrocyanide, NaFeC) for the prevention of salt decay in porous material is still controversial. If nucleation is inhibited or postponed, this could lead to even higher levels of supersaturation, and damage might be even more severe.

Research studies have shown that the effect of inhibitors strongly depend on the type of substrate, which also depend on their pore size distribution and composition (e.g. NaFeC, when applied on NaCl contaminated limestone and brick was able to enhance drying and favouring efflorescence, whereas the effect on other stones was very limited. The effect on salt crystallization damage was not evident in this case).

3.2. *Poulticing and sacrificial mortars*

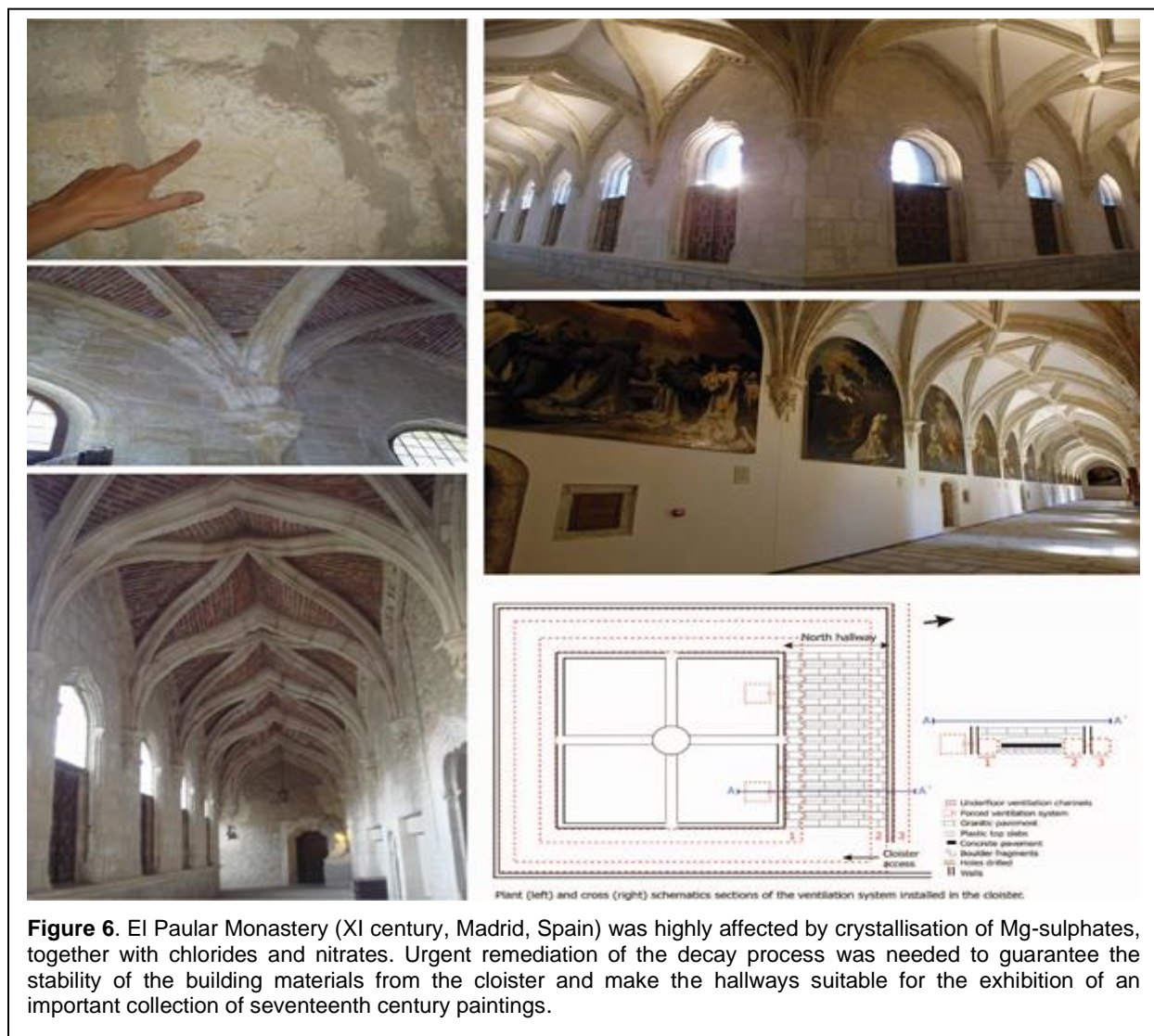
There has been a lot of research on cellulose poultices and mixtures with clays used for desalination, extraction or reduction of salts from affected walls. To be effective, the cellulose fibres should be adapted to the type of substrate (which has different pore size distribution, water capillarity absorption rates, etc.). There are cellulose fibres with different lengths and specific gravities for being adjusted to different materials (e.g. stone, brick, concrete, etc.) with different porosities. This can also be supported by using some additives such as absorbent clays (e.g. sepiolite), etc.

As mentioned before, poulticing is a complementary method to be used once the input of salts has been stopped, being applied as the last cleaning step. If the input of salts is finite (e.g. salts located on the first millimetres from the surface (e.g. from a source of past atmospheric pollution deposition) the poultice extraction might be used as a single method implementing the necessary number of applications. Ion chromatography analyses are usually carried out to assess the performance of the salt extraction and the efficacy of the poulticing method.

Using a simpler tool as a conductivity meter (to measure the conductivity of soluble salts after each poultice application) could be a satisfactory method for the assessment on site, which would really reduce the final costs. It is also important to note that poultices must be impregnated in water before being attached to the wall, so some salts could be dissolved and reintroduced deeper inside some substrates. Sacrificial mortars, render or repointing mortars, are sometimes used as a temporary porous media to allow salts to crystallise in them instead of in the more valuable permanent adjacent materials. Once these mortars are loaded with salts these can be removed and new sacrificial mortars can be applied again for receiving more accumulations of salts.

3.3. Preventative conservation

Preventive conservation aims to keep objects or structures in an environmental state where minimum damage occurs. It includes direct actions undertaken to avoid natural and anthropic decay agents, in addition to improving the environmental conditions. The environmental control is easy to apply to protect objects located in museums. However, achieving this in an outdoor environment is difficult unless the affected area can be confined.



The preventive conservation strategies applied to El Paular Monastery (**Figure 6**) included architectural solutions (e.g. ventilation system to avoid high humidity levels and hence dissolution, mobilisation and crystallisation of salts), petrophysical-based solutions (i.e. exploiting the porosity differences between building materials and poultices to maximise salt reduction) and study the physicochemical behaviour of salts and RH transfer through the stone (to determine the most suitable environmental conditions to prevent crystallisation of the most harmful salt species).

This research represents both a practical and experimental exercise that is useful for conservation scientists and restorers involved in the field of preservation of historic or valuable buildings, and for environmental control to avoid salt crystallisation.

For further information, contact:

Property Care Association

11 Ramsay Court
Kingfisher Way
Hinchingsbrooke Business Park
Huntingdon
Cambs.
PE29 6FY
Tel: Tel: 01480 400000
Email: pca@property-care.org
Web: www.property-care.org

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