

**RISK ASSESSMENT AND CONSERVATION STRATEGY OF A SALT LADEN
LIMESTONE MAUSOLEUM AND THE SURROUNDING FUNERAL CHAPEL
IN BOUSSU, BELGIUM.**

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Abstract

Salt deterioration of stone in Cultural Heritage is a universal and complex phenomenon that is not yet fully understood. A lot of research has been done on the behaviour of salts in porous material. These studies generally involve single salts, for which the deliquescence points are well documented. However, in situ one generally finds complex ion mixtures. In such conditions the crystallisation behaviour can change dramatically, making the conservation strategy much more intricate. Therefore, the assessment of the critical environmental conditions to minimise salt damage requires knowledge of phase transition thermodynamics of salt crystallisation. This paper describes the results of an investigation on the deterioration process caused by the crystallisation of salt mixtures detected in the building materials of the 12th century Gothic funeral chapel of the Lords of Boussu in the community of Boussu, Belgium (*La chapelle funéraire des Seigneurs de Boussu*), as well as in the Avender limestone (*Avendersteen– Pierre d’Avesnes*) of the 16th century Renaissance Mausoleum of *Maximilien de Hénnin-Liétard*, situated in the chapel. Since 1970 the chapel and its mausoleums are listed as exceptional heritage of the Walloon Region. The thermodynamic behaviour of the salt mixtures is modelled with the ECOS/RUNSALT software (Price 2000; Bionda 2002), capable of predicting the crystallisation behaviour of salt mixtures in function of temperature and relative humidity. The software requires data of quantitative salt analyses as input and allows to assess favourable climatic conditions to minimise the number of phase transitions and hence mitigate damage due to salt crystallisation. The salt crystallisation sequences are compared to the climate conditions in the chapel, monitored over a period of 12 months, to diagnose the visible damage. Appropriate restoration treatments and potential preventive conservation measures are presented.

Keywords: Salt Decay, ECOS, RUNSALT, Climate Monitoring, Conservation, Restoration, Heritage, Preservation

1. Introduction

The construction of the funeral chapel of the Lords of Boussu (*La chapelle funéraire des Seigneurs de Boussu*) started in the 12th century. The chapel is built with natural stone and brick, and the walls inside are (re)plastered. The floor consists of black and red Belgian marble tiles on a sand bed. Underneath a section of the floor a double storey crypt was built and sealed with a stone slab and a copper plate. To the southern and northern side of the chapel a brickwork balcony is present. The formerly freestanding chapel incorporates three 16th and 17th century mausoleums and became part of the Church of St. Géry in the 16th century. This paper relates to the walls of the

chapel and the mausoleum of the family of *Maximilien de Hénin-Liétard* positioned against the north wall (Figure 1).



Figure 1. (left). Mausoleum of the family of *Maximilien de Hénin-Liétard*. ©KIK-IRPA
Figure 2. (right). Detail of the deteriorating limestone and one caryatid. ©KIK-IRPA

The mausoleum is approximately 317 cm high and 300 cm wide with four life sized sculptures in the middle. A stone slab is covering the tomb of the mausoleum and is supported by three caryatids with Ionic capitals, one of which is carrying a child, the other a falcon, and the third an unidentified object. This research involves the architectural elements of the mausoleum, excluding the painted limestone sculptures situated on the tomb. The mausoleum is constructed in a fine grained and chalky limestone: Avender stone or *Pierre d'Avesnes-le-sec*. This particular stone was mainly used for sculptures and architectural ornamentation in the Southern Netherlands from the 15th to the 17th century. Avender stone hasn't been quarried for more than a century and has become an important historical stone (Dusar et al. 2009). The surface is finished with polychrome layers imitating black and red Belgian marble and white alabaster or marble and is also partly gilded.

An investigation was carried out to evaluate the moisture and the salt load in the walls of the chapel and in the limestone of the mausoleum combined with monitoring the climate conditions in the chapel. The scientific method and the results are presented and different conservation methods are proposed, which have contributed to formulate a risk mitigation strategy for the preservation of the limestone and building materials of the walls. During the condition assessment it was presumed that rising damp is present, which is causing salt efflorescence, moisture stains and damage to the plaster on the walls of the chapel and material loss of the limestone at the bottom of the Mausoleum (figure 2).

When assessing the deterioration of porous building materials, contaminated with salt mixtures, it is important to understand the thermodynamics of phase transitions. Opposed to single salts it is considerably more intricate to define the deliquescence humidity (RH_{del}) of salts in case of a mixture, which is explained in detail by Price (1994) and Steiger (1996, 2005). An inventory of the types of anions and cations in more than a 1000 samples taken from Belgian historic buildings and sites proved that building materials rarely contain one particular type of salt, but rather a complex mixture of ions (De Clercq 2010). Mainly the formation of double salts makes the prediction of the behaviour of salts in a mixture complex (De Clercq 2008; Larsen 2006). To identify the deliquescence points and thereby the required climate conditions to prevent phase transitions, the thermodynamic computer program Environmental Control of Salts (ECOS/RUNSALT) (Price 2000; Bionda 2002) is used. The model requires data of quantitative salt analyses as input and the output enables the user to determine relative humidity (RH) and temperature (T) ranges in which phase transitions occur and identify states of equilibrium. Phase transitions are identified as the crystallisation-dissolution or hydration points of salts. Outputs of the ECOS/RUNSALT program are compared to the actual climate conditions monitored during one year in the chapel.

2. Sampling

The sample locations in the mausoleum and the chapel are identified with Roman numerals (see fig. 3 and table 1). Samples were lifted by powder drilling at the base of the mausoleum (location I), where the stone is chalking, salt crystallisation is visible, and material loss is perceived. Samples are also lifted from the walls within the chapel (locations II-V), in the plaster, brick, and mortar, where moisture stains, discoloration, and damage is visible. At each location a small hole (\varnothing 5 mm) is drilled in three intermediate steps (0 to 1 cm, 1 to 3 cm and 3 to 5 cm) to determine the in-depth salt profile. In total 15 samples were lifted from five different locations in the limestone mausoleum and 21 samples from seven different locations in the walls. The moisture content, hygroscopicity, and salt content are determined for each sample. Two efflorescence samples are lifted from the plaster on the walls and subjected to X-Ray Diffraction analysis (BRUKER D8 theta/2theta configuration).

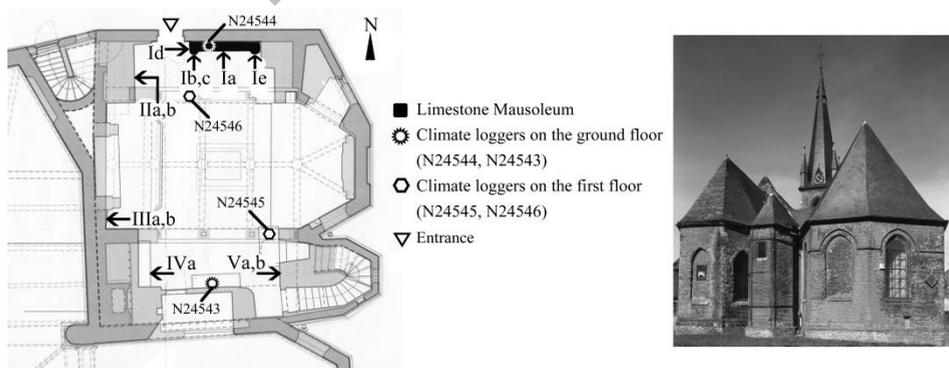


Figure 3. Left: Floor plan with locations of the lifted samples and position of the data loggers for climate monitoring. Modified from the original plans by architects S. Wautier & J-L Vanden

Eynde. Right: View on the east façade of the chapel. The chapel (right) is since the 16th century part of the Church of St. Géry (far left and back). ©KIK-IRPA

- Location I includes five different areas or heights (Ia, Ib, Ic, Id, and Ie) where samples are lifted from the limestone mausoleum.
- Location II includes two different heights (IIa and IIb) where samples are lifted in the plaster on the West wall near the entrance of the chapel.
- Location III includes two heights (IIIa and IIIb) where samples are lifted in the plaster and the brick of the West wall.
- Location IV includes one area (IVa) where samples are lifted in the plaster and the brick from a pedestal.
- Location V includes two heights (Va and Vb) where samples are lifted in the wall to the right of another pedestal.

3. Scientific method

3.1 Moisture content and hygroscopicity

The 38 samples are individually and hermetically sealed in pre-weighed glass containers and transported to the laboratory. The actual moisture content (AMC) is determined gravimetrically by weighing the samples at 60°C until a constant weight is reached. Then the samples are conditioned at 20°C and 95% RH to determine the hygroscopic moisture content (HMC). The results are compared to the weight of the dry samples and presented as weight percentage (wt%).

3.2 Salt content-crystallisation sequence

The salt content is determined by adding approximately 1 g of the dried sample to 100 ml demineralised and deionised water, which is mixed to achieve the extraction of salts. The quantity of anions (Cl^- , NO_3^- and SO_4^{2-}) and cations (Na^+ , K^+ , Ca^{2+} and Mg^{2+}) of the well-mixed and filtered extract is analysed with Ion-Chromatography (IC, Metrohm, the column for anions used is METROSEP A Supp 5 150/4.0 and the column for cations used is METROSEP C4-150/4.0). The results in parts per million (ppm) are converted into milli-equivalents and presented as wt%. If deemed necessary the data, expressed as millimole (mmol) is entered into the ECOS/RUNSALT thermodynamic model. Gypsum is eliminated from the mixture and the salt crystallisation sequence is calculated at 12°C, being the average T recorded in the chapel, and at a RH between 15 and 95%. Conditions below 25% RH are improbable and are not taken into consideration. The results of the modelled crystallisation behaviour of the salt mixtures are compared to the climate conditions recorded in the chapel.

3.3 Climate monitoring-modelled salt crystallisation sequence

The climate was monitored during one year starting on March 9, 2010 with four data loggers (Madgetech RHTemp 1000SS) recording T and RH every 15 minutes (fig. 3). The results are related to the salt contamination and the corresponding crystallisation sequence of the outer centimetre of the investigated materials, modelled with the ECOS/RUNSALT program. The outer centimetre is mainly affected when environmental conditions change. The number of phase transformations detected is used

to assess potential salt damage and determined by counting the times the average daily RH crossed the critical deliquescence points (Grossi 2011).

4. Results

4.1 Moisture content and hygroscopicity

The results of the actual and the hygroscopic moisture content of the most relevant samples are presented in table 1. Samples containing at least 5 wt% are marked in grey. During the condition assessment it was presumed that rising damp is present. Contrary to this presumption the results contain relatively low ambient moisture contents, which can be due to a period of drought before sampling.

Actual moisture contents above 5 wt% are detected in the locations IIa, Va, and Vb and can be attributed to the presence of hygroscopic salts. All of the investigated samples show a high hygroscopic moisture content, which is more pronounced near the surface (the drying front), where the salts have accumulated over time. The situation is critical in locations Va and Vb where the hygroscopic moisture content of the plaster exceeds 30 wt%.

Table 1. Sample overview with actual moisture content (AMC) and hygroscopic moisture content (HMC) (wt%). The sample locations are presented in figure 3.

Sample location	Height (cm)	Support	Sample code	Depth (cm)	AMC (wt%)	HMC (wt%)
Ia	5	Limestone	X001	0–1	3.1	12.6
Ib	15	Limestone	X004	0–1	2.5	7.9
Ic	33	Limestone	X007	0–1	3.2	12.1
Id	50	Limestone	X010	0–1	2.5	10.9
Ie	18	Limestone	X013	0–1	2.4	6.9
IIa	17	White plaster	X016	0–1	5.3	22.0
		Grey plaster	X017	1–3	4.2	4.6
IIb	55	White plaster	X019	0–1	2.9	14.0
		Beige plaster	X020	1–3	0.8	7.0
IIIa	20	White plaster	X022	0–1	4.2	23.4
		Brick	X023	1–3	0.9	3.0
IIIb	55	White plaster	X025	0–1	1.5	10.7
		Brick	X026	1–3	0.5	1.1
IV	70	Brick	X029	0–1	2.1	13.1
Va	15	White plaster	X033	0–1	7.6	46.1
		Brick	X034	1–3	1.4	6.8
Vb	35	White plaster	X036	0–1	5.0	31.6
		Brick	X037	1–3	2.2	11.5

4.2 Salt content

The ion content (expressed as wt%) of the most relevant samples lifted at a depth of a few centimetres is presented in table 2. The overall results show a decrease of the salt load in the depth of the material. Magnesium was not detected.

Table 2. Ion content (expressed as w%) of the most relevant samples lifted at a depth of a few

centimetres. The sample depth is presented in table 1.

Sample location	Height (cm)	Sample code	Cl ⁻	NO ₃ ⁻	SO ₄ ²⁻	Na ⁺	K ⁺	Ca ²⁺	Total ion content
Ia	5	X001	0.1	1.0	2.3	0.1	0.1	1.3	5.0
Ib	15	X004	0.1	0.6	3.6	0.0	0.0	1.7	6.1
Ic	33	X007	0.2	0.9	1.0	0.1	0.1	0.7	3.0
Id	50	X010	0.1	0.9	0.3	0.1	0.1	0.3	1.8
Ie	18	X013	0.1	0.5	1.8	0.1	0.1	0.9	3.4
IIa	17	X016	0.2	1.9	6.4	0.1	0.2	3.1	11.9
		X017	0.0	0.2	9.0	0.0	0.1	3.7	13.1
IIb	55	X019	0.1	1.2	0.2	0.1	0.2	0.3	2.1
		X018	0.1	0.5	0.1	0.1	0.1	0.1	1.0
IIIa	20	X022	0.0	1.0	1.4	0.2	0.5	0.6	3.8
		X023	0.0	0.2	1.5	0.1	0.1	0.6	2.5
IIIb	55	X025	0.0	0.5	2.7	0.1	0.1	1.2	4.5
		X026	0.0	0.1	0.2	0.0	0.0	0.1	0.5
IV	70	X029	0.1	1.1	0.7	0.2	0.3	0.3	2.6
Va	15	X033	0.3	2.0	0.9	1.4	1.1	0.1	5.8
		X034	0.0	0.4	0.1	0.2	0.2	0.1	1.1
Vb	35	X036	0.2	2.8	1.1	0.4	0.7	0.7	6.0
		X037	0.1	0.9	0.1	0.2	0.3	0.2	1.8

The average salt load in the first centimetre in the limestone mausoleum consists of up to 2.8 wt% gypsum, 0.8 wt% nitrates, and 0.2 wt% sodium chloride. There is a significant decrease of gypsum and nitrates in the depth (figure 4).

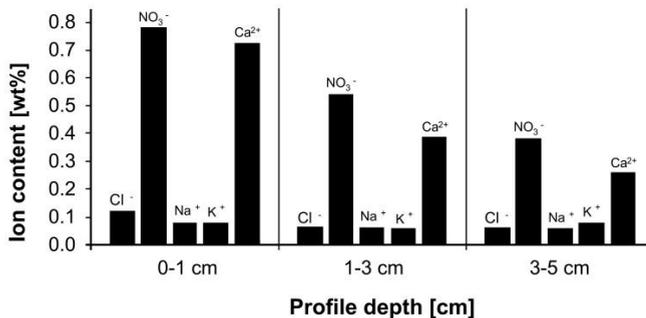


Figure 4. Average wt% of the main ions detected in the samples lifted in the limestone of the mausoleum, after correction for gypsum.

The salt crystallisation sequences, obtained by the ECOS/RUNSALT model, are quite similar for every sample lifted from the limestone in the mausoleum. As an example, figure 5 illustrates the crystallisation sequence of sample X007 lifted from the limestone in location Ic. The limestone is contaminated with a mixture of gypsum, sodium chloride, and nitrates, the latter mainly linked to calcium. Their hygroscopic properties explain the high values of HMC (table 1). The average amount of nitrates in

the first centimetre in location I exceeds 0.7 wt% and approaches 2 wt% in some samples.

Crystallisation mainly occurs when the RH drops below 35% while smaller amounts of potassium nitrate, sodium nitrate, and sodium chloride crystallise below 60%.

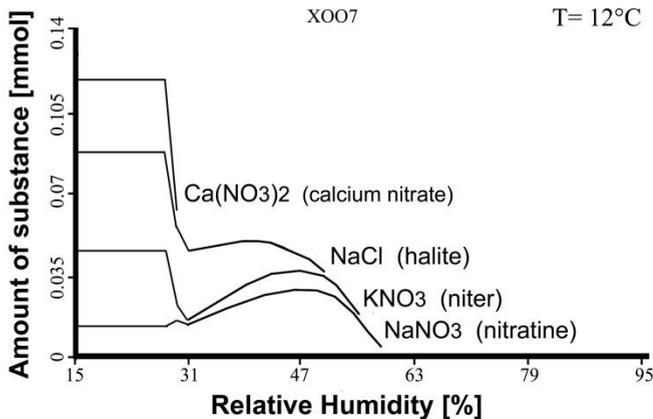


Figure 5. Crystallisation sequence of the detected ion mixture of the aqueous extract of sample X007, after correction for gypsum. (ECOS/RUNSALT modified for publication).

The hygroscopic moisture content measured in the white plaster at both heights in location II indicates the presence of hygroscopic salts. The results of the ion content confirm these results and reveal up to 2 wt% nitrates. The salt content is similar to that of the limestone in location I. The ECOS/RUNSALT outputs predict crystallisation to occur when the RH drops below 30%. The underlying mortar contains lower amounts of nitrates (about 0.5 wt%). The presence of gypsum in the white plaster and the underlying mortar in location II at a height of 17 cm is explained by the underlying cement mortar, which seems to be a recent repair.

The overall salt content in the white plaster in location III is lower than in location II, while their HMC is similar. The ECOS/RUNSALT outputs indicate that crystallisation of potassium and sodium nitrate will occur at a RH between 55 and 90%. At a height of 20 cm, the underlying brick is contaminated with a small amount of sodium sulphate and up to 3 wt% of gypsum.

The brick pedestal in location IV has a finish composed out of a very thin white plaster. The plaster is not analysed because it is a recent repair that will be removed during future restorations works. The brick support is characterised by high HMCs. The salt content in the first centimetre is above 1.5 wt% and decreases with depth. Nitrates are mainly present as nitratine (NaNO₃) and niter (KNO₃) and are also present in the efflorescence removed from the plaster. The ECOS/RUNSALT output indicates that crystallisation of nitrates will occur between 55 and 85% RH.

The plaster on the wall in location V is visibly deteriorated showing loss of material and is characterised by HMCs up to 46 wt%. The quantitative ion results indicate a

complex mixture of salts, with total contents of up to 5 wt%, that are particularly destructive. In the aqueous extract of the sample lifted at a height of 15 cm an ionic imbalance is noticed. An imbalance in favour of cations might indicate the presence of carbonates; in this case sodium carbonate that can occur at different hydrated crystalline forms. Unfortunately the salt crystallisation behaviour of carbonates is not taken into consideration in the ECOS/RUNSALT program. The salt crystallisation sequences of the remaining ion mixture of the aqueous extract revealed at 12°C a crystallisation of mirabilite ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$), aphthitalite ($\text{Na}_2\text{SO}_4 \cdot 3\text{K}_2\text{SO}_4$), darapskite ($\text{Na}_2\text{SO}_4 \cdot \text{NaNO}_3 \cdot \text{H}_2\text{O}$), niter (KNO_3), and halite (NaCl) between 60 and 93% RH. The efflorescence mainly consists of aphthitalite. The salt contamination of the samples lifted at a height of 35 cm is comparable with the one detected in location IV. The ECOS/RUNSALT output indicates that crystallisation of nitrates will occur between 55 and 90% RH, while that of calcium nitrate occurs when the RH drops below 30%, as noticed in locations I and II.

4.3 Climate monitoring

Four data loggers in the chapel (fig. 3) recorded the temperature and the RH over a period of one year. The climate behind the four statues in the mausoleum (datalogger N24544) is illustrated in fig. 6. An important yearly variation in temperature is observed, reaching extremes at 22.4°C in the early summer and 2.4°C in mid winter. The yearly average temperature is 12.0°C. Although the daily variations in temperature are small, the RH varies frequently between 59.0% and 95.5%. Daily variations in RH exceed often 8% within 24 hours.

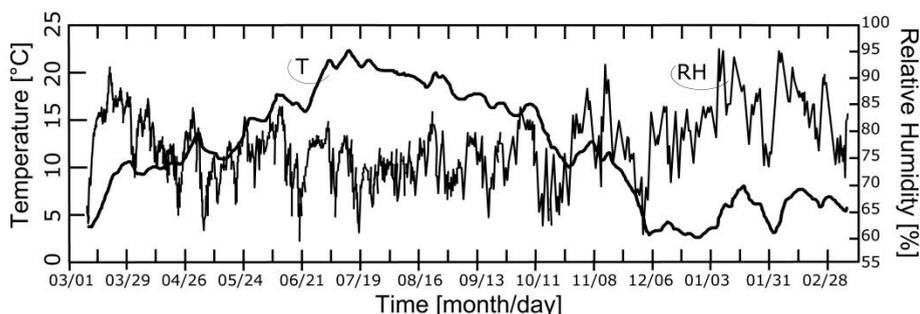


Figure 6. Evolution of the temperature (T) and the relative humidity (RH) recorded during twelve months behind the four statues in the mausoleum (location I). (Modified for publication). The monitoring campaign started in March 2010.

Based on the ECOS/RUNSALT output the frequency of salt crystallisation transitions is calculated according to the variations in T and RH recorded throughout the year. The RH recorded behind the statues reaches a minimum of 59% indicating that crystallisation and deposition of salts other than gypsum detected in locations I and II should rarely occur at a temperature of 12°C, which was the selected T for the modelling of the crystallisation sequence. However, the three other dataloggers in the chapel recorded a lower minimum RH. Moreover, the crystallisation of the salt mixture in the limestone will start at a higher RH when the temperature drops below the average 12°C.

Critical locations are IV and V as the RH crosses frequently the deliquescence points of the detected salts, and hence frequent transitions between crystallisation-deliquescence are expected. In relation to the recorded climate data the crystallisation of potassium and sodium nitrate occur throughout the year.

In location IV potassium nitrate crystallises approximately 20 times during the year and remains in the crystalline form during most of the time. For sodium nitrate 33 cycles are counted. The situation in location V at the lower end of the wall is, as stated in paragraph 4.2, more complex. Different salts show frequent crystallisation cycles: mirabilite ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) 54 cycles, apthitalite ($\text{Na}_2\text{SO}_4 \cdot 3\text{K}_2\text{SO}_4$) 14 cycles, darapskite ($\text{Na}_2\text{SO}_4 \cdot \text{NaNO}_3 \cdot \text{H}_2\text{O}$) 48 cycles, niter (KNO_3) 48 cycles, and halite (NaCl) 11 cycles. Higher in the wall the situation is comparable to location IV with respectively 8 crystallisation cycles of niter and 31 cycles of nitratine (NaNO_3). The high number of crystallisation cycles explains the observed damage in these areas.

5. Conclusions

The results of the hygroscopic moisture contents are supported by the results of the quantitative ion data of the aqueous extract of the lifted samples, which in turn confirmed the presence of relatively high amounts of nitrate salts. Since the site functions as a funeral chapel a contamination with nitrates is quiet logic. The RH behind the statues in the mausoleum never drops below 59%; therefore hygroscopic salts such as calcium nitrate remain constantly in solution and can cause visible moisture stains as well as damage to the polychrome layers on the limestone. The high moisture contents can also encourage biological contamination.

The RH in the chapel ranges approximately from 59 to 95%, while the modelled transition point of nitratine at 12°C is generally situated between 55 and 65% and increases as the temperature decreases. Therefore, it is assumed that the visual damage on the limestone of the mausoleum might to a certain extent be explained by salt activity especially during dry winter periods. Fluctuations of RH across the phase transition points of salts are particularly harmful and should be avoided, certainly if a climate control system is considered. In the future, it is expected that salt damage will increase especially since the restoration campaign is focussed on the general reduction of the moisture load of the stone materials of the chapel, such as treatment against rising damp and drainage of the underground, generally resulting in an overall decrease of the RH which in turn favours the crystallisation of salts. In addition, temperatures descending to 2.2°C, and hence close to the freezing point, are recorded during the winter period. As the data logger was positioned at approximately one-meter height and well protected behind the statues in the mausoleum, some freeze-thaw cycles and therefore frost damage shouldn't be excluded in exposed areas close to the ground, although a non-weathered Avenderstone is considered frost resistant.

To exclude any further deterioration it is deemed necessary to keep the temperature in the chapel above 0°C and to carry out a salt extraction where possible. A salt extraction by means of poulticing is also recommended for the brick masonry in locations IV and V after the removal of the plaster. In these locations the salt contamination includes hygroscopic and destructive salts. The poultice materials to reduce the salt content needs to be tailored in relation to the substrate to desalinate. The pore size distribution of the poultice will influence the mechanism of transport for

desalination, by advection or diffusion (Bourgès 2008; Sawdy 2008). The use of a specific poultice composed of kaolin, sand and cellulose is capable to extract salts from bricks up to a depth of 7 cm (Lubelli 2011). During a later stage of this research the porosity and water absorption of the limestone will be determined to create a specific poultice, by consulting Lubelli's (2010) article. The experimental application of several poultices will be carried out. After each treatment the nitrate content in the limestone and the residual of the poultice will be verified by ion chromatography.

This study revealed the phenomenon of salt accumulation by capillary rising damp and the presence of salts to certain heights in the masonry, plaster, and limestone of the walls and the mausoleum in the funeral chapel. It is not certain that this phenomenon is no longer active at present. Therefore it cannot be guaranteed that effective salt extraction interventions will act as a durable remedy for the salt problems. It is important to consider all options for a sustainable conservation strategy including a treatment against rising damp at the base of the walls or under the elements in natural stone of the mausoleum. If the phenomenon of capillary rising damp is treated accordingly, the application of a new plaster on the walls should be carried out using a traditional lime mortar. In case the treatment against capillary rising damp isn't feasible, then the use of a mortar resistant to salts is considered and if necessary a finish is applied with a mineral based paint with a minimum amount of organic components.

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