5. References


Determination of the water uptake and drying behavior of masonry using a non-destructive method

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Abstract

Historic masonry of different types often suffers from various enrichments in surface and near-surface areas. Salts, atmospheric pollutants and other materials from external sources are possible causes for these enrichments which can directly influence the water uptake and drying of the masonry. Thus, a compactification of the superficial porous network can be expected in the case of crust formation, an enhanced water uptake in the case of hygroscopic salts. Both of these lead to a change of the intrinsic and unaffected water balance. Besides damaging effects, possibly provoked by crusts, and elevated salt contents, it is of interest to investigate how affected walls behave at a given relative humidity, e.g. threshold values for enhanced water uptake or release influenced by salts. The present study reports on a non-destructive chamber method used to investigate the impact of encrustations and salts on the masonry’s behavior concerning drying by determining moisture flow. Advantages of the chamber are its easy and non-destructive application on the masonry using a sealing material that is solely pressed on the wall without leaving residues and the use of harmless water vapor. An investigation of wall paintings in the cloister of the St. Peter Cathedral in Schleswig which in parts suffer from massive yellowish encrustations is presented to demonstrate the promising results obtained with the chamber method.

Keywords: non-destructive method, water uptake and drying, encrustations

1. Introduction

Masonry of different kind is often affected by various types of enrichments at surfaces and near-surface regions. Such masonry includes e.g. bare brickwork, plastered walls, wall paintings and other monuments. Porous building materials are used for the construction of those objects since antiquity. Due to their surface and their porous network they are directly exposed to the atmosphere. Having an additional connection to the ground water, atmospheric and anthropogenic influences can act on them. Enrichments can be present in form of salts, atmospheric pollutants and other materials from external sources. Salts are mostly introduced via capillary rise of ground water or anthropogenic effects like usage of de-icing salt, agriculture, animal excrements and cleaning or conservative actions.1 Also anthropogenic emissions, e.g. caused by the combustion of oil and coal, lead to the deposition of pollutants on the porous objects which are a source for salts as well, especially for the formation of gypsum.2 In addition, cleanings and conservative procedures can introduce other materials like resins or fixatives whose ageing products can also enrich over the course of years.3 Sulfur dioxide pollution was a problem in industrial areas until the 1990s. With the distribution of exhaust gas purification for power plants and a change in lifestyle the promising results obtained with the chamber method.
towards a declining use of coal in domestic heating the problem was reduced, at least in Europe.\textsuperscript{5,6} Regarding the formation of crusts, gypsum as a result of SO\textsubscript{2} impact, might be the most popular one.\textsuperscript{7,8}

The processes of water uptake and release in a porous medium in contact with its surroundings are described by the sorption equilibrium. As a function of the materials' pore size distribution, its water content and the ambient temperature and relative humidity (RH) water vapor may be adsorbed from or released to the atmosphere. Salts in the pores of building materials not only have a high damage potential due to the generation of crystallization pressure.\textsuperscript{9} Salts also have a direct influence on the water uptake and release due to salts may be examined. In this work we report on the application of a chamber method\textsuperscript{10} that was originally developed to investigate the deposition of air pollutants such as SO\textsubscript{2} onto material surface.\textsuperscript{11,12} In contrast to the deposition of atmospheric pollutants, the water sorption is a reversible process. Nonetheless, the chamber method is also suitable for the observation of moisture flows across the material surface in both directions, i.e. hygroscopic water uptake and evaporation during drying. Questions concerning the behavior during drying of masonry containing salts or other foreign substances and threshold values for augmented water uptake and release due to salts may be examined with this non-destructive method.

2. Experimental
2.1. Chamber method

For the measurement of moisture flow a chamber as shown in Figure 1 was used. The cylindrical chamber (1) made of POM (poloxymethylene) and PMMA (poly(methylmethacrylate)) with a diameter of 100 mm is mounted on a POM-base plate with dimensions 300x150 mm\textsuperscript{2}. For the measurements, the plate with a cylindrical aperture on the back side is gently pressed against the wall under investigation using sealing strips (Te-samoll P-profile) which can be removed without residue after the measurements. An adjustable air flow with controlled relative humidity passes the chamber through the inlet (2, 3) and outlet ports (5, 6), respectively. The chamber is ventilated using a fan that is installed on the lid and connected to the external power supply via inlet (4). Ventilation is necessary to achieve ideal mixing in the chamber, thus, to avoid influences of Brownian diffusion on the overall moisture exchange rates. In a well-mixed chamber, the relative humidity at the exit equals the RH inside the chamber and transport of water vapor to the surface or evaporation from the surface are the rate limiting processes. The base plate also carries two RH/T sensors (Hygroclip, Rotronic Messgeräte GmbH) that are used to monitor continuously the relative humidity and temperature both at the entrance and the exit of the chamber (7). The two sensors (±0.8 % RH and ±0.2 K) are connected to a Hygrolab C1 station (Rotronic Messgeräte GmbH).

In the experiments that were carried out on site, the drying behavior of the masonry was investigated. The airflow was generated by a membrane pump such that typical volume fluxes were 3.9 L·min\textsuperscript{–1}. Incoming air was dried in a silica gel drying tube positioned between membrane pump and chamber entrance. For other applications, the drying tower may be replaced with a humidification system using saturated salt solutions to control the relative humidity at the chamber entrance. The flow rate was measured using a gas meter connected to the chamber exit (6).

During the measurements the only data captured are relative humidity and temperature at the entrance and the exit of the chamber. Known quantities are the volumetric flow rate $Q$ (in m\textsuperscript{3}·h\textsuperscript{–1}) of the air flow and the surface area $A$ (in m\textsuperscript{2}) of the wall that is exposed to the cylindrical chamber during the experiment. The relative humidity $\varphi$ is defined as

$$\varphi = \frac{p_{\varphi}}{p_{\varphi,0}}$$

where $p_{\varphi}$ and $p_{\varphi,0}$ are the water vapor pressure and the saturation water vapor pressure, respectively. The latter was calculated using the Wagner Pruss equation\textsuperscript{11}. The absolute humidity $f$ is given by:

$$f = \frac{p_{\varphi} M_{v}}{(R T)}$$
towards a declining use of coal in domestic heating the problem was reduced, at least in Europe\textsuperscript{54}. Regarding the formation of crusts, gypsum as a result of SO\textsubscript{2} impact, might be the most popular one.\textsuperscript{78}

The processes of water uptake and release in a porous medium in contact with its surroundings are described by the sorption equilibrium. As a function of the materials’ pore size distribution, its water content and the ambient temperature and relative humidity (RH) water vapor may be adsorbed from or released to the atmosphere. Salts in the pores of building materials not only have a high damage potential due to the generation of crystallization pressure.\textsuperscript{9} Salts also influence the water uptake and drying behavior of such porous matter. Each salt has a characteristic, temperature dependent value of the relative humidity – the deliquescence humidity – at which it starts to pick up water from the atmosphere to form a saturated solution. Regarding salts in building materials, the hygroscopic behavior of the salt and that of the material both influence water uptake and release. Very hygroscopic salts, like nitrocalcite, Ca(NO\textsubscript{3})\textsubscript{2}·4H\textsubscript{2}O, with a high solubility take up high amounts of water also at moderate relative humidities;\textsuperscript{2} theoretically, leading to a saturation of the pore space with salt solution and consequently to a much higher water content within the masonry than in the salt free material at the same relative humidity. On the other hand salts with an extremely low solubility with this non-destructive method.\textsuperscript{7} Such processes concerning the behavior during drying of masonry containing salts or other foreign substances and threshold values for augmented water uptake and release due to salts may be examined with this non-destructive method.

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\[ \varphi = \frac{p_\varphi}{p_{\varphi,0}} \]  

(1)

where \(p_\varphi\) and \(p_{\varphi,0}\) are the water vapor pressure and the saturation water vapor pressure, respectively. The latter was calculated using the Wagner Pruss equation.\textsuperscript{11} The absolute humidity \(f\) is given by:

\[ f = p_\varphi M_w/(RT) \]  

(2)
where $M_w$ is the molecular mass of water ($0.0180153 \text{ kg mol}^{-1}$), $R$ is the gas constant ($R = 8.3144 \text{ J mol}^{-1} \text{ K}^{-1}$) and $T$ is the absolute temperature.

The difference of the absolute humidities at the exit ($f_{out}$) and at the entrance ($f_{in}$), i.e. $\Delta f = f_{out} - f_{in}$, is the result of either evaporation ($\Delta f > 0$) or water uptake from the air ($\Delta f < 0$). The moisture flux $F$ in units of g·m$^{-2}$·h$^{-1}$ is then given by:

$$F = \frac{\Delta f \cdot Q}{A} \quad (3)$$

For visual presentations the mass of water evaporated during drying, or the mass of water, adsorbed from the atmosphere in case of water uptake, respectively per unit area (in g m$^{-2}$) will be used which is obtained by multiplying the moisture flow rate with the corresponding time (in h). Hence, the transported quantity of water vapor in g·m$^{-2}$ is depicted at any point of the measurement.

### 2.2. Cloister of St. Peter Cathedral

In this study, wall paintings in the cloister of the St. Peter Cathedral in Schleswig, Northern Germany, were investigated. The so-called “Schwahl” is furnished with wall paintings from the 14th century showing monochrome scenes of Christ’s life in 22 bays and polychrome paintings of apostles, mythical creatures and leaf tendrils in the vaults and on the courtyard-sided walls. The restauration history of this object started quite early and involves several interventions including revisions, whitewashes and reparations which are not completely recorded in the years before the late 19th century.

Regarding the state of conservation the walls of the object showed high contents of nitrates and chlorides in most parts, gypsum enrichments on the surface and damage in the form of scaling and sometimes loss of pigmented areas. Due to a very humid climate the salts remain dissolved most of the time which is also the reason for the high moisture content of the masonry. Even though success was noted in the last years concerning salt reduction and gypsum conversion, the removal of yellowish encrustations on parts of the bays was not effective so far. These inhomogeneous encrustations most likely comprise organic materials which are probably alteration products of organic substances introduced in former restoration campaigns. With their massive appearance it was expected that they would not only affect the object in an aesthetic manner but also the natural water balance of the walls by clogging superficial pores.

The chamber method was used on different parts of the most affected bay no. 5 which is depicted in Figure 2. The area under the frieze is not pigmented and comprises a fresh and even lime plaster which does not show any visible encrustations (area 1). Within the frieze which is decorated with paintings of animals (area 2) a patchy yellowish encrustation was clearly visible. The same situation was noted in area 3, while in area 4 on the upper right side of the wall painting a massive and continuous crust was observed. To all these areas the chamber was applied to measure the moisture flow during drying, implemented by passing a dry airflow through the chamber. Two or three replicate measurements were conducted on all areas except area 3.

### 3. Results and discussion

The results of the multiple measurements at the different positions on the wall are represented in Figure 3. In all measurements an increase of the mass flow with time is observed which demonstrates the ongoing release of water vapor from the wall to the atmosphere. The curves representing the measurements in the freshly limed area 1 show the highest mass flow. Compared to that, the results from areas 2 and 3 with patchy encrustations show a significantly reduced water release. For test area 4, the moisture flow is even smaller and the curves reach a plateau after less than one minute of drying which means that there is no further release of water from the wall.

The curves representing replicate measurements in the same areas show some variability which might reflect minor shifts of the chamber position on the wall between two measurements. This includes e.g. a higher or lower extent of encrustations. Different initial moisture contents of the exposed parts of the wall might also be an important cause of the observed differences. Every measurement causes a reduction of the moisture content at a test position due to partial drying. If there is not sufficient time for re-humidification between two consecutive measurements, the initial moisture content is lower in the next run. Nevertheless, the curves belonging to the same

![Figure 2: Bay no. 5, cloister of St. Peter Cathedral in Schleswig. Measurement areas 1 to 4 are depicted.](image)

![Figure 3: Evaporated water versus time curves for the four areas in bay no. 5 (see Figure 2).](image)
where $M_w$ is the molecular mass of water ($0.0180153 \text{ kg mol}^{-1}$), $R$ is the gas constant ($R = 8.3144 \text{ J mol}^{-1} \text{ K}^{-1}$) and $T$ is the absolute temperature.

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Before the results will be discussed, the two distinct drying stages of porous materials may be discussed. During the first stage climatic conditions, air flow rate and surface texture are the limiting factors. Capillary moisture transport to the drying front at the surface provides more moisture than the amount that can evaporate, as climatic and transition conditions limit evaporation in this stage. Consequently, these conditions determine the slope of the curve (see Figure 3) while the material inherent properties, e.g. the moisture transport properties, influence the duration of the first stage. With decreasing moisture content within the material the liquid flow to the surface decreases. As less water is transported to the surface, the drying rate decreases and the drying front shifts into the material.15-17 Regarding the specific case of salt contaminated materials, as the pore solution is transported to the surface due to capillary forces, salts crystallize and accumulate on the surface or in near surface regions. Hence, they provoke pore clogging, hindering capillary transport to the surface and slowing down the drying rate.18

The curves shown in Figure 3 are quite similar during the first seconds with a nearly linear initial increase, i.e. a constant drying rate, characteristic of the first drying stage. The limiting condition at this stage were quite similar in all experiments thus, it is not surprising that equal initial drying rates are observed. In contrast, the material characteristics might differ between the positions of measurement leading to variations of the duration of the first drying stage. The decreasing slope of the curves of areas 2 and 4 represents the decreased drying rate at the beginning of and during the second drying stage. In case of area 2, a change of the slope is not clearly apparent, consequently assuming that the first drying stage was not terminated during the duration of the experiment seems reasonable. Without hindrance in form of a crust like in area 1, water vapor is transported uninhibitedly to the surface and the drying front can recede into the material in the second stage. If crusts represent an obstacle (see area 4), capillary transport to the surface is hindered as it was already mentioned in the preceding section. From microscopic images of samples from bay no. 3 it is known that the yellowish crusts cover the render and block the pores to a large extent as they comprise a dense layer. Thus, it can be assumed that pore clogging is not a consequence of salt crystallization close to the surface induced by drying but an effect caused by (most likely organic) deposits on the wall paintings which were already present before the experiments. In case of area 4, the course of the mass loss curve reaches a plateau after only 20 seconds, suggesting that only the condensed water on the surface of the crust evaporates during drying.

These observations demonstrate that the simple setup with the flow chamber yields useful and promising results concerning the water transport across the surfaces of encrusted wall paintings. Besides, this hindered water vapor transport caused by the encrustations has another consequence for the affected objects which could pose a risk. Dissolved salts within the porous network tend to crystallize on the surface when the relative humidity decreases to form subflorescences or efflorescences. If parts of the surface are covered with impermeable crusts, salts accumulate in uncovered areas adjacent to the crusts causing a risk. Also flaking of the crust is a possible scenario e.g. in the case of heavy accumulations and small cracks within the dense layer which allows drying of the underlying wall and crystallization of salts. The chamber method is a useful tool to detect and validate such critical circumstances on the object. The main advantage for the use on sensitive objects is the non-destructive character enabled by a sealing strip which is normally applied for the sealing of windows and doors. Limitations result for very uneven or bent walls where it is not possible to seal the room between chamber aperture and wall for level differences greater than the height of the strip. However, bare brickwork and other non-decorated walls might be investigated by using another test (Terostat IX, Loctite, Henkel) which adheres to the wall but can be removed without residues as well.

4. Conclusions

A non-destructive chamber method was successfully used to study the influence of encrustations on wall paintings on the drying behavior of the wall. Originally, the chamber method was used to determine the deposition of atmospheric pollutants to material surfaces. In the present study, a simplified version of the chamber was applied in an investigation of water release from wall paintings. It was shown that complete coverage with encrustations hinders the drying process of the render and the underlying wall by clogging superficial pores and blocking the passage for the intrinsic water vapor exchange between masonry and atmosphere. In addition, it was found that the extent of encrustation controls the drying behavior of the surfaces. Patchy and irregular crusts show mass flow curves between unaffected areas and fully covered areas. With these accessible observations potentially critical situations for the object considered as a consequence of hindered water vapor transport and accumulation of salts can be derived. These promising results induce other interesting applications of the chamber method on real objects like, for example an investigation of the hygroscopic water uptake of walls contaminated with salts, or the monitoring of the dynamics of the drying and water uptake behavior in response to daily or seasonal cycles of the climatic conditions.

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References


Measurement of salt solution uptake rates in fired clay brick and identification of solution diffusivity

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Abstract

Salt solution uptake rates in fired clay brick were measured with γ-ray to investigate the influence of salt on solution diffusivity for appropriate numerical analysis of salt solution transfer and crystallization in porous materials. NaCl and Na2SO4 are the main salts confirmed in Hagia Sophia (Aya Sophia Museum), Istanbul, Turkey, and are used in this experiment. We identified solution diffusivity as a function of solution content by a numerical analysis of solution transfer. The main results are as follows. Solution uptake tests demonstrated that saturated solutions take approximately six times longer to attain a steady state than the time needed for pure water to obtain steady state. This was thought to be caused by the viscosity of the dissolved salts and the presence of crystallised salt in the material. We identified solution diffusivity by multiplying moisture diffusivity for pure water by a constant to reproduce the measured solution content in the cases where large amounts of efflorescence was observed at the material surface. In the case of a saturated solution of Na2SO4, which is considered to be the main sources of salt that cause weathering of the structures. In the Hagia Sophia case, there exist complex humid–dry and wet–dry cycles that complicate the deterioration process that comprises adhesion, dissolution, and infiltration processes that transfer salts from the sources in the environment. Therefore, the process is much more complex than when ground water is the main salt source. The objective of this study was to develop an analytical model of the simultaneous transfer of heat, moisture, and salt and the subsequent crystallisation and dissolution of the salt for reproducing the deterioration mechanism caused by these complex salt deterioration processes.

Salt, which exists in the dissolved phase or solid phase (crystal) in porous material, causes significant changes to the material properties of the surface it contacts. This is due to a rise in viscosity of the solution and the descent of vapour pressure of the salt solution and changing pore structure in the solid phase. Therefore, material properties consti-