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## The impact of salt pore clogging on the hygric properties of bricks

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### HIGHLIGHTS

- In-pore salt crystallization affects materials' pore structure and hygric behavior.
- This study experimentally investigates these influences of pore clogging by salt.
- A homogeneous salt distribution in the samples is achieved by use of freeze-drying.
- After which hygric properties are measured for various amounts of crystallized salt.
- The pore clogging impacts on pore structure and hygric behavior are salt-dependent.

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### ABSTRACT

This paper presents an experimental analysis of the impact of salt pore clogging on the hygric properties of bricks, with a focus on  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  and  $\text{NaCl}$ . The study includes the pore clogging induction and hygric property measurement. The former implies establishing an even distribution of salt crystals in the samples, whereas the latter is related to determination of the basic hygric properties. The experiments reveal that even a limited amount of pore clogging can already significantly affect the hygric properties of bricks. It is moreover shown that the impact of pore clogging is highly dependent on the salt type.

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### 1. Introduction

The development of gypsum efflorescence on ceramic brick façades is a rising problem in the UK, the Netherlands and Belgium [1–3]. Forming persistent white-gray stains on façades of newly erected buildings, it causes aesthetical defects that lead to the dissatisfaction of building owners. Motivated by this progressively increasing occurrence of gypsum efflorescence, Chwast et al. [1] performed a literature review and a field survey on this topic, revealing a complex interplay between multiple factors. Subsequently, gypsum wicking experiments under laboratory conditions were conducted, to gain further insight into the phenomenon [4]. The wicking experiment was composed of a ceramic brick sample fed with gypsum solution at the bottom, which was allowed to evaporate at the top. This experimental campaign however revealed gypsum's tendency to crystallize underneath the drying surface, yielding subflorescence instead of efflorescence. That sub-

florescence induces pore clogging below the drying surface and therefore a strong drop in the wicking rate. Similar observations have been reported in [5,6]. Moreover, [7] equally confirmed the occurrence of gypsum pore clogging while studying the mechanisms of plasterboard drying. Most experimental evidence on gypsum wicking hence demonstrates that gypsum primarily precipitates as a thin layer below the surface, leading to a significant reduction of the moisture storage and transport due to pore clogging. These observations formed the primary motivation for the research reported in this paper: the impact of gypsum pore clogging on the hygric properties of ceramic bricks.

In-pore crystallization of salts is however equally crucial in many other fields. When it comes to building materials and constructions, it can cause mechanical damage, like spalling or exfoliation [8–10]. Furthermore, it can cause pore clogging, affecting the pore structure and thus the hygric behavior of porous materials [4–7,11–16]. Besides in building materials, salt pore clogging has an undesirable impact in the petroleum industry [17–20]. The effectiveness of water injection during oil extraction is reduced due to salt deposition in the technical equipment as well as in the porous medium [17–20]. A similar phenomenon is

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encountered in case of water desalination where salt deposition occurs on the semipermeable membrane, reducing the efficiency of the filtration [21–23]. In both fields, these problems are known as scale formation.

Even though salt crystallization in porous materials has already been the subject of study for many years, there is still a lack of knowledge and understanding in relation to pore clogging and its impact on the fluid transfer properties of porous materials. Therefore, this paper aims at quantifying in which way and to what extent pore clogging induced by crystallization of gypsum and sodium chloride affects the hygric properties of ceramic brick. To that end, this paper is divided into several sections. Firstly, we bring a literature review on the impact of salt crystallization on the pore structure and hygric properties of porous materials. Secondly, we present a novel protocol to quantify the impact of gypsum pore clogging on the hygric properties of ceramic brick. This experimental campaign consists of two key parts. The first aspect deals with the induction of pore clogging. In this part, the methodology for overcoming gypsum's low solubility as well as gypsum migration during drying is presented. In the second part, brief descriptions of the measurement techniques for moisture transport and storage properties follow. Subsequently, the impact of gypsum pore clogging on open porosity, capillary and vacuum saturated moisture content, capillary absorption coefficient, pore volume distribution and liquid diffusivity is analyzed. As a result, we show that even a small reduction of open porosity can significantly influence the hygric properties of a porous material. Finally, the impact of sodium chloride pore clogging is also studied, and the comparison with the gypsum results shows that the impact of pore clogging on the hygric properties of ceramic bricks strongly depends on the salt type.

## 2. Literature review

Salt crystallization inside a material's pores, and the impact it has on the material properties, has been investigated for many years. For that purpose, a wide spectrum of experimental and numerical studies have been conducted, covering different types of salts and materials. Such a broad research history does not only contribute to our knowledge and insight of the whole problem, but it also reveals its complex nature, because it does depend on various parameters. Therefore, in this part we first bring an overview of the experiments that are frequently used to induce salt precipitation in porous media. Secondly, we present salts' impact on porosity and pore structure, and further continue with changes of materials' hygric properties due to the in-pore salt crystallization.

### 2.1. Induction of salt crystallization in porous media

Depending on the actual aim of the research, crystallization experiments can be classified into two groups: wicking and absorption tests.

A wicking test comprises continuous uptake of a salt solution from one side and a continuous evaporation either from the opposite surface or from multiple other surfaces. The direction of the liquid transfer depends on the location of evaporation. Accordingly, the wicking test can be divided into two subgroups:

1. *Unidirectional wicking (UW)*: when the lateral sides of the samples are covered to impose a unidirectional liquid transfer. The liquid uptake takes place at the bottom side, while evaporation is permitted at the top side. This experiment is also known as an evaporation experiment. In this case, salt will crystallize at and/or below the top surface only. This type of test is mainly performed to study the impact of salt crystals on the drying behavior of porous materials [6,24,25].

2. *Omnidirectional wicking (OW)*: when all surfaces of the samples are left uncovered. The procedure is virtually the same as above, only here the evaporation can take place from all sides. During that process, salt can precipitate at and/or below the lateral and top surfaces. This type of wicking test is mainly applied when focus is on the damaging mechanisms of salt crystals [26–28].

An absorption test comprises uptake of a salt solution until the sample is completely (capillary) saturated. Once saturation is reached, the source of the solution is removed and the sample is subjected to drying. Based on the absorption protocol, this test can also be divided into two subgroups:

1. *Unidirectional absorption (UA)*: when all surfaces except the bottom side are covered. Solution imbibition takes place only from the bottom side, while evaporation from other surfaces is prevented.
2. *Omnidirectional absorption (OA)*: when sample is immersed into a salt solution – under vacuum or under controlled environmental conditions.

To subtract solvent from the specimens, a single or a combination of drying methods is often applied:

- a. *Oven-drying at high temperatures (OD)*, exposing all surfaces. This approach is used to obtain an even salt distribution inside the sample by shifting the drying front into the bulk of a specimen [12,13]. This drying method follows both the unidirectional [12,13] and omnidirectional [29,30] absorption. The combination of this drying method with any of the absorption tests is mainly applied when the interest is the change of porosity, pore structure and hygric properties by salt crystals.
- b. *Drying under the controlled environmental conditions (ED)*, either subjecting all the surfaces or only one to the surroundings. The former is usually applied when change of the porosity and pore structure is studied [31], whereas latter is used when the impact of salt crystals on a drying rate is of interest [11,16,25,32,33], similar to the unidirectional wicking test. This drying method usually follows the omnidirectional absorption.

Although all these protocols seem to be very similar, it is important to highlight their significant differences. During the wicking test salt crystallizes on or/and below the exposed surfaces already during the test, and therefore, the impact of salt crystals is observed already during the solution imbibition. On the other hand, salt crystallization in the absorption test takes place only during the drying. The choice of the drying method depends on the preferable location for salt crystallization. When a specimen is dried only from one side, the aim is to form a so-called bilayer material, where the zone of salt crystals is located at the evaporating side. On the contrary, exposing all the surfaces to drying conditions, especially to high temperatures of 105–110 °C, aims to obtain an even salt distribution in the sample. Unfortunately, each drying method still leads to a certain degree of efflorescence formation at the exposed surfaces, leaving smaller amounts of salt inside the samples [13], and certainly disturbing the uniform salt distribution.

### 2.2. Impact of salt crystals on porosity and pore structure

Despite of the differences in the protocols all the above mentioned tests have something in common: they all result in salt in-pore crystallization, which leads to modifications of the material's porosity and pore structure. Experimental studies have been

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