

Salt extraction by poulticing in the archaeological site of Coudenberg

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Abstract

A common method for the extraction of salts is the use of poultice materials. Despite extensive scientific research, the salt extraction process in practice often remains empiric. This paper aims to further the understanding of a salt extraction by poulticing in practical experiments. Three types of poultice materials are tested on brick masonry in 12 different locations in the subterranean archaeological site of Coudenberg in Brussels. The tested poultice materials have different compositions including kaolin clay, sand and/or cellulose fibres, theoretically favouring advection and/or diffusion. It is shown that for an accurate interpretation of the results it is crucial to evaluate the salt content in the poultice and to a depth of at least 20 cm in the different materials of the substrate, while considering the different densities and surface area of each material. Furthermore, it is shown that the presence of gypsum overshadows the results, hence the exclusion of equimolar contents of Ca^{2+} and SO_4^{2-} is essential for the evaluation. Finally, the results of these experiments reveal that regardless of the different procedures, such as, the poultice type, pre-wetting or application time, salts are being redistributed into the wall rather than extracted.

Keywords: salt extraction, desalination, poultice, historic masonry, archaeological site

1. Introduction

The archaeological site of Coudenberg, the last material evidence of the former Palace of Brussels, is situated at the heart of Belgium's capital underneath the Royal Square and its 19th century buildings. The project is the culmination of twenty-five years of archaeological work, which have progressively enabled different areas of the site to become accessible to the public. Not all the materials of the site have been preserved in the same condition and the project faces considerable challenges in both conservation and presentation considering the relatively recent exposure from burial.¹ Major water infiltrations and salt damage are now visible in a large part of the site.

The presence of salts in the subterranean site is essentially caused by water and ion transport that occurred over an extended period of time. During this time salts accumulated unevenly at the evaporation front, near the surface at certain heights and preferentially in the materials with the smallest pores. Damage phenomena are naturally explained by the accumulation of salts and the occurrence of crystallisation cycles. These cycles are promoted by changes in relative humidity (RH). Daily RH changes typically influence the first centimetres and diminish rapidly in depth of the substrate, with crystallisation cycles occurring less frequently further in the substrate from daily, weekly, monthly to seasonally. The amount of cycles that influence the first centimetres can easily run up to more than a 100 cycles per year.² Hence, visible

salt damage is seen at certain heights or in specific regions, with mainly the first centimetre of a substrate being affected. Lowering the salt content near the surface and diminishing daily changes in RH both in intensity and recurrence will mitigate salt damage, contributing to a sustainable preservation and an appropriate presentation for visitors.

While the removal of salts from movable stone objects by means of the water bath method is quite well known, in-situ salt extraction of brick masonry by means of poultices is often carried out on the basis of general assumptions, and in most cases without control of the efficiency or the effect in the depth of the substrate. This paper goes deeper into the practice of salt extraction on brick masonry using three different poultice types, focusing on the influence of some of the execution parameters on the salt extraction efficiency.

2. Materials and methods

2.1. Sampling, salt analysis and efficiency evaluation

Before and after each poultice application samples are taken from the mortar and brick at different depths. Samples are obtained by powder drilling ($\varnothing 8$ mm) in intermediate steps, 0-1, 1-3, 3-5, 5-10, 10-15 and 15-20 cm, to obtain an in-depth moisture and salt profile. For each sample, the actual and the hygroscopic moisture contents are determined gravimetrically. The quantity of anions (Cl^- , NO_3^- , and SO_4^{2-}) and cations (Na^+ , K^+ , Ca^{2+} , and Mg^{2+}) of the filtered aqueous extract is measured by ion chromatography (IC, Metrohm). The theoretical amount of carbonates (CO_3^{2-}) is determined from the excess of analysed cations. The results are presented as $\text{kg}\cdot\text{m}^{-2}$ (per sampled depth), based on the specific density (drilled powder) of the dry mortar/brick.

The density, the porosity and the pore size distribution of the dry poultice materials, brick and mortar are determined by mercury intrusion porosimetry (MIP, Autopore IV, Micromeritics). From the results the theoretical properties of the poultice, that is, the advective or diffusive properties are determined.

The evaluation of the poultice efficiency and the salt extraction/migration processes is based on a comparison of the evolution of the salt content in the depth of the masonry and in the poultice, before and after salt extraction treatment. The total amount of ions, including or excluding equimolar amounts of Ca^{2+} and SO_4^{2-} (reflecting the gypsum content), in $\text{kg}\cdot\text{m}^{-2}$ (per sample depth) are considered during the evaluation. The salt content at a certain sampling depth in the masonry (brick and mortar) is determined as follows:

$$\varphi_{\text{brick}} \cdot x_{\text{brick}} \cdot d \cdot A_{\text{brick}} + \varphi_{\text{mortar}} \cdot x_{\text{mortar}} \cdot d \cdot A_{\text{mortar}} \quad (1)$$

With φ_{brick} and φ_{mortar} , respectively the mass density (specific density) of the brick and mortar (in $\text{kg}\cdot\text{m}^{-3}$), x_{brick} and x_{mortar} , respectively the total content of soluble ions in each material, excluding equimolar amounts of Ca^{2+} and SO_4^{2-} (in wt%), d , the sampling depth (in meter) and A_{brick} and A_{mortar} , the proportional surface area (in m^2/m^2 wall surface) of respectively brick and mortar. The total amount of salts in the sampled volume is obtained by adding the amount of salts found at every sampling depth for both brick and mortar. The result is expressed as the amount of salts per unit of surface area ($\text{kg}\cdot\text{m}^{-2}$). The comparison of the salt content in the substrate after treatment with the initial salt content indicates the amount of salts which have disappeared out of the sampled volume and which either have been absorbed by the poultice or either migrated away within the substrate to a greater depth or outside of

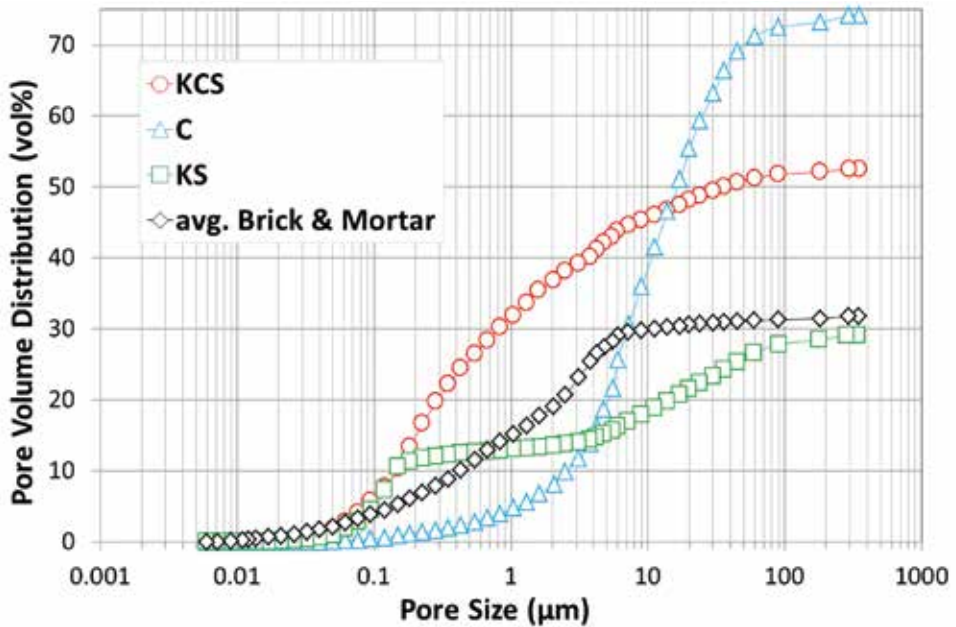


Figure 1: Cumulative pore volume distribution (vol%) of the dried poultice materials KCS (○), C (Δ) and KS (◻) compared to the avg. pore volume distribution of the brick and mortar from 7 locations (◇).

the sampled area. As only the salts absorbed by the poultice are effectively extracted, the amount of salts disappeared out of the sampled volume is compared to the amount of salts detected within the poultice ($\text{kg}\cdot\text{m}^2$), determined as follows:

$$\varphi_{\text{poultice}} \cdot x_{\text{poultice}} \cdot t \quad (\text{II})$$

With $\varphi_{\text{poultice}}$ the mass density (bulk density) of the poultice (in $\text{kg}\cdot\text{m}^3$), x_{poultice} the total content of soluble ions in the poultice, excluding equimolar amounts of Ca^{2+} and SO_4^{2-} (in wt%) and t the total thickness of the poultice (0.01 m). The efficiency of the salt extraction process is determined as the amount of salts that have been absorbed by the poultice in comparison to the initial salt content in the masonry, expressed in %.

2.2. Poultice materials and application procedures

The following poultice materials have been tested:

Poultice KCS (Saltpull type 1, Rewah) is a poultice based on the findings described by Lubelli & van Hees^{3,4} and was selected for the purpose of this project as it has enough small pores compared to the substrate to theoretically favour an advective flow towards the poultice (Figure 1). The poultice contains cellulose fibres (Arbocel PZ8 1.4 mm), kaolin clay (IMERYS quality China Clay Speswhite) and calibrated sand (Sibelco CEN196 sand fraction 0.5-1 mm) (1:2:1 by weight). The poultice is mixed with water (61 wt% H₂O for 39 wt% of dry poultice material) and contains 0.2% biocide.

Poultice C (Cellulose Saltpull, Rewah) is a commonly and traditionally used poultice in the restoration practice and was selected as it has enough large pores compared to the substrate to theoretic-

cally favour a diffusive flow towards the poultice (Figure 1). The poultice contains cellulose fibres (Arbocel PZ8 1,4 mm). The poultice is mixed with water (78 wt% H₂O for 22 wt% of dry poultice material) and contains 0,2% biocide.

Poultice KS is based on a poultice described and recommended by Bourgès & Vergès Belmin.^{5,6} This poultice has enough small and large pores compared to the substrate to theoretically favour both an advective and a diffusive flow towards the poultice (Figure 1). The poultice contains kaolin clay (IMERYS quality China Clay Speswhite) and calibrated sand (Sibelco CEN196 sand fraction 0,5-1 mm), 1:5 by weight (0,8:1 by volume). The poultice is mixed with water (20 wt% H₂O for 80 wt% of dry poultice material).

The three different poultices (KCS, C and KS) are tested on brick masonry in a total of 12 different locations (1-12). Locations 1, 6, 10 and 11 are free standing walls suffering from rising damp. Locations 2, 3, 7 and 8 are located on vertical earth retaining walls, while locations 4, 5, 9 and 12 are situated on vaults that are presumably in contact with earth. The main salts, excluding gypsum, found within the walls are identified as sodium chloride (NaCl) in locations 1, 2, 4, 5, 6, 8, 9, 10 and 12, together with calcium nitrate (Ca(NO₃)₂) in locations 4, 5, 8 and 9. Sodium sulfate (NaSO₄) is found in locations 3, 7 and 11, presumably in combination with smaller amounts of sodium carbonate (Na₂CO₃), as theoretically derived from the excess of sodium ions in these locations.

Poultices KCS, C and KS are applied to the salt contaminated masonry in respectively 5 (1-5), 4 (6-9) and 3 locations (10-12), and removed after respectively 11, 19 and 47 days. Before each poultice application samples are taken from the substrate (as described earlier). Before application of poultices KCS and C the

masonry is pre-wetted with a known amount (0,5 2 or 4 l·m⁻²) of demineralized water by spraying. Pre-wetting is carried out with intermediate steps to prevent runoff and allow maximal absorption. In the case of the application of poultice KS, the masonry is not pre-wetted. All poultices (KCS, C and KS) are applied to the masonry with a spatula in a thickness of approximately 1 cm on a surface of approximately 1 m². Poultice C is immediately covered with a water/vapour impermeable foil after application. The different poultice application conditions are based on experiences and traditions found in literature and discussions with practitioners. At the end of the experiments samples are taken from the poultice material and from the substrate as described earlier. For the purpose of this paper the results of the experiments are averaged per poultice type.

3. Results

In total 3 poultice types were tested in 12 locations with the results of each experiment showing considerable variations, making the interpretation rather challenging. However, several important results can be described looking at the individual test results (not shown) and at the averaged results per poultice type. Although tests were carried out with different amounts of water for pre-wetting the surface, no significant influence could be derived. Exceptionally, an irregular migration of salts was recorded to unknown locations in the substrate when pre-wetting the surface. Similar exceptions were derived when the substrate showed high moisture contents before the experiments; water added during the pre-wetting procedure or from the poultice itself provoked in such a case an oversaturation of the bedding mortar which allowed water and salts to migrate from the mortar towards the brick. Whe-

re the moisture content was lower before the experiments this phenomenon was naturally seen reversed as the mortar in all locations has smaller pores than the brick, and hence tends to absorb rather than expel its pore water (and salts).

3.1. Poultrice KCS

The average results of the five experiments with poultrice KCS are shown in figure 2. The amount of salts, excluding gypsum (CaSO_4), that have disappeared from the masonry (0-20 cm) after the poultrice application is 4% of the total salt content before application, whereas in the case the presence of gypsum is included an increase of the salt content with 14% is observed. The salt content, either excluding or including gypsum, that has migrated towards the poultrice amounts to only 6 and 4%, respectively, of the total salt content before application. Hen-

ce, as compared to the total amount of salts in the substrate, the poultrice treatment is not so efficient, certainly in areas where salts might migrate back to the surface. When considering the first cm of the substrate (0-1 cm) the results are easily misinterpreted as an efficiency of 54 and 35% is obtained. When looking at the results, excluding gypsum, a general decrease of the salt content in the wall is seen up to a depth of 10 cm. Some of these salts will have migrated to the poultrice, however, the rest most probably diffused into the depth of the substrate as an increase of the salt content is observed from 15 cm onwards.

3.2. Poultrice C

The average results of the four experiments with poultrice C are shown in figure 3. The amount of salts, either excluding or including gypsum, that has disappe-

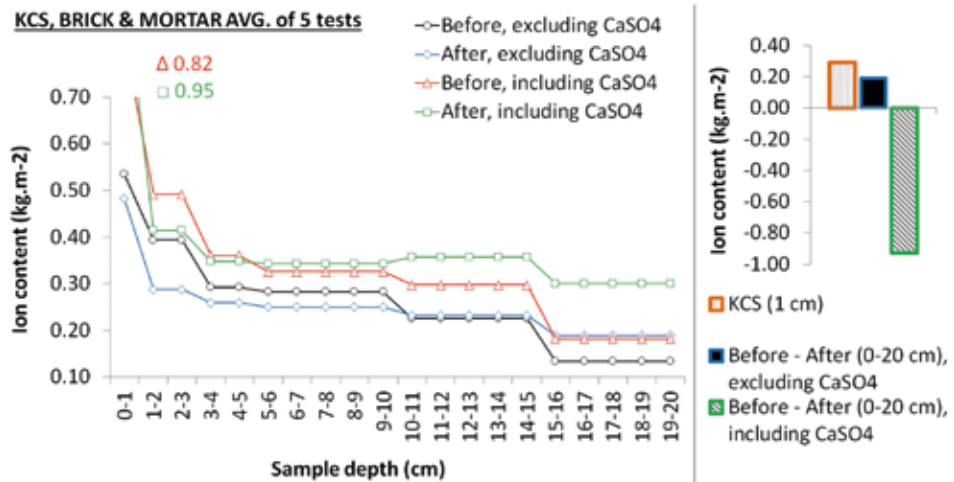


Figure 2: Poultrice KCS, avg. of 5 locations (1-5). Left: Results of the avg. ion concentration ($\text{kg}\cdot\text{m}^{-2}$, per cm), detected in the masonry, while taking the respective surface areas of the brick and mortar into account: before (○, black line) and after (◇, blue line) excl. CaSO_4 , and again before (△, red line) and after (□, green line) incl. CaSO_4 . The vertical bars on the right show respectively the avg. amount of salts deposited in the poultrice (dotted and orange column, $\text{kg}\cdot\text{m}^{-2}$) and the avg. amount of salts, excl. CaSO_4 that have disappeared from the substrate (full and blue column, $\text{kg}\cdot\text{m}^{-2}$, total of sampled depth 0-20 cm) and idem, incl. CaSO_4 (striped and green column). For the latter the negative value indicates an average increase of the salt content over the sampled volume.

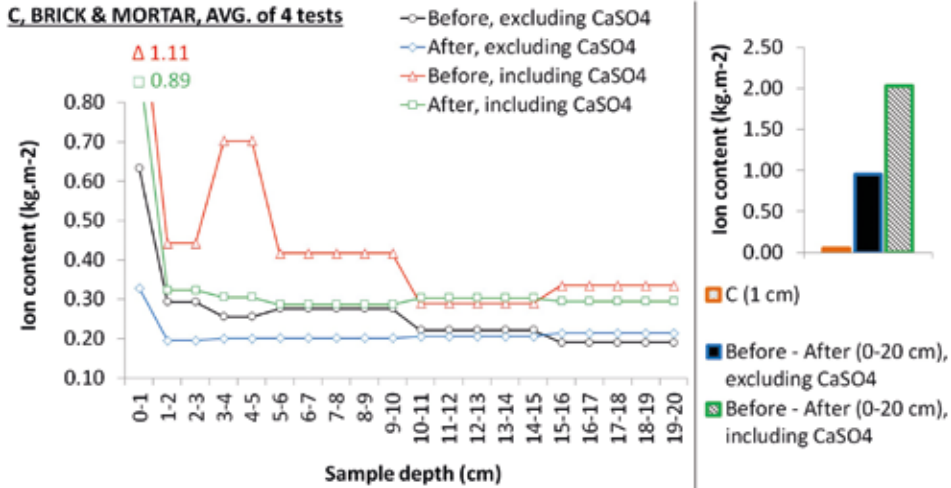


Figure 3: Poultice C, avg. of 4 locations (6-9). Left: Results of the avg. ion concentration ($\text{kg}\cdot\text{m}^{-2}$, per cm), detected in the masonry, while taking the respective surface areas of the brick and mortar into account: before (○, black line) and after (◇, blue line) excl. CaSO_4 , and again before (△, red line) and after (□, green line) incl. CaSO_4 . The vertical bars on the right show respectively the avg. amount of salts deposited in the poultice (dotted and orange outlined column, $\text{kg}\cdot\text{m}^{-2}$, total thickness of the poultice: 1 cm) and the avg. amount of salts, excl. CaSO_4 , that have disappeared from the substrate (full and blue outlined column, $\text{kg}\cdot\text{m}^{-2}$, total of sampled depth 0-20 cm) and idem, incl. CaSO_4 (striped and green outlined column, $\text{kg}\cdot\text{m}^{-2}$, total of sampled depth 0-20 cm).

ared from the masonry (0-20 cm) after the poultice application is respectively 18 and 24%, while the amount of salts that migrated towards the poultice is in both cases only 1% of the total salt content before application. When considering the first cm of the substrate (0-1 cm) the results would indicate an efficiency of 8 and 5%. Although the efficiency concerning the extraction is slightly lower compared to poultice KCS, the salt content in the substrate, excluding gypsum, has decreased significantly further from the surface up to a depth of 15 cm. While practically none of these salts have migrated to the poultice, approximately 17% of them have disappeared, and therefore most likely migrated to lesser salt contaminated areas and into the depth (beyond 20 cm) of the substrate.

3.3. Poultice KS

The average results of the three experiments with poultice KS are shown

in figure 4. The amount of salts, either excluding or including gypsum, that has disappeared from the masonry (0-20 cm) after the poultice application is respectively 14 and 27%, while the amount of salts that migrated towards the poultice is respectively only 4 and 3% of the total salt content before application. Taking into account the first cm of the substrate (0-1 cm) the calculated efficiency is instead 73 and 25%. A general decrease of the salt content, excluding gypsum, is seen up to a depth of 20 cm of which only a small amount have migrated to the poultice.

4. Conclusions

The results and interpretation of the extraction process show important differences when considering the gypsum content and different sampling depths in the substrate. An inaccurate interpretation is highly possible when including gypsum in the calculations as it is often

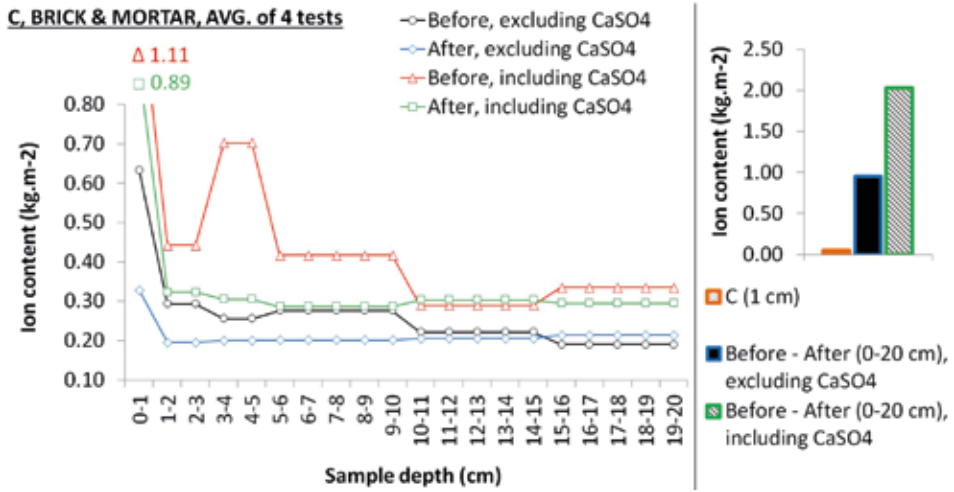


Figure 4: Poultrice KS, avg. of 3 locations (10-12). Left: Results of the avg. ion concentration ($\text{kg}\cdot\text{m}^{-2}$, per cm), detected in the masonry, while taking the respective surface areas of the brick and mortar into account: before (○, black line) and after (○, blue line) excl. CaSO₄, and again before (△, red line) and after (□, green line) incl. CaSO₄. The vertical bars on the right show respectively the avg. amount of salts deposited in the poultrice (dotted and orange column, $\text{kg}\cdot\text{m}^{-2}$) and the avg. amount of salts, excl. CaSO₄, that have disappeared from the substrate (full and blue column, $\text{kg}\cdot\text{m}^{-2}$, total of sampled depth 0-20 cm) and idem, incl. CaSO₄ (striped and green column).

unevenly distributed in the substrate and generally occurs in much higher concentrations as compared to other salts. As shown in the results these aspects, in combination with its low solubility ($\sim 2.0\text{--}2.5$ g/l at 25 °C), overshadow the overall extraction process. When comparing the salt content in the poultrice to the total salt content in the substrate (from 0-20 cm depth), the average results of the poultrice applications indicate a rather low efficiency for the tested poultrices, KCS, C and KS, with respectively an efficiency of only 6, 1 and 4% of salts extracted. When considering only the first centimetre of the substrate, extraction efficiency values of 54, 8 and 73% are obtained, which evidence the ease of misinterpretation of the salt extraction efficiency. Although the extraction of salts at the surface is of utmost importance, as it is the area mainly influenced by daily RH cycles and, hence, most likely subjected to severe damage, the significant influence of the poultricing proce-

sure on the migration processes further into the depth of the substrate forces an evaluation of the salt content up to greater depths. A general rule applies in that salts tend to migrate to further depths and to areas with lower salt contents.

The overall reduction of the salt content in the substrate to a depth of 20 cm is 4 and 14% for the poultrices KCS and KS (favouring advection). Although the extraction of salts seems the least effective for poultrice C, favouring diffusion, it is clear that the salt content in the sampled depth to 20 cm has decreased with 18%. Although hardly any salts are extracted, the results of this poultrice show that the salt content is sufficiently low to prevent further salt damage at the surface, at least for the time being.

Surprisingly poultrice C is the preferred poultrice in this case, the decision is essentially based on practical concerns and its repeatability. As long as the subterranean site is subject to water infiltrations it can be assumed that any treatment

will have a limited lifespan. Besides the ease of use of poultice C and a significant difference in cost concerning the raw materials, both poultices KCS and KS leave a white haze behind on the substrate, caused by the kaolin clay, which raises concerns regarding the aesthetic changes and loss of material during the subsequent cleaning of the surface, increasingly so when considering a repeated treatment.

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