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Salt crystallization damage: how realistic are existing ageing tests?

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Abstract

Salt crystallization is a major cause of damage in porous building materials. Notwithstanding the extensive research in this field, the complexity of the problem has hindered the use of mathematical models for forecasting ageing and damage due to salt crystallization. Nowadays, the durability of materials with respect to salt crystallization is mostly determined by accelerated ageing tests, carried out in laboratory following different test procedures. An effective ageing test should simulate in laboratory, in a reliable way and within a relatively short period of time, the behaviour in practice. The question is whether existing test procedures are able to do so. This paper reports a critical overview of existing procedures and suggests directions for further research.

Keywords: salt crystallization, ageing test, porous building materials, damage.

1 Introduction

Salt crystallization is a major cause of damage in porous building materials. When selecting a stone or stone-like material for building and/or restoration purposes, its resistance to salt crystallization is an important factor influencing the final choice. At present, the ability of a building material to withstand salt crystallization is generally evaluated by subjecting the materials to accelerated salt crystallization tests in laboratory. In fact, the complexity of the salt crystallization process in porous building materials has limited up to now the development and the use of durability estimators and mathematical models for forecasting ageing due to salt crystallization, as done e.g. for other weathering processes (e.g. chloride ingress in concrete).

According to most current models, damage due to salt crystallization occurs when the crystallization pressure developed by salts precipitating in pores overcomes the strength of the material. Crystallization pressure depends on supersaturation of the solution and on pore size and these two factors are interrelated [a.o. 1]. When trying to assess a relation between properties and durability of a material with respect to salt crystallization, parameters as pore size and related properties (e.g. specific surface area. capillary absorption, etc.), being easy to measure, have been used more often than supersaturation in pores. In some cases simple correlations have been proposed between some material properties (e.g. porosity, pore size distribution, water absorption, mechanical strength, swelling clay content) and the damage due to salt crystallization [e.g. 2]. Mostly different parameters have been combined in durability estimators [e.g. 3-5]. Notwithstanding the fact that usually correlations can be identified between these parameters or indicators and the durability of the stone, they are not straightforward nor unique.

Existing mathematical models applied to durability of materials with respect to salt crystallization all starts from the assumptions of the already mentioned salt crystallization theory, without taking into account other possible mechanisms [e.g. 6-8]. Besides, most models focus mainly on the calculation of the development of pressures in singles pores [a.o. 1, 9]. The process of salt solution transport during drying (governing the location of crystallization) and the translation of the crystallization pressure developed in a pore to the damage at the macro scale of the building material are generally not included in the model. Some attempts have been undertaken to come to more complex models considering the different factors governing salt crystallization damage in porous building materials [10-12]. However, the complexity of the process has hindered until now the use of these mathematical models for forecasting the effect of salts crystallization on ageing of materials.

Another approach to the problem has been attempted by developing stochastic models on ageing on the basis of experimental results from laboratory or in situ measurements [13-14]. However, as these models are generally based on a limited set of data (e.g. data from a limited number of

building materials, contaminated with a certain type of salt and under specific environmental exposure conditions), their validity is limited to similar cases only.

The considerations described above, make clear why, still nowadays, the durability of materials with respect to salt crystallization is mostly determined by accelerated ageing tests, carried out in laboratory following different test procedures.

The following sections of this paper report a critical overview of existing procedures and suggest directions for further research.

2 Comparing existing salt crystallization laboratory tests

The first known example of a test procedure for the determination of stone durability with respect to salt crystallization dates back to 1828; the socalled Brard test, published by Héricart de Thury [15] was actually meant for the determination of the frost resistance, but it used a sodium sulfate solution to simulate frost decay. This test was further modified during the 19th century [16]. In the first decades of the 20th century, the idea of classifying stone durability on the basis of salt crystallization tests was proposed [a.o. 17]. A detailed report of the early history of salt crystallization tests can be found in [18-19]. During the 20th and 21st centuries a large number of different salt crystallization procedures has been developed: an extensive overview can be found in [20]. Some of these test procedures have resulted in codes and recommendations, which are generally used for the evaluation of the durability of building materials to salt crystallization.

Most common test procedures for the evaluation the durability of material to salt weathering, reproduce the process of salt solution penetration on one side of the material, transport of the salt solution through the pore network and drying (with consequent salt crystallization) on the opposite side; a process as occurring in practice e.g. in the case of rising damp transporting salts in a wall. Next to these procedures, sea-salt spray test exists too, reproducing the effect of sea-salt spray in buildings in marine environment. This last type of test procedure will not be further discussed in this paper.

A test procedure includes the type of specimens (a.o. single material or combination), the salt type and load, the contamination procedure, the temperature and relative humidity conditions and the assessment method: each of these parameters can differ from one test procedure to another, in one or more details. Hereafter, some commonly used international recommendations for salt crystallization tests (RILEM MS-A.1 [21] and MS-A.2 [22], WTA [23], EN 12370 [24]) are compared with respect to the above mentioned parameters (table 1).

An effective ageing test should simulate in laboratory, in a reliable way and within a short period of time, the behaviour of the investigated material in the field. In order to accelerate the ageing, laboratory tests generally use conditions harsher than in practice. Questions arise as to what extent laboratory conditions need to be similar to those in the field and if the chosen harshest conditions would still give reliable results. In the following sections the effect of the choices for each of the parameters is discussed.

Standard/ recommen dations	Specimen	Salt type	Salt load Wet-dry cycles	Wet- dry cycles	T & RH during drying	Assess- ment method
RILEM MS-A.1	Combined (masonry)	Na₂SO₄ or NaCl	Different Wet-dry cycles	yes	Constant 20 °C 50%RH	Visual/mas s debris
RILEM MS-A.2	Single (Brick/ston e)	Na ₂ SO ₄	Saturated solution Wet-dry cycles	yes	Constant 20 °C 50%RH	Visual
EN 12370	Single (stone with porosity >5%)	Na ₂ SO ₄	14% Wet-dry cycles	yes	105 °C high RH	Mass change specimen
WTA	Single (plaster)	NaCl+ Na₂SO₄+ NaNO₃	Continuous absorption from salt solution (55g salt/l water)	no	Constant 20°C 65%	Visual

Table 1:	Most common salt cr	ystallization standard	procedures
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2.1 Specimens

The first step in an accelerated test is the choice of the type of specimen, which should be as much as possible representative for the practice situation. From table 1 it is clear that most procedures are carried out on single materials, while it is well known that salt accumulation (and thus damage) depends on the moisture transport properties of the combination of materials [25]; testing single materials can thus lead to misleading results. On the other hand, the use of large masonry specimens [26] or even full scale models [27] while being more realistic, may require too long test periods. A compromise between fully representative test specimens and duration of the test is therefore necessary.

An interesting example with respect to this issue, is given by the definition of an adequate laboratory specimen for the assessment of the salt resistance of mortars (bedding, pointing, plaster or render). Experience shows that the behaviour of a mortar is strongly affected by the material with which it is combined [28-29]. Moreover, the pore structure of the mortar itself is different and depends on whether this is prepared on a non-absorbing substrate (e.g. Teflon or metal mould) or on a porous substrate, (e.g. brick or stone unit) [30-31]. Therefore testing 40 x 40 x 160 mm3 mortar specimens prepared in a metal mould for evaluating salt resistance of a 15 mm thick plaster layer on a brick/stone substrate might lead to distorted results. For testing of mortar, a 20 mm thick layer prepared on (plaster/render) or in between a brick or stone substrate (bedding mortar), might give a more realistic specimen for salt crystallization tests (figure 1) [32].



Figure 1: Specimens for testing the durability to salt crystallization of plaster (left) and bedding mortar (right)

When discussing the reliability of predictions made on the basis of an accelerated laboratory test, it should also be stated that the durability of a fresh stone from the quarry differs, and is not always higher, from that of a stone in a historic building. The results of ageing of the stone, as for example gypsum crust formation on limestones, can affect the response of the stone to salt degradation processes [33-34]. Also the water absorption / hygric behaviour and strength of a material itself may be time dependent, as is the case for many volcanic tuffs. Such aspects are generally not taken into account in any durability tests.

Another situation in which the choice and preparation of specimens is of outermost importance, is that in which durability of conservation treatments, in particular consolidant products, to salt crystallization needs to be assessed. Consolidants are meant to restore the cohesion of decayed surfaces and they are therefore applied in practice on already decayed substrates. This implies that, in order to achieve reliable data on the behaviour, also the substrate used in laboratory tests should be artificially aged prior to the application of the treatment and the execution of the salt crystallization test. However, mainly due to the difficulty to prepare decayed substrates in a reproducible way, this is usually not the case and surface treatments are commonly tested on sound (fresh) substrates. Recently, different methods to prepare reproducible, decayed specimens for testing consolidation treatments have been proposed (figure 2) [35-36].



Figure 2: Specimen with a surface layer presenting insufficient cohesion, obtained by re-aggregating stone grains used for testing consolidant products

2.2 Salt type and amount

The salt type chosen in the test is extremely relevant for the obtained results. Sodium sulfate is the most damaging salt type in crystallization tests, for this reason it is often prescribed in test procedures. However, the harmfulness of sodium sulfate strongly depends on the test conditions used (wet-dry cycles, temperature and RH). The harmfulness of sodium sulfate in salt crystallization tests lays mainly in its tendency to supersaturate (and thus to develop high crystallization pressures) and in the different solubility of its different (hydrated) forms [a.o. 37]. Research has demonstrated that high crystallization pressures are developed by rewetting by liquid water and partial dissolution of anhydrous sodium (thenardite) followed by immediate re-crystallization sulfate into agglomeration of fine crystals of mirabilite (Na2SO4.10H2O) at a very high supersaturation [38]: re-wetting with water is in fact a crucial part of most crystallization tests. From this it is clear that the salt type should be chosen keeping in mind that using one salt or another is not simply a way to speed up the damage, but might simulate damage mechanisms different from those occurring in practice. Studying the aging of a wall exposed to marine environment by immersing a brick repetitively in a saturated sodium sulfate solution might lead to unreliable results.

Next to the salt type, the salt amount should be realistic: saturating a specimen with a salt solution at high concentration several subsequent times [EN 12370] or by continuous supply [e.g. 39] might be unrealistic with respect to the practice situation, not only because such saturated solutions do not exist in practice, but also because the type of damage

obtained might be unrealistic. Salt damage manifests in the field usually in the form of powdering and scaling of the material surface, sometimes accompanied by fluffy or whisker-like efflorescences; features as cracks perpendicular to the evaporation surface or dense salt crusts as observed in accelerated tests with highly concentrated solutions [5, 39-40] are quite unusual in practice.

A high salt content might in some cases, for example in sodium chloride crystallization tests, even be contra-productive for the effectiveness of the test. Slightly higher salt amounts than generally observed in practice can obviously be used in accelerated tests, but our experience shows that in case of NaCl, increasing the salt amount too much might sometimes even delay damage development. It is mainly the number of dissolution/re-crystallization cycles of the salt that may speed up the damage; increasing the amount of salt delays the drying speed of the salt contaminated specimen and thus decreases the number of wet-dry cycles possible in a certain time period. 1-2% Na2SO4 and 2-4% NaCl (weight salt/weight dry specimen) are realistic salt contents, which are sufficient to make a test effective [e.g. 41].

2.3 Test conditions: wet-dry cycles, temperature and RH

A very important parameter in crystallization tests is constituted by the presence of wet-dry cycles and by the environmental conditions (temperature, RH and air speed) during these [42]. First of all, not all test procedures foresee cycles of wetting and drying. In some cases continuous wetting from one side of the specimen and continuous drying on the other side (tested side) are used (e.g. 23, 43). Most tests include wet-dry cycles, by re-wetting of the specimens with water or with salt solution, either by full immersion or capillary absorption from the side opposite to that to be tested. As wet/dry cycles are the trigger for the occurrence of damage, it is advantageous when a sufficient number of wet-dry cycles occurs in a limited period of time. A wetting cycle (dissolution of the salt) is useful if the specimen is sufficiently dry, i.e. if a large part of the salt present has crystallized. For this reason, it might be advantageous to speed up the drying phase and to re-wet the specimen only when most of the water (> 80%) has evaporated. When using 20 °C/50% RH drying conditions and relatively large specimens, as prescribed e.g. by RILEM MS-1, it might take longer than a year to get some conclusions from the test. Apart from reducing the specimen size [a.o. 29], enhancing the drying within reasonable limits can be an option to speed up the test. A temperature of 105 °C, as prescribed in EN 12370, is clearly unrealistic. A too fast drying leads to salt accumulation in-depth with consequent cracking in the specimen [e.g. 5, 44]. As mentioned, this is not the typical type of damage for salt crystallization. Moreover, such high drying temperatures may provoke breakdown of some phases (such as primary ettringite) in for example a cement-based mortar or (reversible) dehydration of phases (zeolites) in for example zeolitized volcanic tuffs (common natural stones in many European countries).

Another approach to speed up the test, consists of introducing, next to rewetting, RH changes across the RH of equilibrium of the salt used during the drying phase. In this way, not the complete specimen is rewetted but, by hygroscopic adsorption, only the outer layer in which the salts have accumulated. Even if the presence of a high RH period slightly increases the drying time, the alternation of RH across the RH of equilibrium of the salt causes several dissolution/crystallization cycles in a limited period of time. This procedure has been proven particularly effective for testing the resistance of material to sodium chloride [29, 45]. This type of salt, well-known for its aggressiveness in the field, is generally just slightly damaging in accelerated laboratory tests based on continuous supply of solution or on re-wetting and drying. The additional use of RH cycles improves the effectiveness of the test, ensuring at the same time a realistic simulation of the field situation.

2.4 Assessment method

In order to assess the effects of salt crystallization in laboratory tests, different methods can be used. In most procedures visual and photographical observations are used. Salt damage is mainly affecting the surface, therefore those techniques measuring changes of the surface are appropriate. The limit of visual observations is their subjectivity and the absence of numerical values, which might be used for an easy comparison between results or for the development or the validation of a model. In order to overcome this limit, other techniques (e.g. laser scanning of the surface [a.o. 27, 46]) are sometimes applied.

The weight of the specimen during the execution of the test, as recommended by the EN 12370 standard, is often used as criterion too [a.o. 40, 44, 47]. The main limit of this last method is that the weight of the specimen depends both from salts absorbed and material loss, and these two factors cannot be distinguished, unless washing of the specimen is performed to remove the salts, an action only possible at the end of the test. A better way in the authors' opinion, consists in measuring the weight of the debris (salt + material) which can be brushed from the surface of the specimen after each wetting cycle; the salt and material can then be further separated by dissolving the salts in water followed by a filtration of the aqueous solution [45].

The reduction in strength of the specimens is considered in some cases as criterion for evaluation, measured by mechanical tests or by ultrasonic measurements [a.o. 5, 48-49]. This last technique has the advantage of being non-destructive and might, in theory, be used for monitoring the development of damage during time. However, also in this case the presence of the salts can alter the results; washing of the specimens before the measurements solves the problem, but, again, this procedure can only be done at the end of the test.

3 How far can accelerated tests predict the in-situ durability of materials with respect to salt crystallization?

The aim of a salt crystallization test is the evaluation and, whenever possible, the prediction of the durability of materials subjected in-situ to the action of salt crystallization. At present, results from crystallization tests cannot easily be translated to in-situ situations. A step forward in this direction might be achieved by the definition of classes of material durability to salt crystallization, following the approach used in the Dutch NEN 2872 standard [50] for the determination of the frost resistance of materials.

A first factor limiting the definition of a classification of material according to their durability, is the use of many different procedures in crystallization tests. The reluctance of researchers to the use of a common standard procedure hinders comparison between results of different studies. As previously discussed, this may be caused by the unrealistic character of some existing standards as well as by the necessity of simulating situations which are not well represented by the existing standard tests. A reliable and commonly accepted standard procedure should be the first step towards a classification of materials according to their durability with respect to salt crystallization.

To reproduce in laboratory the different aggressiveness of different in-situ situations an approach, which recalls the one used by Van der Klugt [51-52] in the definition of the Dutch NEN 2872 standard [50] for frost testing, might be used. Van der Klugt defines 3 classes of aggressiveness of the test (by varying some variables as the water saturation degree of the specimens), each of which can be used to simulate different in-situ situations. Even if frost damage is perhaps a better understood process which can be described by relatively few variables (mainly saturation degree and temperature, next to material properties), whereas the mechanism of salt crystallization damage is still under discussion and much more complex, this kind of approach might be valid. By considering parameters such as salt type, salt content, wet-dry cycles, temperature and RH cycles and defining their importance for the development of the damage, aggressiveness classes of salt crystallization tests can be defined.

For the choice of a suitable aggressiveness of laboratory test, the aggressiveness of environmental in-situ conditions should be considered. Most relevant parameters in this case would have to include moisture supply, salt type, salt load and environmental conditions, as frequency and amplitude of temperature and RH changes.

Next to different aggressiveness classes of the test, the standard procedure should include objective measuring methods and criteria for the evaluation of the decay. Nowadays, the durability of a material, as resulting from the salt crystallization test, is only rarely measured in absolute values (e.g. material loss after n cycles, % of decayed surface); often durability is expressed in a relative sense, i.e. with respect to that of other substrates included in the study. This complicates the comparison between results from different experiments (even if performed according to the same standard) and hinders a classification of materials on the basis of their salt resistance. Defining objective methods for the measurement of the damage and criteria (see examples reported in section 2.4) is a first step in this direction. Durability classes can be then defined and the materials could be classified according to these at the end of the test, on the basis of the test conditions used and the damage observed.

A further step in the classification of materials according to their durability with respect to salt crystallization, is represented by models able to predict service life of material in different in-situ situations.

For the development and validation of models for service life prediction on the basis of laboratory test results, which include a time ratio between lab and in-situ durability, first of all field studies are necessary. Unfortunately, these are limited because of the difficulty of retrieving historical information about the studied buildings (age, eventual historic events as fire, flooding etc., past conservation interventions, etc.), the co-presence of different weathering agents and the large number of samples which need to be collected in order to get statistical significance (see e.g. [53]). Moreover, direct comparison between practice and laboratory is complicated due to the following factors [19, 33]:

- The use of a single damage process (in this case salt crystallization) and often of a single salt in laboratory, while in the field a combination of different salt types and different weathering mechanisms, enhancing or delaying the decay, is generally present.

- The use of a fixed number of cycles (e.g. wet-dry cycle, temperature and RH) with a unified frequency and magnitude in laboratory, whereas in the field cycles are more heterogeneous.

- The uncertainties, deriving from 1 and 2, in estimating the effect of differences, with respect to environmental conditions, salt and moisture load etc., between the real situations and the laboratory.

- The lack of quantitative data on the evolution of the damage in-situ; this is due not only to the long-time monitoring necessary on site, but also to the difficulty of defining a method for a quantitative and objective assessment of the damage. Damage assessment in laboratory is generally done by measuring changes in weight and strength of the specimens, methods, which are not easily applicable on site. Defining a relation in time between the laboratory and the field is therefore very complex, since it would require a long term monitoring of the behaviour of the material in the field (according to the same method used in the laboratory) as well as of the environmental conditions relevant to salt crystallization, as salt types and content, moisture supply, drying conditions (temperature and RH) amplitude and length of RH cycles. Moreover, the relative importance of each of these parameters as well as their interaction should be evaluated. This knowledge is at the moment not fully available and hindered also by the lack of a definitive and complete understanding of the damage mechanism of salt crystallization.

4 Conclusions

For the time being laboratory crystallization tests are the main source of information for forecasting material durability with respect to salt crystallization. They still constitute, with all the above discussed limits, a useful method for the a evaluation of the resistance of porous building materials to salt weathering.

A step forward in forecasting material durability in situ by accelerated test can be done by defining a classification of materials according to their durability to salt crystallization. This objective can be pursued by:

- Defining an effective standard salt crystallization test, i.e. a test reproducing in a realistic way and within a relatively short time the damage mechanism and decay patterns occurring in situ.

- Including different aggressiveness classes in the test procedure (by varying some of the parameters as salt content and moisture supply), corresponding to different aggressiveness of conditions in-situ. Depending on the foreseen application of the material, the most suitable class within the laboratory test can be chosen.

- Defining an objective, quantitative, method for measuring the decay (e.g. material loss, % decayed area).

- Determining different classes of durability of materials with respect to salt crystallization on the basis of their response to the test.

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