

## **Salt sources revisited**

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### **Abstract**

The hierarchy of possible approaches to conservation if maximal effectiveness and sustainability are aimed for, are preventive conservation i.e. addressing the causes, passive intervention i.e. addressing the activation mechanisms, remedial intervention i.e. strengthening the object and the least efficient restoration, which camouflages the damage.

To apply this principal of minimal intervention on buildings suffering from salt damage and especially when aiming to conserve preventively it is necessary to know the sources of the salts.

The possible sources of salts are:

Autochthonous salts – salts coming from the materials they damage.

Salts from neighbouring building materials.

Salts from human activity in the vicinity of the building.

Salts in connection with the use of the monument throughout history.

Salts having to do with the general geographic situation of the building.

In the paper general indications are given which salts can be released from which sources and some examples for common as well as uncommon salt sources are reported on, some of which for the first time.

**Keywords:** salt source

## 1 Introduction

Salt weathering is mainly a physical process. Even though the exact mechanisms provoking this kind of weathering are still not completely understood [1], the weathering itself has been observed on countless monuments and shown by laboratory experiments. It is generally agreed that this weathering process is mainly triggered by the salt system reacting with a changing environment, by letting different salt phases crystallise, re-crystallise, hydrate, de-hydrate, deliquesce according to the environmental conditions. On non-porous materials salt growth or transformation processes will not lead to damage, as can easily be demonstrated with laboratory experiments with salts e.g. in glass containers. When the salts are however sitting within an inorganic porous material such as most natural stones or mortars, the recurring change of their physical form will, in many cases, lead to the degradation of the material [2]. It can be observed that the porous system of a material determines the type and intensity of salt deterioration caused by a specific salt system in a specific environment to a large extent [3].

If maximal effectiveness and sustainability are aimed for, the most effective approaches to conservation are preventive conservation i.e. addressing the causes or passive intervention i.e. addressing the activation mechanisms [4]. On buildings, suffering from salt damage passive intervention consists of sheltering from unsuitable environmental conditions. Preventive conservation consists in stopping the salt supply, which requires to know the salt source.

Practitioners confronted with the task of finding the sources of the salts occurring in a specific case might find this text and its bibliography useful and it might enlarge the appreciation of the question to newcomers in the field.

## 2 Salts

Salts are chemical compositions consisting of cations (metal ions) and anions (acid residue anions) connected by ionic bonds. Therefore most of them are at least slightly soluble in water. The ions found most commonly on monuments are sodium ( $\text{Na}^+$ ), potassium ( $\text{K}^+$ ), ammonium ( $\text{NH}_4^+$ ), calcium ( $\text{Ca}^{2+}$ ), magnesium ( $\text{Mg}^{2+}$ ), chloride ( $\text{Cl}^-$ ), sulphate ( $\text{SO}_4^{2-}$ ), carbonate ( $\text{CO}_3^{2-}$ ), hydrogen carbonate ( $\text{HCO}_3^-$ ), nitrate ( $\text{NO}_3^-$ ) and oxalate ( $\text{C}_2\text{O}_4^{2-}$ ) [2]. But salts with less common ions like nickel ( $\text{Ni}^{2+}$ ) [5], copper ( $\text{Cu}^{2+}$ ) [6] or aluminium ( $\text{Al}^{3+}$ ) [7], to name only a few, can be found as well.

Some salts can crystallise either in a hydrated or non-hydrated form according to the environmental conditions surrounding them. In humid air salts can take up water from their environment, start to creep, deliquesce and finally form a salt solution. If ionic solutions from different sources are mixed, their cations and anions can regroup on drying and form “new” salt

species. The specific salt minerals found in an efflorescence will strongly reflect the environmental conditions at or shortly before the time of sampling and they can be formed by only some of the ions actually present within the pores of the material while the other ions remain in solution (see e.g. [8]).

Looking for the sources of salts means therefore looking for the sources of the different ions rather than the sources of individual salt minerals. More so because in most cases the ions found in a porous material on a monument are coming from diverse sources to form the salt system on hand at the time and place of observation.

### 3 Sources for salt ions

#### 3.1 Salt ions from building materials

##### 3.1.1 Salts in stone materials and natural waters

Most minerals and hence stones are at least partly soluble in water. Therefore, the mineralization of spring water depends on the geological environment the water passed through on its way to the surface [9].

Basically all natural stones contain at least small amounts of water-soluble ions. For instance the average values measured in seven different types of molasse-sandstones from Bern, Switzerland (CH), per gram of material were:  $\text{Na}^+$  (32  $\mu\text{g}$ ),  $\text{K}^+$  (96  $\mu\text{g}$ ),  $\text{NH}_4^+$  (7  $\mu\text{g}$ ),  $\text{Ca}^{2+}$  (510  $\mu\text{g}$ ),  $\text{Mg}^{2+}$  (111  $\mu\text{g}$ ),  $\text{Cl}^-$  (12  $\mu\text{g}$ ),  $\text{NO}_3^-$  (1  $\mu\text{g}$ ),  $\text{HCO}_3^-$  (2249  $\mu\text{g}$ ),  $\text{SO}_4^{2-}$  (29  $\mu\text{g}$ ) [10]. In spite of the fact that such values would be necessary to estimate the salt input from the stone material itself, autochthonous salt contents astonishingly seldom are published.

Common natural rocks do not contain nitrates, if these ions are present in spring water or a rock they indicate a contamination from the infiltration of surface waters.

Like water in the soil, rainwater will contain ions. The main source of dissolved material over the ocean and near coasts, is sea salt [9]. Other sources of dissolved matter in rain are diverse atmospheric pollutants including sulphates, nitrates and ammonia as well as organic acids. Generally “the composition of rain in any one location may vary greatly with time. The first drops of rain at the beginning of a storm may contain most of the soluble material available in the atmosphere; rain toward the end of a storm is relatively dilute” [9].

##### 3.1.2 Salts in mortars

Most mineral mortars contain water-soluble parts. This is not astonishing as mortars are mainly made by mixing natural rock particles (gravel, sand, clay) with a binder, produced by calcinating natural rock materials, and

adding water. As explained before all these constituents can contain water-soluble parts.

Historical mortars usually have been made using local materials. If the locally found sand contains water soluble parts, these will contribute to the salt content of the mortar. Such was the case for a lime mortar from the Luegisland tower in Luzern (CH) where the sand contained a noticeable amount of gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). During a few hundred years this did not cause any degradation problems, but it “falsified” our salt analysis, in fact the measured high calcium and sulphate content only could be explained after the thin section analysis of the mortar had revealed the gypsum grains.

Some building materials such as gypsum stucco or plaster of Paris consist predominantly of soluble salts. On the Swiss Plateau pure gypsum mortars were e.g. used frequently as jointing mortars for facades out of sandstones. In a recent study, sulphates participating in stone deterioration could be attributed not only to air pollution but also to nearby gypsum mortars [11].

Another group of mortar binders consisting mainly of water-soluble minerals are dolomitic limes. In the central Alpine regions, until quite recently, these materials were not well known and the common assumption was that the binders of historical mortars consisted generally of lime. Recent analysis however has shown that especially in remote areas with no suitable limestone but abundant dolomite, the latter was the (logical) material to use for the production of mortar binders. One such an example is Müstair monastery, a world cultural heritage site situated in the Val Müstair (CH) where dolomites are the predominant sedimentary rocks [12]. Analysis of plasters, wall painting supports and white washes dating from the 9<sup>th</sup> until the 19<sup>th</sup> century have shown that dolomitic lime had been used quite exclusively. These mortars and washes contain amongst other minerals the following slightly water soluble minerals hydromagnesite ( $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ ), brucite ( $\text{Mg}(\text{OH})_2$ ) and periclase ( $\text{MgO}$ ) [13].

The mortars at the ruined church San Gaudenzio in Casaccia consisted of dolomitic lime too. When the restoration works began the architect was puzzled by the occurrence of strange salt efflorescence, which could be identified as being hydromagnesite, washed out of the mortars and deposited on stone and plaster surfaces as efflorescence and crusts.

Modern hydraulic lime or cement mortars contain water soluble sodium and potassium reacting with  $\text{CO}_2$  of the surrounding air to form alkali carbonate or hydrogen carbonate salts [14]. Our analysis of mortars that contain such binders have shown that another major water soluble constituent of such materials seems to be sulphate. After setting these materials contain abundant amounts of calcium hydroxide, which, if washed out and getting in contact with  $\text{CO}_2$  from the air, will cause white calcite crusts on monument surfaces.

Tetmajer reports in 1894 of the addition of sodium chloride to the fresh mortar mixture when building at low temperatures in winter, giving examples from Switzerland, Germany and France [15]. He suggests to use combinations of alum, sodium chloride, sodium carbonate and burnt lime when building at temperatures below  $-10^{\circ}\text{C}$ .

## 3.2 Pollution situations

### 3.2.1 Air pollution

A few decades after the end of the Second World War the damaging effects of air pollution, primarily  $\text{SO}_2$  and  $\text{NO}_x$ , on stones became a major subject of concern amongst conservators and conservation scientists especially from the highly developed and industrialized countries [16]. The phenomenon at that time was however not new as already in 1864 the effects of air pollutions on stone are studied [17]. Today air pollution seems not anymore to be considered to be a major problem for stone conservation in Europe. In fact the  $\text{SO}_2$  emissions measured in today's Switzerland even in the most polluted areas do not even reach a tenth of the values measured in the nineteen eighties [18] and the pollution levels evolved probably similarly all over Europe. However, in other parts of the world the situation seems to be very different [19] and even in Europe the sulphate (and other) ions deposited on stone and mortar surfaces during the high times of air pollution are of course still present within the material surfaces and continue to exert their deleterious effects [11].

### 3.2.2 Waste water and faeces

Faeces are generally rich in nitrogen from which ammonium and nitrate ions can derive. Soils near human settlements contain salts from the decomposition of organic materials and especially from faecal matter. The hygienic conditions in cities and other settlements in ancient times, were quite different from today's [20]. Evacuation systems for faeces and other waste waters were rather open systems and cattle, draught and other animals lived all around the houses and in the streets. Important amounts especially of nitrates have infiltrated into the soils and from there into the walls during these times [20].

Apart from the nitrogen containing ions, the manure of cattle and other herbivore animals is dominated by potassium [21]. In the urine of ruminant animals, potassium is generally largely dominant over sodium (oral communication 2005, Ms. Dr. Kaufmann, Tierspital, Bern). In human urine, however sodium and chloride are quite abundant and the content in inorganic ions of a so called 24 hours human urine are [22]:  $\text{Na}^+$  (5.9 g),  $\text{K}^+$  (2.7 g),  $\text{NH}_4^+$  (0.8 g),  $\text{Ca}^{2+}$  (0.5 g),  $\text{Mg}^{2+}$  (0.4 g),  $\text{Cl}^-$  (8.9 g),  $\text{PO}_4^{3-}$  (4.1 g),  $\text{SO}_4^{2-}$  (2.4 g). From the same source, it can be calculated that a 24h human urine contains about 10 g of nitrogen, which would result in over 40 g of nitrate per human being per day.

In Durham Cathedral, nitromagnesite ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) together with a little gypsum and quartz ( $\text{SiO}_2$ ) was at the basis of slippery, oily looking patches occurring locally on the floors [23]. When looking for the sources of the magnesium nitrate the authors could exclude the local sandstones “employed in the cathedral flooring” which “are normally exceedingly poor in magnesium, and contain virtually no nitrogenous material” [23]. The most likely source of magnesium is the seating crushed Magnesian Limestone from the region upon which these flagstones were formerly laid. As for the nitrate source decomposition of cadavers, extensive use of incandescent gas mantles or the storage of gun powder in the cathedral, were considered but could be excluded [23]. It could finally be shown that the most likely source for the nitrate are a few 1000 prisoners that were kept in winter 1650-1651 in the cathedral and who most likely stayed at the warmer end of the cathedral and used the colder end for their toilet.

On the ruined castle of Belfort in Brienz/Brinzauls (CH) where severe degradations had been found below two medieval toilets (garderobes) one of which had been only in use a rather short time and the other had been used for over a hundred years. The salt composition beneath the latter was clearly dominated by sodium and chloride in very similar proportions to human urine [24]. The salt system underneath the precociously abandoned garderobe, was however, clearly dominated by potassium and nitrate. This could be attributed to a herd of Alpine Ibex (a sort of wild goats) regularly gazing and resting on top of the walls, right above the former toilet. To verify this attribution we analysed the water extraction of faeces from these animals and found the following inorganic ion content per gram sample:  $\text{Na}^+$  (49  $\mu\text{g}$ ),  $\text{K}^+$  (5166  $\mu\text{g}$ ),  $\text{NH}_4^+$  (157  $\mu\text{g}$ ),  $\text{Ca}^{2+}$  (741  $\mu\text{g}$ ),  $\text{Mg}^{2+}$  (629  $\mu\text{g}$ ),  $\text{Cl}^-$  (674  $\mu\text{g}$ ),  $\text{NO}_3^{2-}$  (2538  $\mu\text{g}$ ),  $\text{PO}_4^{3-}$  (651  $\mu\text{g}$ ),  $\text{SO}_4^{2-}$  (247  $\mu\text{g}$ ).

### 3.3 Common salt, table salt

Many human activities involve the use of sodium chloride, which explains the huge importance this material always has and had [25]. In former times, salt was transported in wooden barrels or linen sacks [25] and it often was stored as piles of salt without any container. Whoever has handled salts in laboratory knows that salts have an annoying tendency to creep up the walls of their containers and even out of them [26, 27], it is therefore easily imaginable that such creeping can explain how salts moved away from their stocking places into the surrounding materials. Fact is, that big amounts of this salt are found in walls at or near places where the salt market took place, e.g. at the Salzlaube in Bern [28] or where salt was stocked e.g. in the crypt of St. Maria im Kapitol in Köln [3].

### 3.4 Professions dealing with salt and general salt uses

Many food-producing professions use apart from sodium chloride diverse other salts for different purposes. For example: Salt-cured meat is

produced using sodium chloride but also sodium and potassium nitrate. Cheese making facilities use the same salts. Bakeries use potassium nitrate and sodium carbonate or hydrogen carbonate.

The process of tanning of leather consists of many steps where acids, lyes and salts are used. Amongst others, sodium, chloride, sulphate and aluminium ions can have their source in this activity [29].

Since the end of the 16<sup>th</sup> century it was known that mixtures of ice, water and salts can be used to reach temperatures well below freezing [30]. Before the invention of the refrigerator, this was used to cool for medical or laboratory purposes or to cool drinks or food. An often used and easily available salt for this purpose was potassium nitrate [31].

On the other hand, salts are used for de-icing. Apart from NaCl de-icing salts can contain: potassium chloride, calcium chloride, magnesium chloride, potassium and sodium acetate; potassium and sodium formate or even urea [32].

Very hygroscopic salts like calcium chloride are used to keep natural tracks humid to bind the dust.

We have observed that sodium chloride is sometimes used as an herbicide on paved surfaces adjacent church walls to prevent herbs growing between the stones.

Synthetic fertilizers contain nitrates, phosphates, potassium, calcium and magnesium; they often are applied as a powder or in granulated form. These materials are distributed over the fields like seeds would be and can be thrown this way far away from the fields. We have observed such materials landing on nearby surfaces of monuments [33].

Potassium nitrate is an important constituent of gun powder. In historic time, it was mainly produced by leaching out the floors of stables and settlements [34]. In Switzerland this was mainly done while the cattle were on the mountain pastures during summertime by the so-called "Salpetersieder", a highly regulated profession [35].

### **3.5 Salts resulting from conservation or restoration measures**

Waterglass is a solution of sodium, potassium or sodium and potassium silicate in water. In a building context, it is used e.g. as the binding media for silicate mineral paint, for fireproofing or in injection materials against rising moisture in walls. Waterglass is applied as a liquid and sets to an insoluble aqueous SiO<sub>2</sub> and water soluble sodium or potassium carbonates [2]. During the 1950ies and 1960ies waterglass was extensively used in the German Rheinland to consolidate wall paintings in most cases with quite disastrous side effects due to the high sodium and/or potassium input [36].

Interesting enough since a few years lithium waterglass is sold for the consolidation of mortars and renders [37]. We are actually expecting lithium salts to occur in the future at buildings treated with these materials.

Desalination poultices can not only extract salt ions from the treated support but also release ions into the support; this should be controlled before the use of such material. In a commercial poultice material containing cellulose fibres and montmorillonite, 1051 ppm of sodium as well as traces of ammonium, potassium and chloride were analysed in the water extraction [38].

In the vaults of the St. Johannes chapel in the monastery Allerheiligen in Schaffhausen (CH) we did find large amounts of calcium nitrates, whose occurrence can only be explained as the residue of the uncovering of the wall paintings by using nitric acid [39].

Acid cleaning can easily lead to salt damages and the salts involved will strongly reflect the acid used. Accordingly, [40] report on formate salts effloresce after the use of formic acid.

After acidic cleaning lyes might be used to neutralize and by this of course producing salts. Lyes such as sodium hydroxide and ammoniac can be constituents of paint removers or cleaning poultices. P. and L. Mora have suggested a poultice containing ammonium and sodium bicarbonate and sodium EDTA for the cleaning of crusts on limestone surfaces [41].

Since a long time people have observed the deleterious effects of salts concentrated at the bases of walls and many attempts have been made to remedy by transforming the harmful salts present to less harmful ones. An early such proposal was published in 1845 [42], who believed that the salts consisted mainly in the very deliquescent calcium nitrate sometimes together with some calcium chloride. Therefore he suggested that the salts should be transformed to much less deliquescent salts by applying solutions of sodium sulphate, phosphate or carbonate of which he clearly preferred the sodium carbonates. He believed that the newly formed calcium salt would remain insoluble and trapped within the sandstone whereas the also resulting sodium nitrate would form efflorescence on the surface from where it could easily be washed off.

Even today new restoration techniques are liable to add salts into the treated stones. For example, to increase the stability of a newly proposed consolidation treatment based on the formation of hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ), it is proposed to add calcium salts such as calcium chloride to the other reagents [43, 44].

Up until today attempts are made to chemically transform harmful salts present within porous building materials into less or not harmful salts. There are situations where such methods correctly applied can be very successful to fulfil the task wanted [45]. However the products sold nowadays as remedy for salt problems on a grand scale do seem to be rather potential producers of salts than real solutions to the problem. In the



course of a very recent expertise, we did come across products containing amongst other things the following constituents alone or in various mixtures: barium phosphate, phosphoric acid, magnesium, zinc or lead fluorosilicates. All sellers of these products indicated that none of the products can be used when nitrates are present, because the nitrates formed as a result of such a treatment are all very water soluble and/or hygroscopic and additionally the nitrates of barium, zinc and lead are highly toxic. This means that these products cannot be applied in the zone of rising humidity at the basis of walls, which for many buildings are the parts that are the most contaminated by salts.

It has been suggested already more than 100 years ago to use fluorosilicates as stone and mortar consolidants [46] and these products are in use until today. Other treatment products were en vogue at former times but are now forgotten. One such treatment was called Testalin; it consisted in a consecutive impregnation of the stone surface with aluminium acetate and potassium soaps [47]. The mentioned historical treatments are suspected to be at the basis of aluminium and ammonium salts found on diverse buildings in northern and north-eastern Germany [7, 48-50] and recently in Switzerland too [51].

#### **4 Conclusions**

To find the sources of the salts of an individual building, it is necessary to know the autochthonous soluble salt content of the original and repair building materials, the potential salt input from the environment, how the building and its surroundings have been used throughout its history and what restorations have taken place.

If uncommon ions are found when analysing the salts from a monument, uncommon sources have to be looked for. Very often these sources have to do with the use of the building or with former conservation/restoration treatments. Conservation projects often take place on objects that have been treated at least once in the course of the past fifty or sixty years and in many cases (modern) materials have then been used. Such materials might be at the basis of the problems encountered nowadays. Our latest encounters in this direction are acrylates and probable lactates as efflorescence on historical monuments.

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