

## **Gypsum efflorescence under laboratory conditions: preliminary study**

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### **Abstract**

Gypsum efflorescence (GE) is a recent problem affecting modern clay brick masonry and permanently alters an aesthetic aspect of a building. This type of efflorescence affects exclusively masonry built in the last 30 years and usually appears a couple of years after construction. The GE characteristics indicate that the components are derived from the masonry itself and not from the atmosphere. The GE genesis and mechanism, and specifically the gypsum crystallisation behaviour in porous materials, are poorly understood.

We have designed and executed a series of crystallisation experiments, to compare gypsum behaviour with common efflorescing salts: NaCl and Na<sub>2</sub>SO<sub>4</sub>. Our experimental wick test setup consists of a brick sample mounted in a cell. The salt solution is supplied to the bottom sample side while the drying process exclusively occurs at the top. The experiments are carried out under three drying regimes: 'accelerated' (35 °C, 19% RH), 'laboratory' (24 °C, 58% RH) and 'cold' (12 °C, 80% RH). The first simulates accelerated efflorescence formation, the second is comparable to common wick tests while the third and last imitates early spring conditions. The experiments demonstrated that gypsum accumulates below the surface, unlike natural GE occurrences. Since the basic wick test does not simulate frequent masonry wetting, we have implemented wetting cycles which resulted in a considerable GE formation. We conclude that gypsum intrinsically has a subflorescing tendency, but frequent surface wetting progressively leads to GE formation.

**Keywords:** gypsum, efflorescence, masonry

## **1 Introduction**

Gypsum efflorescence (GE) is a recent problem affecting clay brick masonry. It is reported to occur in the UK [1] and the Netherlands [2], and there is a growing number of cases in Belgium. It concerns the formation of a thin white film at the brick surface, what considerably alters the aesthetic aspect of a construction. This problem affects exclusively recently erected constructions, while older buildings remain unblemished. White discoloration develops only on wind and rain exposed facades, leaving the sheltered ones intact. This indicates that the GE components are solely derived from the masonry, transported with moisture and accumulated at the surface. GE is apparent only after a few years, hence either its formation is delayed or very slow.

Apart from the recent gypsum efflorescence on brick masonry and the established black crust formation on calcareous stones, gypsum is rarely reported to create surface deposits. For non-calcareous stones gypsum is reported to be found just below the surface [3]. The same is observed for wick tests, where feeding a historic brick [4] or limestone sample [5] with gypsum solution resulted in solely subsurface enrichment with gypsum. It remains to be seen whether this behaviour is also valid for the alternating wetting and drying of facade masonry, since the typical salt crystallisation tests simulate only the drying process. Aside from these few studies, little work is devoted to gypsum crystallisation in porous materials: such studies are mostly focused on crystallisation of more soluble salts like alkali sulphates and chlorides. It is thus of interest to investigate further this controversial gypsum behaviour.

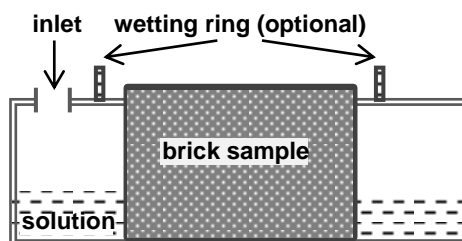
Consequently, in this investigation we apply a wick test setup to study efflorescing potential of salts. The choice of the wick test setup is motivated by maintaining the drying front at the surface, and then promoting efflorescence formation. We present preliminary results on gypsum crystallisation behaviour in porous clay brick, and compare it with this of common efflorescing salts: NaCl and Na<sub>2</sub>SO<sub>4</sub>, under various drying regimes. This comparison aims at evaluating whether severe drying conditions are suitable for reproduction and acceleration of efflorescence formation, and assessing whether efflorescing tendency is salt-dependent. Nevertheless, wick test simulates solution uptake via the bottom sample side only, unlike in field conditions, where the fluid is supplied at the drying side. Additional tests are therefore carried out to investigate the effect of frequent surface wetting on the efflorescing potential of gypsum.

## **2 Materials and methods**

Cylindrical samples with a diameter of 3 cm are drilled out of a common Belgian clay brick (density 1826 kg/m<sup>3</sup>, capillary absorption coefficient 0.68 kg/(m<sup>2</sup>·s<sup>0.5</sup>), and capillary and saturated moisture content 210 and

325 kg/m<sup>3</sup> respectively). Experiments on raw samples in demineralised water resulted in quick formation of sample-derived salt deposits. To limit this undesirable effect samples are initially subjected to a leaching procedure by placing them in a tank with demineralised water maintained at a temperature of 40 °C. Each 24 hours the leachate's conductivity is measured. The samples are then dried with a towel to aid in extracting the leachate from their pores, and are again submerged in fresh demineralised water. This procedure is repeated until the measured leachate conductivity reaches a constant background value. Once this requirement is achieved, samples are dried at 105 °C.

An optimal drying setup for promoting efflorescence formation should maintain the drying front at the sample surface. We have accordingly selected a wick test setup, which is depicted in Figure 1. The experimental setup consists of a cylindrical brick sample mounted in a cell. The top sample surface, being the original brick stretcher side, is exposed to the external conditions. The bottom side is in continuous contact with a chosen solution. The lid closing the experimental cell contains an inlet allowing for pressure levelling between the setup and the drying environment. The inlet is covered with a perforated tape, to limit solution evaporation from the cell. The complete setups are frequently weighed and pictures of the drying surfaces are taken to monitor the drying rate (DR) and the surface appearance, respectively.



**Figure 1:** Scheme of the experimental wick test setup.

Natural GE is often observed to occur only after a couple of years, what might be related to 'slow drying' field conditions. We have thus investigated the effect of more severe drying conditions on reproduction and acceleration of the efflorescence formation. The experiments are carried out in parallel under different drying conditions and by use of various salt solutions. An overview of the setups is presented in Table 1.

Three drying regimes are applied where both temperature and relative humidity (RH) are fixed during the experiment. The 'accelerated' (35 °C, 19% RH) conditions are implemented by a climatic chamber. This severe drying regime is chosen to accelerate the efflorescence formation. Both the 'laboratory' (24 °C, 58% RH) and 'cold' conditions (12 °C and 80% RH) are realised in an air-conditioned laboratory room. The former is similar to conditions commonly implemented in salt crystallisation tests,

while the latter imitates early spring conditions, as this season is often believed to favour efflorescence formation [1].

Despite widespread GE occurrence, gypsum is reported to crystallise below the surface of porous materials, while under the same experimental conditions other salts form efflorescence [4, 5]. The efflorescing tendency of gypsum is compared with those of sodium chloride and sodium sulphate, while setups impregnated with demineralised water are used as a reference. Both additional salts are often reported as efflorescing, and are as such suitable for assessing gypsum crystallisation behaviour. This is realised by impregnating the samples with appropriate salt solutions, prepared from pro-analysis quality salts: calcium sulphate dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), sodium chloride ( $\text{NaCl}$ ) and anhydrous sodium sulphate ( $\text{Na}_2\text{SO}_4$ ). All salt solutions are prepared at the same concentration of 2.2 g/L, equivalent 85% gypsum saturation at 20 °C. This way we aim to limit the effect of solubility on the rate of salt accumulation at the surface, what in turn allows for a better comparison of the efflorescence formation tendency. For convenience, we refer to the experimental wick setups containing brick samples impregnated with different salts or pure water, by simply gypsum,  $\text{Na}_2\text{SO}_4$ ,  $\text{NaCl}$  or water setups. Each measurement, for each drying conditions and salt solution, is carried out in triplicate (see Table 1).

**Table 1:** Summary of the investigated wick test setups.

| Conditions \ Setup                               | Accelerated<br>(35 °C, 19% RH) | Laboratory<br>(24 °C, 58% RH) | Cold<br>(12 °C, 80% RH) | Accelerated<br>+ Wetting |
|--|--------------------------------|-------------------------------|-------------------------|--------------------------|
| Gypsum   | 3 setups                       | 3 setups                      | 3 setups                | 3 setups                 |
| $\text{NaCl}$ , $\text{Na}_2\text{SO}_4$ , water | 9 setups                       | 9 setups                      | X                       | X                        |

Under the wick test conditions solution is supplied to a brick sample through the bottom side only. On the other hand, under field conditions it is the drying surface which receives wind-driven rain, being absorbed and then evaporated via the same masonry side. In order to simulate surface wetting we have modified the wick test by mounting a plastic ring around the exposed sample surface (see Figure 1), allowing for applying water on top of the sample, while the sample is maintained in gypsum solution. The wetting procedure is carried out under the accelerated conditions and involves 44 wetting episodes over 30 days, realised by applying 2 g of demineralised water on top of the sample.

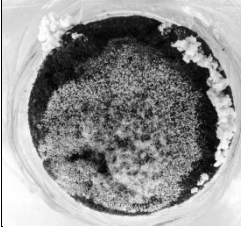
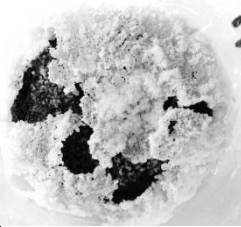
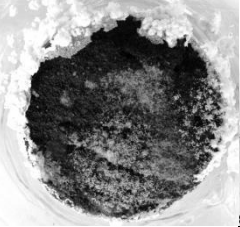
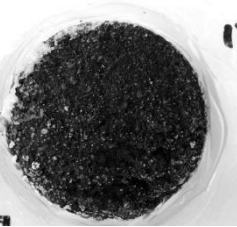



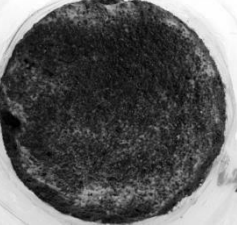
### 3 Results

#### 3.1 Visual observations

The photographs in Table 2 show sample surface appearance at the end of the experiment. Under laboratory conditions, surface appearance of the  $\text{NaCl}$  and  $\text{Na}_2\text{SO}_4$  setups is greatly affected by mould growth.

Nevertheless, it is still possible to distinguish between salt accumulation limited to peripheries and organic deposits.

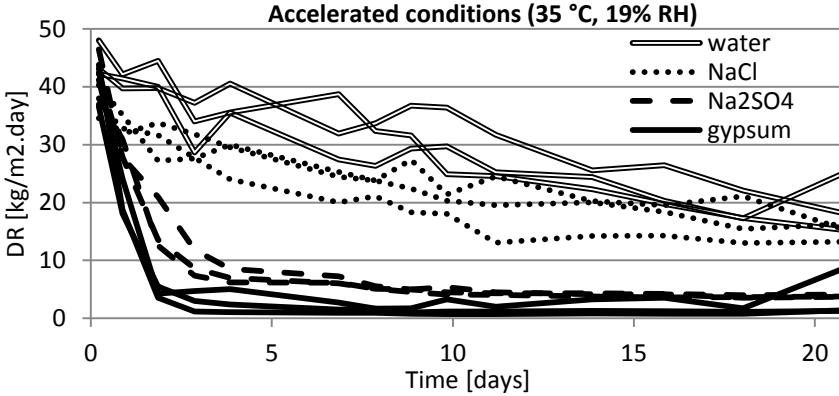
**Table 2:** Comparison of sample surface appearance after the wick test.

| NaCl setups   |   | Na <sub>2</sub> SO <sub>4</sub> setups  |  |
|---|---|---|--|
| Laboratory (59d)  | Accelerated (14d)   | Laboratory (59d)  | Accelerated (14d)  |
|  |  |  |  |
| Gypsum setups   |   |   |  |
| Cold (50d)  | Laboratory (59d)  | Accelerated (66d)   | Accelerated + wetting (30d)  |
|  |  |  |  |

The water setup samples do not show any deposit formation under both the accelerated and laboratory conditions (results not shown in Table 2). Deposit formation is absent or limited for all the gypsum setups under all the drying conditions, with few exceptions. Some fine dendrite growth on the sample edges is observed during the first days of the experiment under the accelerated conditions, but mostly it does not progress after two days into the experiment. Surprisingly, only one out of three samples under the cold conditions shows deposit growth at multiple locations over the surface which progresses, but only after 30 experimental days. In contrast, all three samples subjected to wetting cycles developed significant GE. For all the NaCl setups, both under the accelerated and laboratory conditions, a significant amount of salt deposit is formed. Even though the deposit formed under the accelerated conditions is covering almost the entire sample surface, it is almost uniquely anchored to the sample edges, where its growth was initiated and is then progressively wrapping over the sample surface. Under the laboratory conditions the deposit is localised uniquely at the edges, whereas substantial growth is observed only after 30 days. The Na<sub>2</sub>SO<sub>4</sub> setups behave similarly to NaCl under the laboratory conditions, but the accelerated setups produced only a slight deposit, most of it formed during the first two days after which it does not progress any further.

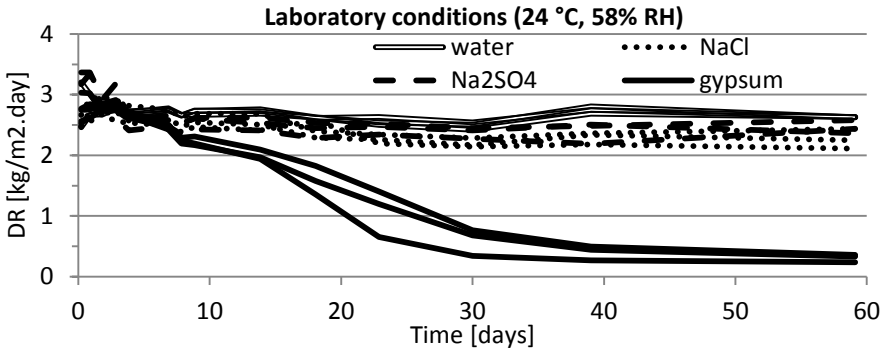
### 3.2 Drying rate

Figure 2 shows the DR of different setups under the accelerated conditions. The water and NaCl setups show the highest DR, which is progressively decreasing. For both gypsum and Na<sub>2</sub>SO<sub>4</sub> setups a rapid DR decrease takes place within the first days, after which it stays at a low and stable level. However, one of the gypsum setups exhibits elevated DR compared to the rest.



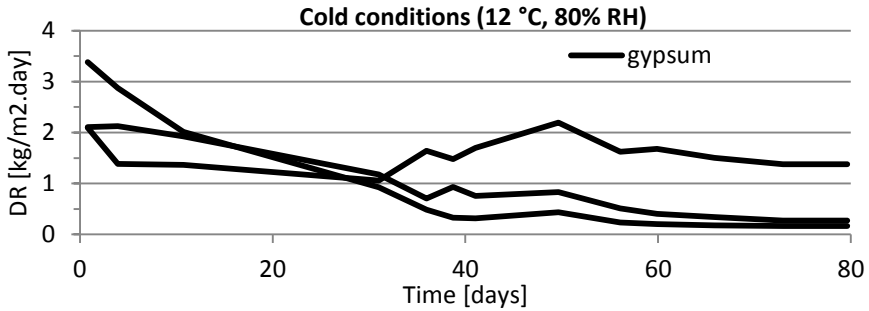
**Figure 2:** DR evolution of brick samples laden with water and various salt solutions and exposed to accelerated conditions.

Under the laboratory conditions (Figure 3) a stable DR is observed for all setups besides the gypsum ones, which exhibit a slow but steady drop in the DR, levelling after 40 days at a low DR.



**Figure 3:** DR evolution of brick samples laden with water and various salt solutions and exposed to the laboratory conditions.

A DR drop is also observed for the gypsum setups under the cold conditions (Figure 4), which stabilises after around 60 days. However, for one sample an increase in the DR is observed after 30 days.



**Figure 4:** DR evolution of brick samples laden with gypsum solution and exposed to the cold conditions.

Table 3 compares the initial and final DR between different setups.

**Table 3:** Comparison of the initial and final DR for all investigated wick test setups.

| Drying regime                     | Initial DR [kg·m <sup>2</sup> /day] |            |                                 |            |
|-----------------------------------|-------------------------------------|------------|---------------------------------|------------|
|                                   | gypsum                              | NaCl       | Na <sub>2</sub> SO <sub>4</sub> | water      |
| Accelerated                       | 40.5 ± 3.7                          | 37.3 ± 2.8 | 42.3 ± 4.2                      | 43.4 ± 2.3 |
| Laboratory                        | 2.7 ± 0.1                           | 2.5 ± 0.1  | 3.2 ± 0.2                       | 2.7 ± 0.4  |
| Cold                              | 2.5 ± 0.7                           | X          | X                               | X          |
| Drying regime<br>(number of days) | Final DR [kg·m <sup>2</sup> /day]   |            |                                 |            |
|                                   | gypsum                              | NaCl       | Na <sub>2</sub> SO <sub>4</sub> | water      |
| Accelerated (21d)                 | 1.3 ± 0.1*                          | 14.9 ± 1.5 | 3.8 ± 0.2                       | 19.5 ± 5.1 |
| Laboratory (59d)                  | 0.3 ± 0.1                           | 2.3 ± 0.2  | 2.5 ± 0.1                       | 2.6 ± 0.0  |
| Cold (80d)                        | 0.2 ± 0.1*                          | X          | X                               | X          |

\* A single particularly elevated DR curve is excluded for this estimation

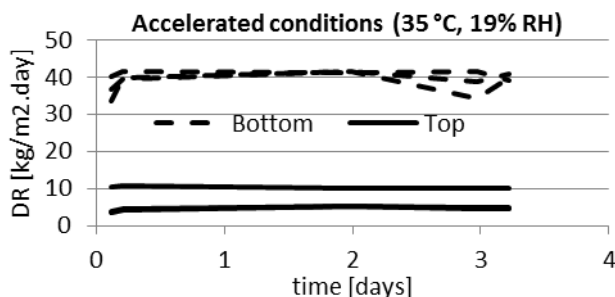
## 4 Discussion

### 4.1 Crystallisation at the peripheries

In cases where a substantial salt deposit develops at the sample surface, it is mostly limited to the sample's edges. Such localised crystallisation behaviour is also reported by Veran-Tissoires et al. [6] and explained by a variable evaporation flux over the exposed sample surface, which is highest at the sample's peripheries. Nevertheless, their study demonstrates that this effect can be eliminated by means of rings shielding the sample surface, which results in a similar efflorescence extent but distributed evenly. For this reason, the edge-localised salt accumulation reported here is a valid measure for evaluating salts' efflorescing behaviour.

## 4.2 Water samples' performance

The water laden samples under the accelerated conditions exhibit a considerable DR drop (see Figure 2), which is not accompanied by any major surface discoloration. To indicate the origin of this phenomenon, the samples were halved and subjected again to the wick test (see Figure 5). The bottom face exhibits a much higher DR than the top, demonstrating that some brick-derived components are accumulated under or within the brick surface and hindering the evaporation process.



**Figure 5:** DR of water setups through bottom and top face. The samples were first subjected to a wick test under the accelerated conditions.

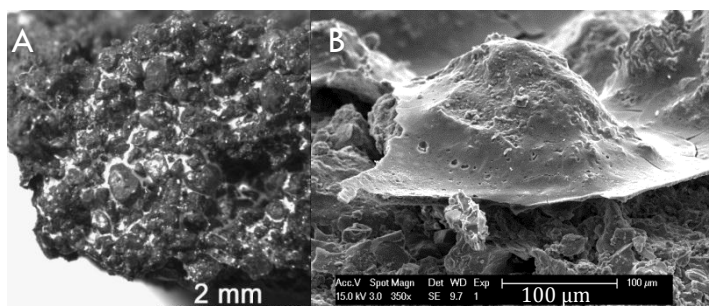
Sample surface analysis with a stereomicroscope revealed some local inter-grain deposit formation (see Figure 6A). A cross-section scanning electron microscope (SEM) image (see Figure 6B) from the same sample shows that it forms a thin sheet of a few micrometres thickness. SEM-EDAX analysis shows presence of 50% CaO, 30% of SiO<sub>2</sub> and a few other oxides. This is consistent with the formation of a portlandite layer, which is further carbonated to calcite, on a brick matrix rich in SiO<sub>2</sub>.

The water setups prepared with raw (not leached) brick samples resulted in the formation of abundant, water soluble deposits. This undesirable effect was successfully eliminated by applying the leaching procedure. Accordingly, the majority of well soluble brick components, for instance the alkali sulphates, are likely extracted from the samples. Nevertheless, raw bricks may contain CaO which hydrates to Ca(OH)<sub>2</sub> upon contact with water. The latter exhibits low solubility at 0.14 g / 100 mL (40 °C), thus it may persist in brick samples after the leaching procedure. The applied leaching procedure is in consequence effective against well soluble salts, but the low soluble compounds like calcium hydroxide are likely to be preserved. This undesirable effect appears also to affect the drying behaviour of the NaCl setups, as their drying behaviour follows the same trend (see Figure 2).

However, no such effect is observed for the water samples at the laboratory conditions after 60 days (see Figure 3). An equivalent amount of water evaporated after only 4 days under the accelerated conditions, and was accompanied by a considerable DR decrease of 15%. It appears therefore that this effect takes place exclusively under the accelerated



conditions, or is significantly delayed under the laboratory conditions. The solubility of calcium hydroxide at 25 °C is only 8% lower than at 35 °C, so does not seem to account for this effect. On the other hand the difference in the drying conditions is translated into a 16 times higher initial DR for the water samples under the accelerated conditions (see Table 3). Slower drying conditions favour the diffusion process over the advective transport, which may help to delay portlandite precipitation under the laboratory conditions. Under the accelerated conditions local calcium hydroxide saturation at the surface can be reached much faster leading to its precipitation, which inhibits the drying process.



**Figure 6:** Surface (A) and cross-section (B) images of a water sample tested under the accelerated conditions, pictures taken with stereomicroscope (A) and SEM (B).

### 4.3 Mineral-specific efflorescing potential

The three tested salt setups demonstrate distinctly different crystallisation behaviour, even though tested under the same experimental conditions.

#### 4.3.1 Sodium chloride

Sodium chloride crystallises exclusively as halite under both drying regimes. Irrespective of the drying regime, halite forms extensive deposits accompanied by drying rates similar to these of water samples.

#### 4.3.2 Sodium sulphate

The  $\text{Na}_2\text{SO}_4$  setups display heterogeneous responses: under the accelerated conditions they exhibit a DR decrease and scarce efflorescence, while under laboratory conditions they behave like NaCl ones, with extensive efflorescence and a DR at the level of a water sample (see Figures 2 and 3).

Steiger and Asmussen (2008) discuss in their paper the  $\text{Na}_2\text{SO}_4$  crystallisation pathways [7], what aids in explaining the different crystallisation behaviour of the  $\text{Na}_2\text{SO}_4$  setups. Even though thenardite ( $\text{Na}_2\text{SO}_4(\text{V})$ ) is the stable phase under both drying regimes according to the  $\text{Na}_2\text{SO}_4\text{-H}_2\text{O}$  phase diagram, the crystallisation pathway for its formation may include intermediate crystallisation of mirabilite

( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ). The origin of the observed discrepancies for the  $\text{Na}_2\text{SO}_4$  setups may stem from the fact that the experiments at 24 °C and 35 °C are carried out below and above the thenardite-mirabilite transition temperature of 32.4 °C, respectively. Assuming that the process is completely controlled by the thermodynamics, mirabilite crystallisation takes place first under the laboratory conditions, followed by dehydration to the thenardite phase during drying while thenardite precipitates directly under the accelerated conditions. Our results then show that mirabilite shows a tendency to form extensive efflorescence, while direct crystallisation of thenardite results in pore clogging and only scarce surface deposits. Nevertheless, thenardite formation upon mirabilite dehydration does not alter the overall efflorescence formation tendency.

It appears that the propensity towards efflorescence formation may be mineral-specific, even in case where both forms are different hydrates of the same base anhydrous compound, as demonstrated for  $\text{Na}_2\text{SO}_4$ .

#### 4.3.3 Calcium sulphate

Calcium sulphate occurs in the mineral forms of anhydrite ( $\text{CaSO}_4$ ), hemihydrate ( $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ ) and gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), but only the last one crystallises under ordinary conditions [3], including the three drying regimes. Sodium chloride also crystallises as a single phase, halite, and thus is a good proxy for evaluating gypsum crystallisation behaviour. The surface appearance and DR trend illustrate that the two minerals show contrasting crystallisation behaviour. Halite forms extensive deposits and the NaCl setups exhibit DR similar to that of water setups, while gypsum setups display a DR drop without or with scarce efflorescence formation. This DR drop is followed by a low and stable drying phase irrespective of the drying conditions, as shown in Figures 2-4. Two particular samples show a fast drying behaviour, each under either the accelerated or cold conditions which, unlike others, show some gypsum crystallisation at the surface. Table 4 shows characteristic data derived from gypsum DR plots, comparing the DR before (Initial DR) and after the transition to the low DR phase (Transition DR), and the time and amount of gypsum solution needed to accomplish this transition.

**Table 4:** Comparison of characteristic data derived from DR plots for gypsum setups under various drying conditions.

| Drying regime | Initial DR<br>[ $\text{kg} \cdot \text{m}^2/\text{day}$ ] | Time<br>[days] | Transition DR<br>[ $\text{kg} \cdot \text{m}^2/\text{day}$ ] | Gypsum solution<br>[ $\text{kg}/\text{m}^2$ ] |
|---------------|---|----------------|--|---|
| Accelerated   | $40.5 \pm 3.7$  | $6 \pm 4^*$    | $2.0 \pm 1.3^*$  | $32 \pm 8^*$                                  |
| Laboratory    | $2.7 \pm 0.1$   | $52 \pm 12$    | $0.3 \pm 0.0$  | $57 \pm 10$                                   |
| Cold          | $2.5 \pm 0.7$   | $60 \pm 0^*$   | $0.3 \pm 0.1^*$  | $61 \pm 8^*$                                  |

\* A single particularly elevated DR curve is excluded for this estimation

The initial DR is considerably higher under the accelerated conditions, compared to the laboratory and cold ones. Nevertheless, the beneficial effect of the fast drying is abruptly suppressed, as the DR drops by a factor of 20 after about six days. The same effect is observed under

laboratory and cold conditions, though it is taking place less rapidly and less extensively. Under both drying regimes the transition is accomplished after about 60 days, yielding a DR decrease by a factor of nine. This is also associated with a two times higher amount of gypsum solution evaporated during the slow transition, than under the accelerated conditions. A higher amount of gypsum can be hence accumulated during a slow process, before the DR drop is accomplished. This indicates that the amount of accumulated gypsum is not directly related to the DR drop. Instead, its distribution in the pore structure likely has a more profound effect on the permeability decrease.

The DR drop occurs for all gypsum setups, thus is not related to a specific drying regime. It is then rather the consequence of an intrinsic gypsum property, an apparent tendency for crystallisation under the surface, even under efflorescence-favourable conditions. This observation seems at odds with the commonly observed GE phenomenon, but is in line with some literature reports [4, 5]. Calcium sulphate is a ubiquitous component of various building materials, but gypsum deposits are rarely reported to accumulate on their surface, apart from the recent occurrence of GE on masonry and crusts formed via the interaction with atmosphere. This rare occurrence might therefore be a consequence of both its tendency to crystallise below the surface and its limited solubility.

#### 4.3.4 The factors determining crystallisation location

The location of the crystallisation depends on the complex interplay between many factors like properties of supersaturated solution, drying conditions, matrix porosity, and crystal growth pattern. The first parameter is of importance for highly soluble salts of which supersaturated solutions may show significantly altered viscosity and surface tension. Nevertheless, it does not seem significant for scarcely soluble gypsum. We have demonstrated that, on one hand, gypsum tendency for subflorescence formation is not directly related to the drying rate. On the other hand, formation of halite and intermediate mirabilite efflorescence shows that the brick porous matrix is not a barrier itself for crystallisation at the surface. It appears hence that the propensity for efflorescence formation may be mineral-specific, as shown on the example of efflorescing halite and mirabilite and subflorescing thenardite and gypsum. It is not clear though, whether the origin of this behaviour is specific crystal growth or other mineral related parameter.

#### 4.4 Accelerated efflorescence formation

The difference in the drying conditions between the laboratory and accelerated setups translates into a 16 times higher initial DR for the water samples (see Table 3). The vapour pressure difference between surface and environment, under the 'accelerated' and 'laboratory' conditions is 4500 Pa and 1200 Pa, respectively, thus they do only partly contribute to such a high difference in the DR. It is hence the higher air flow in the climate chamber which enhances this effect. The effect of fast drying

conditions on the efflorescence formation is evident for the NaCl setups, resulting in a rapid formation of an extensive deposit under the accelerated conditions and reflected by a high DR similar to this of the water sample (Figure 2). Under accelerated conditions efflorescence is initiated very early, after only two days, while it takes more than 30 days under laboratory conditions. In both cases, deposition continues over time.

The long delay under the laboratory conditions may be explained by a high supersaturation necessary for triggering NaCl crystallisation, which is achieved much slower under these conditions. The slower evaporation rate may also facilitate the diffusion process to counteract advection, further delaying supersaturation and NaCl precipitation. A similar argument can be made for the Na<sub>2</sub>SO<sub>4</sub> setups under the laboratory conditions.

Gypsum setups under all the 3 drying regimes and Na<sub>2</sub>SO<sub>4</sub> setups at the accelerated conditions behave differently than NaCl ones, the effect of the fast drying conditions is quenched by the pore clogging effect of gypsum and thenardite respectively, markedly reducing the DR (see Figures 2-4).

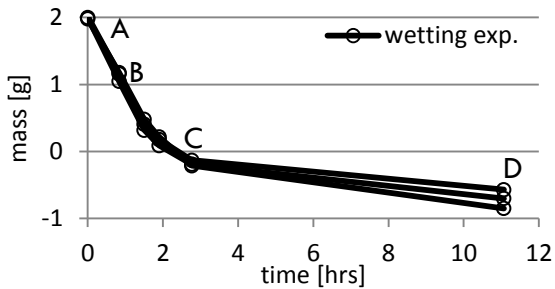
The acceleration of efflorescence formation by applying more severe drying regime is consequently mineral dependent. It can be easily realised for halite, whereas the pore-clogging effect for gypsum and direct thenardite crystallisation inhibits efflorescence. Consequently, it is likely that sodium sulphate efflorescence formation may be accelerated by carrying the experiment just below the thenardite-mirabilite transition temperature of 32.4 °C.

#### **4.5 The effect of wetting-wicking cycles**

The application of wetting cycles resulted in a significant gypsum efflorescence formation. This was not achieved for the basic wick test, irrespectively of the drying conditions (see Table 2). We have then followed more closely the drying behaviour during the wetting phase of an 11 days old sample, subjected already to 18 wetting episodes.

Figure 7 shows the evolution of the setup mass in function of time, from the moment when 2 g of water is placed over the sample surface (point A). The water gets absorbed slowly: it was observed that the sample surface turned dry only at the point B, corresponding to 1 g of absorbed water. Therefore during the phase A-B 1 g of added water has evaporated, and 1 g was absorbed. The phase B-C corresponds to the evaporation of 1 g water, absorbed and supplied to the surface by wick action. From the C point onwards all wetting water has evaporated and gypsum solution is brought to the surface followed by evaporation of water from the solution. From the graph it is evident that the DR during A-B is similar to B-C, while the C-D is characterised by a very low DR. The C-D low DR is related to the pore clogging caused by crystallisation upon evaporation of water from the gypsum solution. This effect can be eliminated by wetting the sample surface, which dissolves the pore plugs. The similar magnitude of the

water-wick DR (B-C) and the free water surface drying rate (A-B) indicates that the drying process is no longer constrained by pore blockage.



**Figure 7:** Evolution of mass of the gypsum setups after wetting with 2g of water.

During the 11 days preceding the experiment, water from about 30 g of gypsum solution has evaporated via the top sample surface leaving gypsum behind. For the wetting experiment only 2 g of water is used, which is then able to dissolve only a small fraction of the accumulated gypsum. Nonetheless, it effectively restores the high DR, even though much gypsum is still present in the sample pores. It is plausible then that most of the accumulated gypsum narrows the pores, without completely closing them, allowing for a persistent high DR (phase B-C). The DR decrease is then caused by an increasing number of pores becoming completely blocked by gypsum-plugs. Once a pore is clogged, no further gypsum accumulation can take place in an isolated pore, what makes the pore plugs relatively fine and susceptible to dissolution even with a low amount of wetting water.

The basic gypsum wick test demonstrates that, although a low amount of gypsum may initially crystallise at the surface, its further growth is quickly inhibited by pore clogging. However, these short episodes of surface accumulation may be repeated by wetting cycles, reopening the pores and progressively leading to efflorescence formation.

Even though wetting appears to contribute remarkably to GE formation, it does not explain its only recent occurrence. Nevertheless, implementation of the wetting cycles allows for further investigation of the problem. The factor which has actually led to GE triggering may be either source or process related. Considering the former, it appears that there were no recent changes leading to an increased availability of sulphates in either brick or cement. However, the recent GE occurrence might be associated with significant changes in mortar quality, particularly in recent application of various surfactant-based admixtures. There are premises showing an important role of such compounds in enhancing gypsum crystallisation on masonry [1]. However, the admixtures potential for exerting a long time effect in real masonry systems remains questionable.

## 5 Conclusions

However exploratory, this study offers some insights into elucidating the salt's efflorescing tendency. The results obtained from a series of wick experiments allow formulating the following conclusions:

- 1) Salt's efflorescing tendency appears to be mineral specific: halite (NaCl) and mirabilite ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) tend to form extensive efflorescence while gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) and thenardite ( $\text{Na}_2\text{SO}_4$  (V)) accumulate under the surface. The former is accompanied by a high drying rate, sustaining salts' growth at the surface. The latter results in pore clogging and consequently a major drying rate drop, reducing efflorescence development.
- 2) Under wick test conditions only halite efflorescence formation can be accelerated by applying more severe drying regime. It is likely that the same holds for direct crystallisation of mirabilite, hence for temperatures below the thenardite-mirabilite transition. For gypsum and thenardite the effect of severe drying conditions is quenched by the salt's pore clogging tendency.
- 3) A standard wick test is not effective in simulating efflorescence formation when minerals show a pore clogging tendency. However, it was possible to simulate gypsum efflorescence formation by implementing frequent surface wetting into the standard procedure. This allows the re-opening of plugged pores and progressive events of gypsum crystallisation at the surface, resulting in significant efflorescence development. The improved procedure is consistent with frequent masonry wetting under field conditions.

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