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Irreversible dilation of NaCl contaminated lime–cement mortar due to crystallization cycles

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Abstract

The mechanism of damage occurring in NaCl contaminated materials has not been clarified yet. Apart from crystallization pressure, other hypotheses have been proposed to explain the cause of decay. Irreversible dilation has been observed in a few cases but has never been studied in a more systematic way. The aim of the research is to contribute to the modeling of this phenomenon.

In the present paper the effect of NaCl on the hydric and hygric behavior of a lime–cement mortar is extensively studied. The results indicate that NaCl influences the hydric and hygric dilation behavior of the material. The material contaminated with NaCl shrinks during dissolution and dilates during crystallization of the salt. This dilation is irreversible and sufficient to damage the material after few dissolution/crystallization cycles. This behavior is not restricted to NaCl, but is observed in the presence of other salts as well ($NaNO_3$ and KCl). Outcomes of electron microscopy studies suggest that salts causing irreversible dilation tend to crystallize as layers on the pore wall.

Keywords: Mortar; Chloride; Degradation; Expansion; Hygric behavior

1. Introduction

The damage mechanism due to NaCl has not been unequivocally explained yet. According to the most accepted theory, salt crystallization damage [1–3] occurs because salt fills the pores and creates pressure on the pores' walls. This pressure is proportional to the supersaturation ratio. In an equilibrium situation the supersaturation that can be reached depends on the pore radius: the smaller the pores the higher the supersaturation. Following this theory, in order to produce crystallization pressures high enough to cause damage, NaCl should crystallize at high supersaturation filling pore sizes in the range of a few nanometers [4,5].

Some objections have been raised to these theoretical conclusions: NaCl has a low tendency to supersaturate, therefore would hardly produce high crystallization pressures [6]. Besides, most traditional building materials do not have pores in the nanometer range but show, nevertheless, serious damage in the presence of NaCl.

The reported discussion suggests that damage due to NaCl may not only be related to crystallization pressure as described in literature starting with Correns' work, but involves other mechanisms. An important reason that hampers the clarification of the damage mechanism is the lack of systematic experimental investigations of the phenomenon of irreversible dilation in the presence of salts. Up to now, only few experiments have been carried out on a restricted number of materials (old bricks [7] and sandstone containing clay [8,9]). The research reported in this paper aimed at studying the dilation phenomenon in detail and verifying it for different salt types. The research was carried out in steps. Firstly the effect of NaCl on the hydric and hygric dilation behavior was investigated. Secondly, to check if the observed behavior was limited to NaCl or common to more salts, the behavior of two more salts, $NaNO_3$ and KCl, was studied. These two salts were selected because they are both hygroscopic, they do not have

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Table 1
Open porosity lime–cement mortar as determined by immersion (according to [11]) and by Mercury Intrusion Porosimetry (MIP); Water Absorption Coefficient (WAC) determined according to [12]

Dry density (g/cm^{3}0	By immersion	1.795
	By MIP	1.888
Porosity (V/V%)	By immersion	32.3
	By MIP	27.6
WAC (kg/m^2/h)		0.064

hydrated forms and each of them has one ion in common with NaCl. Finally, the effect on the dilation behavior of a crystallization inhibitor, modifying the crystallization habits of the salt, was investigated. All experiments were performed on a lime–cement mortar.

2. Materials and methods

2.1. Material properties

A lime–cement (lime/cement/siliceous sand = 1:4:20 in volume) mortar has been selected for this study. This mortar is extremely susceptible to sodium chloride damage [10].

The physical properties of the hardened mortar were determined on 5 × 5 × 2 cm specimens and are summarized in Table 1. The pore size distribution was determined by Mercury Intrusion Porosimeter (MIP) (Fig. 1). The mortar has a high porosity mainly constituted by large (4–7 μm) and very large (>80 μm) pores. The presence of very large pores, not measured by MIP, may explain the difference in the open porosity measured by immersion and by MIP respectively.

The material has only a limited number of pores in the hygroscopic range (<0.1 μm).

2.2. Preparation and salt contamination of the specimens

Specimens with a size of 20 × 10 × 2 cm were used for the study of the hydric and hygric behavior described in the following chapters. Since the physical properties of the mortar are strongly influenced by the suction of the substrate on which it is applied [13], the plaster slabs were not prepared in a mould but on top of a brick substrate from which they were detached after 1 day. A filter paper was used between substrate and plaster to facilitate the detachment. The specimens were cured under plastic sheets for 24 h; then they were detached from the substrate and cured for 15 days at 20 °C 65% RH to guarantee the hardening of the lime binder and allow carbonation. Carbonation was obtained by storing the specimens for 2 weeks in a cabinet with 3% CO_2. After this period the complete carbonation of the mortar was verified by spraying phenolphthalein on a broken section.

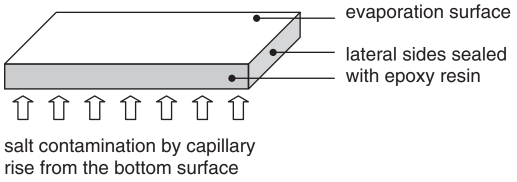

Fig. 2. Procedure followed for the salt contamination of the mortar slabs.

Before measuring the hydric and hygric behavior the specimens were contaminated with NaCl according to the following procedure (Fig. 2): the mortar slabs were sealed on the four lateral sides by epoxy resin and an NaCl saturated solution (concentration of ≈6 M) was introduced from the bottom by capillary rise. An amount of solution equal to the Capillary Moisture Content (C.M.C.) of the mortar (i.e. the moisture content enough to wet the upper surface) and leading to 2% m/m of NaCl in the specimen was used. This amount of salt is comparable to the salt load measured in practice and is enough to damage this material in laboratory tests [10]. After contamination, the specimens were closed at the bottom with removable tape and dried at 10% RH and 20 °C until a constant weight was reached. This drying process led to almost no efflorescences; salts accumulated just beneath the evaporation surface of the specimen. When an experiment was performed with a crystallization inhibitor the specimens were dried at 40 °C to prevent efflorescence.

Once the specimens contaminated with salt were dried, metal supports for the Linear Variable Differential Transformer (LVDT) to be used in the measurement of the hydric and hygric

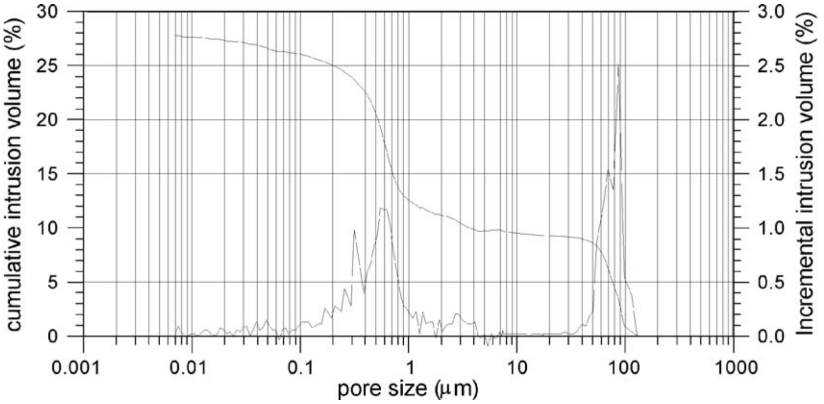

Fig. 1. Total porosity and pore size distribution of the lime–cement mortar as measured by Mercury Intrusion Porosimeter.

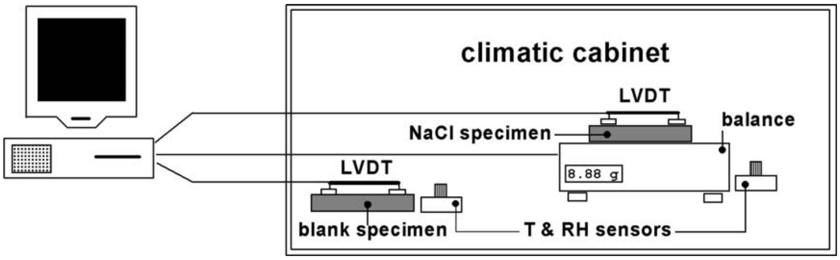

Fig. 3. Test set up: the specimens, on which the LVDT are applied, are placed in a climatic cabinet. A PC collects data on the dilation and on the weight changes. Temperature and RH sensors are used for additional checking of the environmental conditions.

behavior were glued, at a distance of 150 mm from each other, on the surface of the mortar slabs by a two component glue.

2.3. Test set up

The hydric and the hygric behavior of the specimens were monitored inside a climatic cabinet in which the temperature and RH of the air can be programmed and controlled. In order to reach a very low RH, dry air was blown in the cabinet. The measuring apparatus (Fig. 3) was composed by:

- A balance with an accuracy of 0.1 g, connected to a PC, for monitoring the variation in specimen weight due to hygroscopic moisture uptake and release,
- LVDT fixed to the supports glued on the mortar and connected, through the data acquisition system Labview, to a PC,
- Two data loggers (ESCORT) for double checking of the temperature and RH of the air in the climatic cabinet.

3. Results

3.1. Hydric behavior of NaCl contaminated specimens

The hydric behavior was studied both on NaCl contaminated and blank mortar slabs. The specimens were set in the climatic cabinet at 20 °C and 50% RH and they were allowed to absorb water by capillary rise from the bottom for a period of 2 days. Then the bottom of a specimen was sealed with tape, the RH in the cabinet lowered to 0% RH and the drying started. After about 4 weeks, the wet–dry cycle was repeated.

Data on the temperature and RH of the air, as well as the dimensional changes of the specimens at its surface, were collected continuously. Fig. 4 shows the results of the experiment. During wetting, both the salt contaminated and the reference specimens have a similar behavior, consisting of initial shrinkage (due to the wetting of the lower part of the sample) followed by dilation (corresponding to the migration of water to the surface of the sample). During drying, the blank specimen shrinks, while the salted specimen, after a period of about 4 days in which it does not show any change in size, starts dilating. It can be reasonably supposed that dilation occurs when, due to evaporation of water, the salt starts crystallizing. The maximum dilation is about 0.13 μm/mm and is reached in 5 days. During the re-wetting both specimens dilate, but the dilation is larger in the salt contaminated than in the blank specimen. During drying the blank specimen shrinks while the salted specimen dilates further.

This experiment shows that the hydric behavior of this material is strongly influenced by the presence of sodium chloride. The salt contaminated specimen undergoes an irreversible dilation; on the contrary, the displacements of the blank specimen, apart from being smaller, are reversible.

In the described experiment the effect of dissolution and crystallization of salt on the shrinkage/dilation of the material cannot be followed in detail. In fact the amount of absorbed and evaporated water cannot be measured without handling of

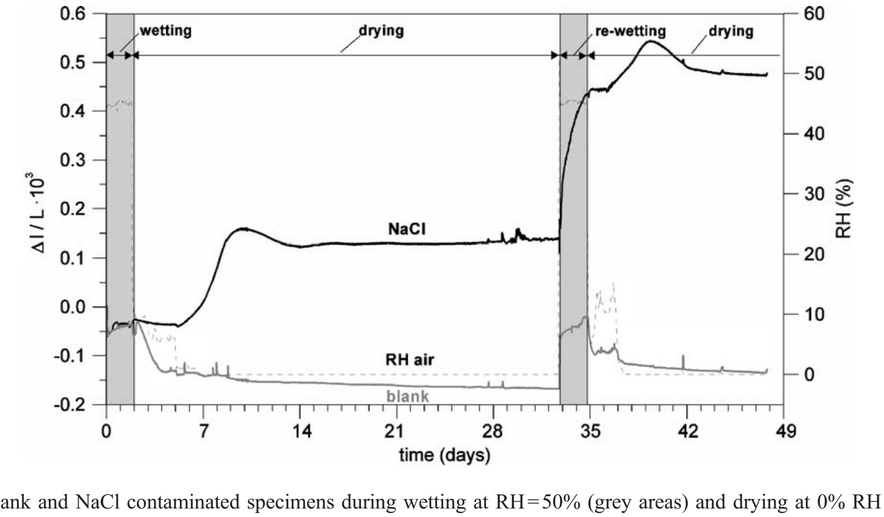

Fig. 4. Dilation of blank and NaCl contaminated specimens during wetting at RH = 50% (grey areas) and drying at 0% RH (white area) cycles.

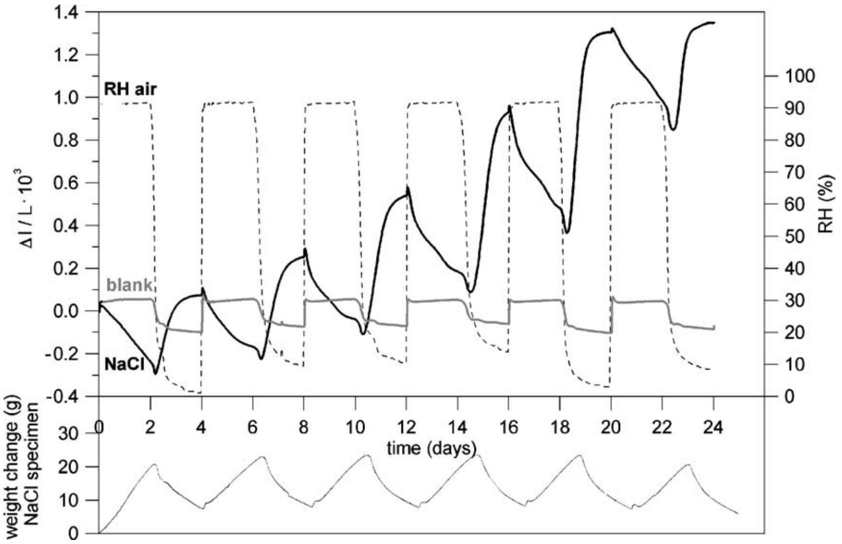

Fig. 5. Dilation of blank and NaCl contaminated specimens (upper graph) and weight change of NaCl contaminated specimen (lower graph) during RH cycles.

the specimen; besides it is not known at which moment water, absorbed by capillarity from the bottom of the specimen, reaches the surface where most of the salt accumulates. For a better understanding of the dilation phenomenon, a slower and more controlled moisture absorption and release is necessary. The use of RH changes through the RH of crystallization of NaCl ($RH_{cryst} = 75\%$) allows to reach this aim. In the following section the study of the effect of RH changes on the dilation of salt contaminated specimens is described.

3.2. Hygric behavior of NaCl contaminated specimens

The hygric behavior of NaCl contaminated and blank (reference) mortar slabs was measured. The specimens, prepared as described in Section 2.2, were placed in the climatic cabinet. The temperature was maintained constant at 20 °C and the RH was varied between 10% ± 2% RH and 92% RH every 48 h. Six RH cycles were performed. During the test the NaCl contaminated specimen was put on a balance and its weight was continuously monitored. The results, reported in Fig. 5, show that the blank specimen dilates during the high RH period and shrinks during the low RH period as expected [14,15], while the NaCl contaminated specimen behaves in the opposite way. The dimensional changes of the salt contaminated specimen are not only of opposite sign but also much larger than in the case of the blank specimen. When the cycles are repeated, dilation increases while shrinkage remains about the same: this leads to an increasing irreversible dilation that

Table 2
Hygric dilation in material contaminated with NaCl

Material	Hygric (RH changes)
Lime–cement mortar without NaCl (this study)	0.13 μm/mm (5–90% RH)
Lime–cement mortar in the presence of NaCl (this study)	1.3 μm/mm (after 6 cycles 5–90% RH) 0.3 μm/mm (after 1 cycle 5–90% RH)
Sandstone with clay without NaCl [8,9]	0.2 μm/mm (35–90% RH) (after 1 or 6 cycles)
Sandstone with clay in the presence of NaCl [8,9]	0.5 μm/mm (after 6 cycles 35–90% RH)
Old brick in the presence of NaCl [7]	0.3–4 μm/mm (after 9 cycles 40–85% RH)
New brick in the presence of NaCl [7]	0.1–1 μm/mm (after 9 cycles 40–85% RH)

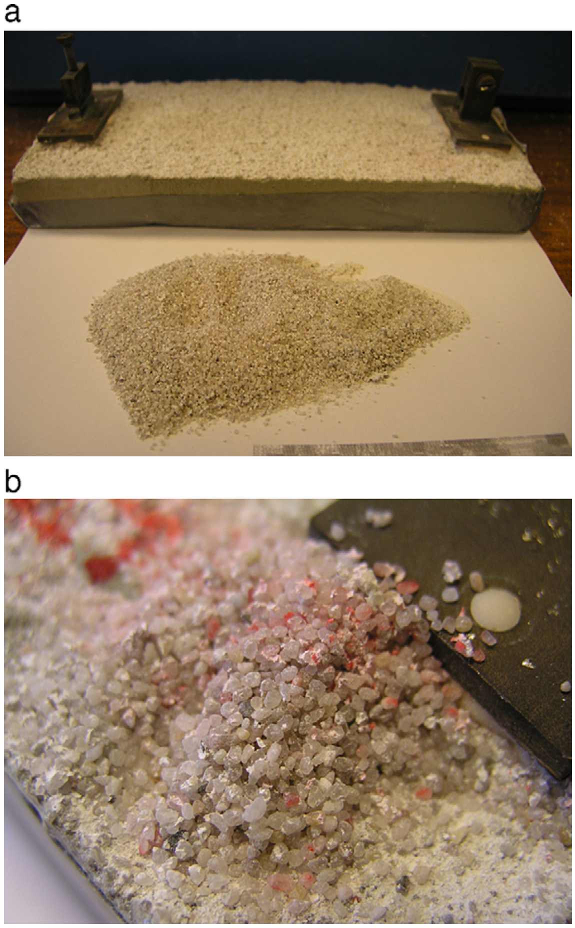

Fig. 6. (a–b) Sanding of the outer layer of the lime–cement mortar in the presence of NaCl after 6 RH cycles.

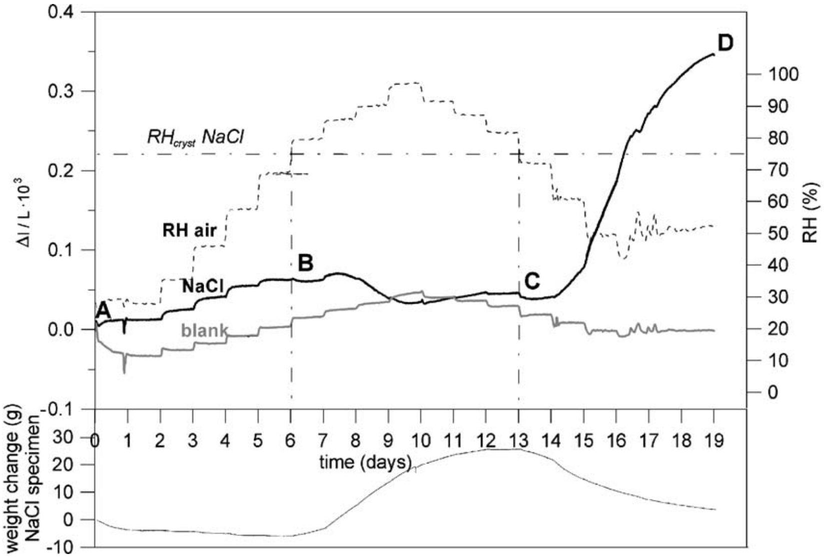

Fig. 7. Dilation of blank and NaCl contaminated specimens (upper graph) and weight change of NaCl contaminated specimen (lower graph) during a single RH cycle (max. RH 95%).

reaches, at the end of the sixth cycle, 1.3 μm/mm. On the contrary, the dimensional changes are completely reversible in the case of the blank specimen.

The hygric dilation measured in the NaCl contaminated specimen is considerable, as shown by a comparison with the results obtained in similar experiments performed on other materials and reported in literature (Table 2). The dilation leads to a stress that is likely to exceed the low tensile strength of the plaster and in fact results, at the end of the test, in serious damage. The damage consists in sanding of the outer layer of the plaster (Fig. 6a–b); the material loss is about 7% of the initial weight of the specimen. Sanding occurred only in the areas of the specimen where adsorption and evaporation took place. Under the metal plates on which the LVDT were fixed, no evaporation took place and consequently no damage. At the end of the experiment, the good adhesion of the plates to the mortar was verified: some strength was necessary to detach the plates from the mortar.

The serious damage observed in the mortar slab after the test stresses the importance of RH changes for the development of the salt decay in weak lime–cement mortars. Our results are in accordance with the data reported in literature for clay containing sandstone and old brick [7–9]. Moreover, the experiments on the lime–cement mortar allow to definitely assert that the presence of reactive clay is not a necessary factor for the occurrence of irreversible dilation in NaCl contaminated materials.

In order to move a step forward in the understanding of the mechanism, more detailed experiments were necessary. Performing an RH cycle slowly and with small increments would allow identifying the point at which dilation and shrinkage occur and relate them unequivocally to either salt crystallization or dissolution. A new experiment was set up in

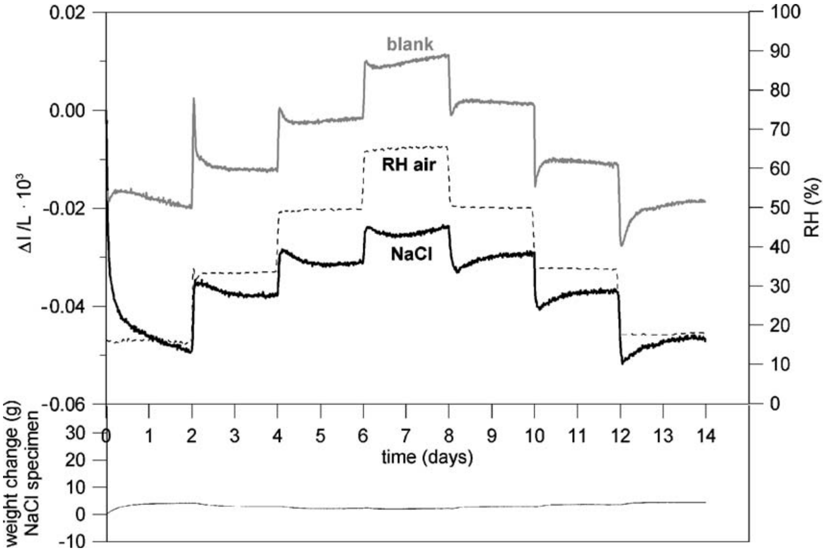

Fig. 8. Dilation of blank and NaCl contaminated specimens (upper graph) and weight change of NaCl contaminated specimen (lower graph) during an RH cycle below 75% RH.

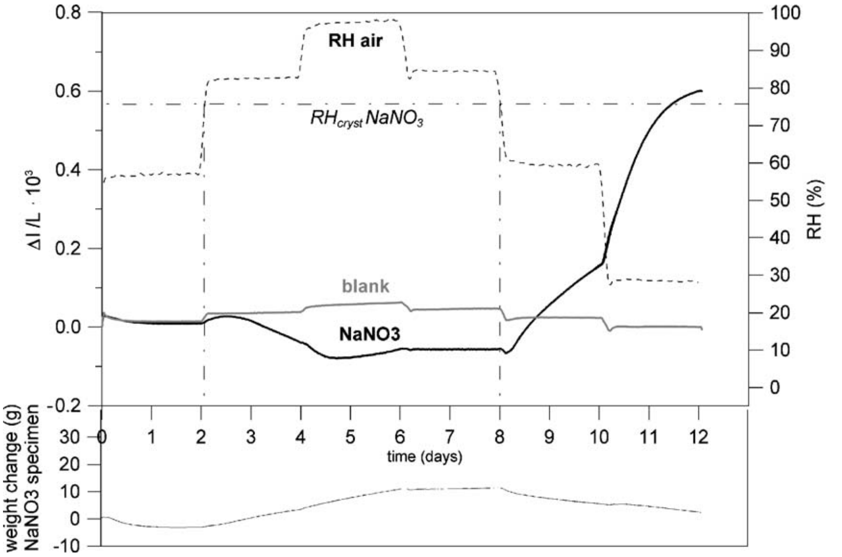

Fig. 9. Dilation of blank and $NaNO_3$ contaminated specimens (upper graph) and change in weight of the $NaNO_3$ contaminated specimen (lower graph) during an RH cycle.

which the RH was varied stepwise between 30% and 96%. Each step in RH was done in 1 h and was followed by 23 h of equilibration at constant RH. The experimental data (Fig. 7) show clearly that the blank specimen dilates while the RH increases and shrinks while the RH decreases. On the other hand the NaCl contaminated specimen shrinks at RH higher than 75% and considerably dilates when the RH drops below the 75%. Note that in the presence of NaCl water condenses when RH > 75%, while in the absence of NaCl it starts to condense at RH ~ 100%.

Three parts can be distinguished in the curves:

(1) A–B: increasing RH (30–75% RH). In this range of RH the behavior of the blank and of the NaCl contaminated specimens is similar: both dilate when the RH increases. The dilation is due to water vapor adsorption [14,15].

(2) B–C: increasing and decreasing the RH while RH > 75% of NaCl (75–96–75% RH) After crossing 75%, the behavior of the NaCl contaminated specimen starts to deviate from the specimen without salt: as soon as liquid water is present in the plaster (as shown by the weight change) and the salt starts dissolving, shrinkage is measured. The shrinkage of the salt contaminated specimen can be explained by the fact that the tensions, developed during the crystallization of the salt during the initial contamination of the specimen, are released when the salt dissolves.

(3) C–D: decreasing the RH (75%–45% RH) When the RH drops below 75%, the salt contaminated specimen starts drying (as shown by the weight loss) and dilates considerably. The curve becomes steeper with decreasing RH. At the end of the test, after 4 days of drying, the

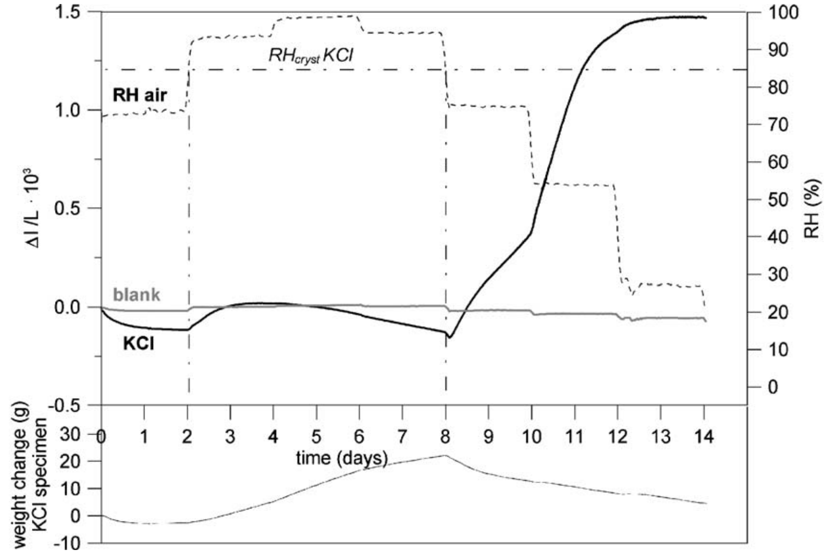

Fig. 10. Dilation of blank and KCl contaminated specimens (upper graph) and change in weight of the KCl contaminated specimen (lower graph) during an RH cycle.

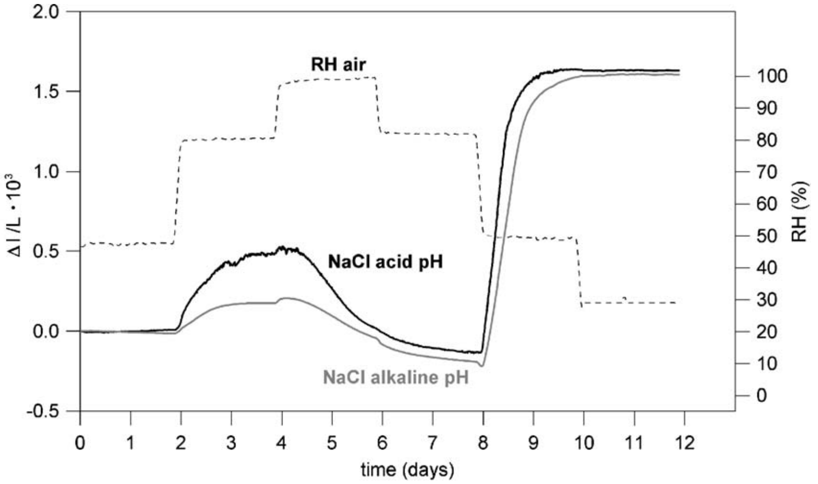

Fig. 11. Dilation of specimens contaminated with acid and alkaline NaCl solution during an RH cycle.

dilation measured is about 0.35 μm/mm. The uncontaminated specimen shrinks, as expected, when the RH is lowered; at the end of the cycle it comes back to the original size.

A further proof that the behavior of a salt contaminated specimen is modified only if the equilibrium RH of the salt is exceeded is given by another experiment in which the RH was varied but kept below 75% (Fig. 8): in this case no irreversible displacement took place.

3.3. $NaNO_3$ and KCl

The above-described experiments proved that NaCl is able to produce irreversible dilation during crystallization. To check whether this behavior is specific for NaCl or occurs also in the presence of other salts further experiments were set up.

Two hygroscopic salts were selected having respectively the cation or the anion in common with NaCl and no hydrated forms: KCl and $NaNO_3$. The specimens were prepared in the same way as for NaCl. The same amount of solution was introduced in the mortar. The concentration of the solution was defined in such a way as to reach in all cases a salt content of 2% m/m of the weight of the dry specimen. As shown in Figs. 9 and 10, in both cases shrinkage is observed when the RH exceeds the values where the salts dissolve in the adsorbed water (at 20 °C this happens at 85% and 75% for KCl and $NaNO_3$, respectively [14]). When the RH decreases below these values the specimens start drying, the salts crystallize and dilation occurs. Therefore it can be concluded that this behavior is not typical for NaCl but is a more general phenomenon.

3.4. Acid and alkaline pH

The possible effect of acidity or alkalinity of the NaCl solution on the hygric dilation has been investigated by contaminating two specimens with NaCl solutions having a pH of respectively 1.5 and 14. The response of the two

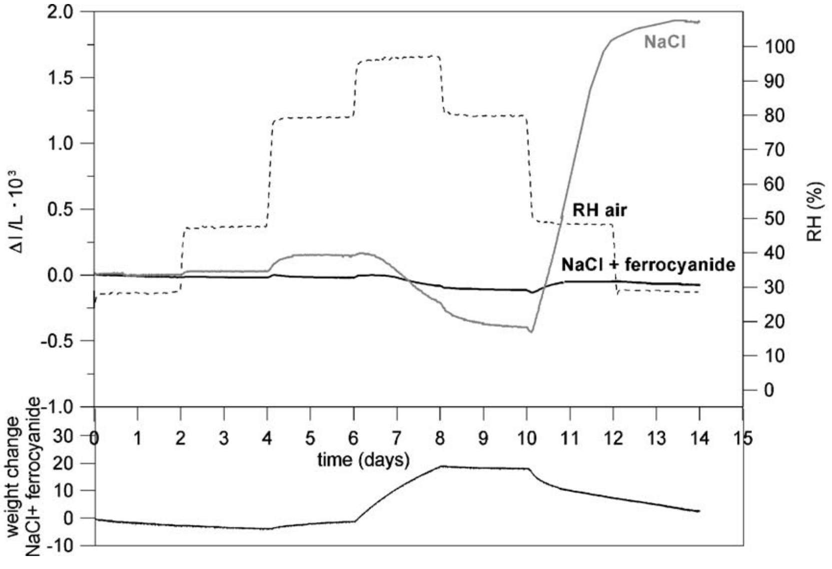

Fig. 12. Dilation of NaCl contaminated specimen with and without inhibitor (upper graph) and weight change of NaCl contaminated specimen with ferrocyanide (lower graph) during an RH cycle.

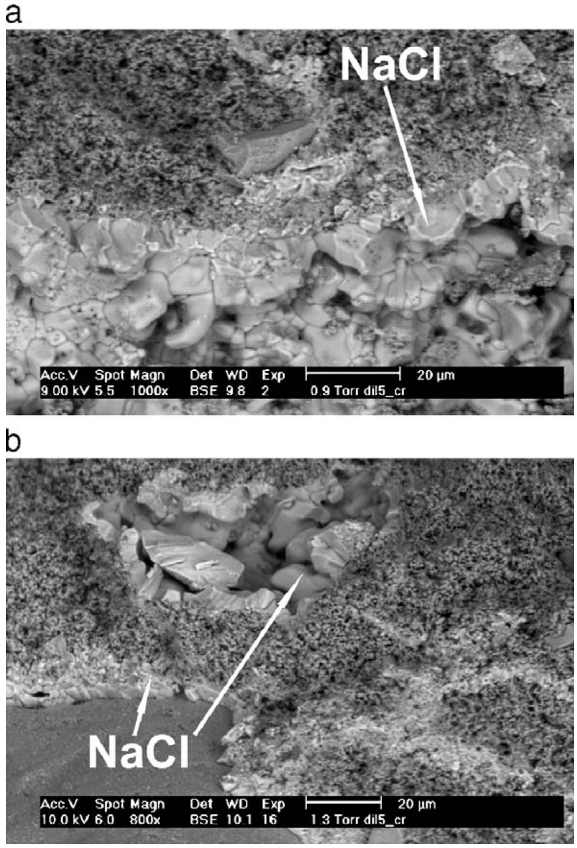

Fig. 13. (a–b) ESEM photomicrographs showing NaCl (lighter areas) crystallizing as a layer over the pore walls of the binder.

specimens to the RH changes is similar and comparable to the one measured on NaCl contaminated specimens: shrinkage is measured during dissolution and expansion during drying (Fig. 11). Therefore we conclude that the pH does not play an important role in the studied mechanism. It should also be mentioned that the acid pH is probably neutralized in the alkaline environment of the mortar.

3.5. NaCl with crystallization inhibitor

The use of crystallization inhibitors may also help answer some questions about the damage mechanisms since they are supposed to increase the supersaturation level (and therefore the theoretical crystallization pressure) at which salt crystallization occurs and modifies the crystal habits [15]. Sodium ferrocyanide ($Na_4Fe(CN)_6 \cdot 10H_2O$) has been chosen since it has proven to be very effective in inhibiting NaCl crystallization [15].

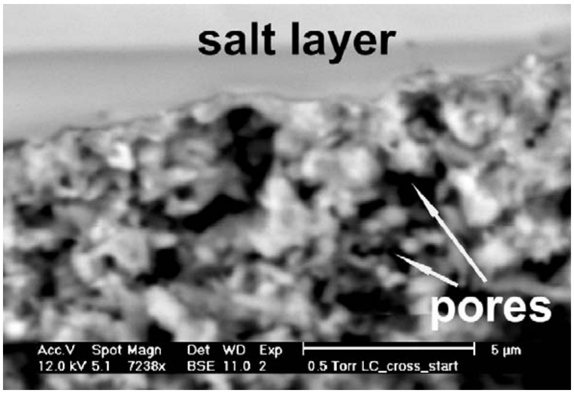

Fig. 14. ESEM photomicrograph showing that, under the salt layer (white area), the pores of the material are not filled with salt.

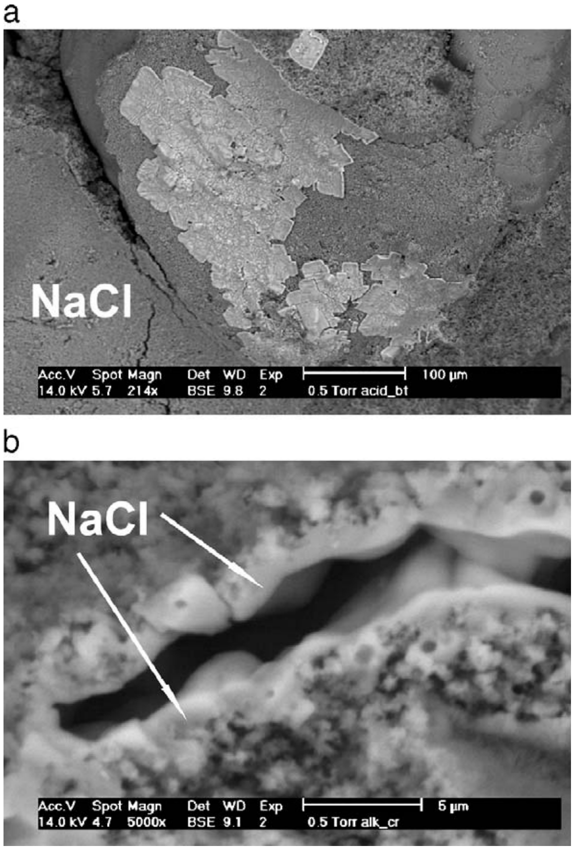

Fig. 15. (a–b) ESEM photomicrographs showing NaCl (lighter areas) crystallizing as a layer in specimens contaminated with acid (a) and alkaline (b) NaCl solution.

Two mortar specimens were contaminated with an NaCl saturated solution with 0.1% (m/m) and without sodium ferrocyanide, $Na_4Fe(CN)_6 \cdot 10H_2O$. In both specimens the same amount of salt was introduced.

The specimens were then placed in the climatic cabinet and the RH was varied step by step between 30 and 98% RH. In

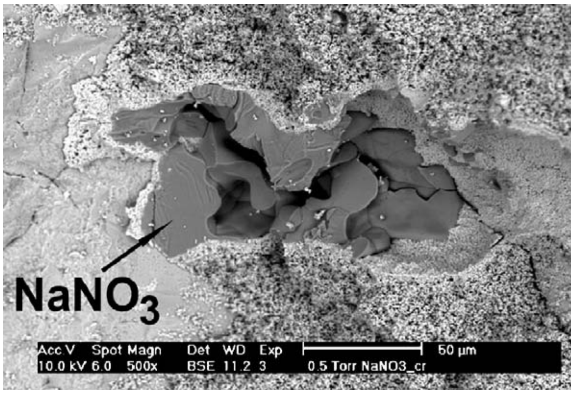

Fig. 16. ESEM photomicrograph showing $NaNO_3$ (dark-grey area) crystallizing as a layer on the pore wall.

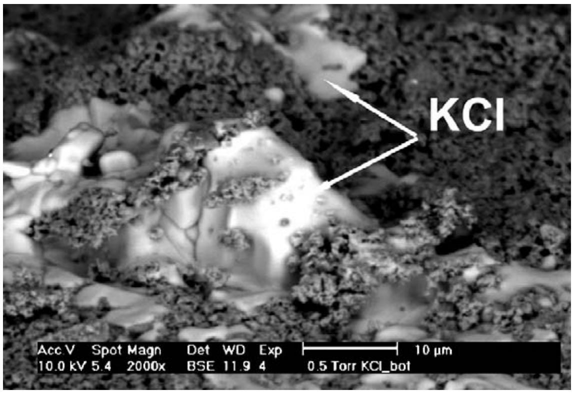

Fig. 17. ESEM photomicrograph showing KCl (white area) crystallizing as a layer over the binder.

Fig. 12 the obtained dilations are reported. The specimen contaminated with NaCl solution shows significant dilation during drying, whereas the specimen with NaCl plus inhibitor displays almost no change in size. The almost complete drying out (and therefore the necessary occurrence of salt crystallization) of the specimen containing the inhibitor can be checked by looking at its weight at the end of the experiment.

Experiments in which more RH cycles are performed are planned in order to study the long-term effect and the consequences of the presence of the inhibitor on the damage.

4. ESEM observations

Environmental Scanning Electron Microscopy (ESEM) studies have been performed on the specimens subjected to RH cycles, after their complete drying.

The cross sections of the outer layer (about 3 mm thick) of mortars contaminated with NaCl, $NaNO_3$, KCl and NaCl plus inhibitor were observed using a Back Scattered Electron (BSE) detector. The samples to be observed in the ESEM were obtained either by breaking or cutting the mortar slabs, without the use water in order to avoid any dissolution of the salt. The pressure used in the ESEM chamber varied between 0.3 and 1.9 Torr at the temperature of about 20 °C, corresponding to 1.7 up to 10.9 RH. The composition of the salt crystals was checked with Energy Dispersive Spectroscopy X-ray Microanalysis (EDX). The investigations aimed to study the location and the habits of salt crystallization in pores.

In the ESEM picture of the lime–cement mortar contaminated with NaCl, the salt is visible in larger pores, creating a layer over the pore walls (Fig. 13a–b). It looks as if a strong interaction exists between the material and the salt crystals. This is in accordance with the observations reported in other studies [16]. Under the salt layer, most of the distinguishable pores appear to be empty (Fig. 14). It should be mentioned that the possibility that smaller pores, not detected by the ESEM, are filled with salt cannot be excluded.

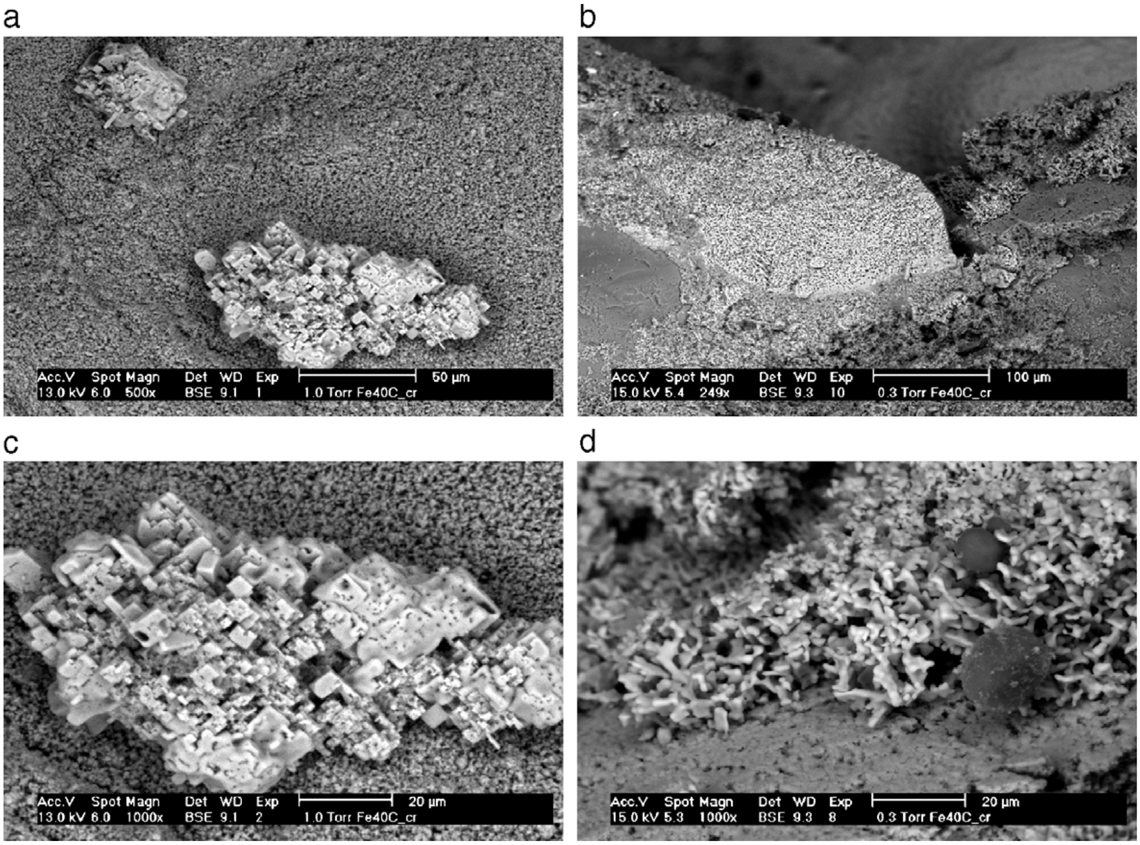

Fig. 18. (a–d) ESEM photomicrograph showing NaCl crystallization in the presence of inhibitor; crystal agglomerations (white areas) are observed (a–b); the crystals may be more (c) or less regular (d).

The presence of acid or alkaline pH does not affect the crystallization habit of sodium chloride (Fig. 15a–b) in the studied lime–cement mortar.

Crystallization habits similar to the one observed for NaCl were found also in the case of $NaNO_3$ (Fig. 16) and KCl (Fig. 17).

In case the crystallization inhibitor was added to the salt solution a different crystallization habit of the NaCl was observed. The salt does not show anymore a strong affinity with the substrate: it does not cover the pore walls in the form of a layer, but crystallizes mainly as agglomerations of crystals, not strongly attached to the material. The salt is not homogeneously distributed in the contaminated area but accumulates in definite zones (Fig. 18a–d).

5. Discussion and conclusions

Our experiments unambiguously prove that NaCl is able to modify the hydric and hygric behavior and the related dilation of a material completely, even in the absence of reactive clay. In the lime–cement mortar NaCl produces dilation during the drying phase of the RH cycle, when the salt crystallizes. The dilation observed in the NaCl contaminated specimens is irreversible and increases with repeated RH cycles until damage occurs. Damage appears as sanding of the outer layer of the specimen where most of the salt has accumulated. It has been proven that this dilation is due to crystallization since it only occurs in the presence of salts, when the RH of the air is low enough to drive the evaporation of water.

The same effect of salt has also been observed for other hygroscopic salts ($NaNO_3$ and KCl), showing that this phenomenon is not restricted to NaCl but is more general.

The presence of a crystallization inhibitor also modifies the hygric behavior of NaCl contaminated specimens: no more relevant dilation is observed during drying and crystallization.

The ESEM investigations have shown that NaCl, as well as $NaNO_3$ and KCl, crystallize as a layer on the pore walls. When a crystallization inhibitor is added to the NaCl solution, the salt crystallizes as crystals not adhering to the pore walls.

Combining the ESEM observations with the dilation occurring during crystallization leads to a theory in which the damage mechanism is based on a mechanical interaction between salt and pore wall [17]. Salts crystallizing as a layer on the pore walls seem to be able to strongly affect the hygric dilation of the material producing relevant expansion during drying of the specimen and crystallization of the salt. It seems that these salt layers are able to transfer stress to the pore walls and thereby causing dilation [7,17]. A similar effect would then not be possible when the same salt crystallizes without adhering to the material, as in the case in which the crystallization inhibitor is added. Further experiments have been set up in order to check this hypothesis.

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