



Influence of temperature and salt concentration on the salt weathering of a sedimentary stone with sodium sulphate

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ABSTRACT

The aim of this study is to evaluate how the ambient temperature and the salt concentration affect the salt decay of a sedimentary stone. Samples of a detritic limestone have suffered cycles of accelerated ageing at 5, room temperature (around 22 °C) and 50 °C with brines which had different sodium sulphate concentration. The weight of the samples and of the pieces fallen off during the cycles has been monitored. The results show that the damage is more important at 5 °C than at room temperature. The samples at 50 °C were intact at the end of the experiment. Second, the weathering patterns are different at different temperatures: fine crumbling at 5 °C; coarse crumbling and contour scaling at room temperature. The decay seems also to be of a different kind for each concentration at room temperature: crumbling at low concentration, contour scaling at high concentration. Crystallization seems to take place deeper inside the porous network of the stone when the concentration of salts in the brine is higher, that is to say when the brine viscosity is higher.

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1. Introduction

Salts, and particularly sodium sulphate, are known to be among the most destructive agents in porous stones, concrete or brick weathering. The study of its crystallization mechanism is thus very important to fully understand its damaging effect on porous networks and, in the future, to find a way to prevent or limit it. Recent studies attribute the importance of sodium sulphate decay to the salt crystallization pressure of its decahydrate phase (mirabilite $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$), rather than of its anhydrous phase (thenardite Na_2SO_4) (Rodríguez-Navarro and Doehne, 1999; Tsui et al., 2003; Angeli, 2007). Nevertheless some recent works have noted that the unstable heptahydrated phase could also have an effect on the damaging effect of sodium sulphate (Rijniers et al., 2005; Hamilton and Hall, 2008; Steiger and Asmussen, 2008). Thermodynamic studies (Benavente et al., 1999; Flatt, 2002; Scherer, 2004; Steiger, 2005a,b; Coussy, 2006) as well as experimental studies (Goudie, 1986, 1993; Rodríguez-Navarro and Doehne, 1999; Angeli, 2007; Angeli et al., 2007) show that damage depends on the quantity of salt in the stone and the characteristics of the porous network, as well as on the environmental conditions (e.g. temperature and relative humidity).

This study aims to understand what role the temperature and the salt content in the sample play in the decay of a sedimentary stone, regarding the type and intensity of damage. For this, accelerated ageing tests are performed on only one type of stone, a detritic limestone, and with only one salt, sodium sulphate, but in different thermodynamic conditions (temperature and salt concentration of the brine).

2. Materials and methods

2.1. Materials

Twelve cubic ($\sim 7 \times 7 \times 7 \text{ cm}^3$) samples of a lutetian limestone from the Parisian Basin commercially known as “Roche fine” have been used in this study. This rock has been chosen for several reasons: its detritic structure with unimodal porosity is much simpler to study than other bioconstructed or crystallized limestones; it has been demonstrated in previous tests (Angeli et al., 2006, 2007) that it is not very resistant to salt decay hence the tests are quite fast; it is homogeneous and very regular from one sample to the other, which allows a very good reproducibility of the tests; it has been widely used for construction in Paris in the past time (buildings and historical monuments); and it is one of the rocks imposed for restoration and construction in the protected areas of the “Ile de France” region. It is a fine-grained detritic limestone made of calcite (90%) and quartz (10%) with a high porosity (37.2%) and a very low tensile strength (1.5 MPa). Its pore distribution is unimodal which makes the study of its modification easier. Complete hydromechanic properties are given in Table 1.

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Table 1
Hydromechanic properties of the “Roche Fine” (from Angeli et al., 2007).

	Porosity %	Absorption %	Bulk density g/cm ³	Evaporation coef. g/m ² /s ^{1/2}	Capillary coef. g/m ² /s ^{1/2}	Median pore radius μm	P wave velocity m/s	Tensile strength MPa
Roche fine (St Maximin)	37.2	75.9	1.7	67	1106	12	2898	1.5

2.2. Methods

The twelve samples were subjected to accelerated ageing tests adapted from the European standard EN 12370. Those tests were run under three different ambient temperatures: 5 °C, room temperature (RT ~ 25 °C), and 50 °C. It is important to notice that two ambient conditions are below the temperature limit of mirabilite stability (32.4 °C), the last one being above which means that no mirabilite crystallization will occur for this experimental condition.

Three different concentrations of salt in solution have been used in this study: 5, 12 and 25% of anhydrous sodium sulphate (thenardite) in weight. The tests have only been performed under six thermodynamic conditions: indeed the three following conditions (12%/5 °C, 25%/5 °C and 25%/RT) are supersaturated with respect to mirabilite, that is to say that under these conditions a liquid solution would be metastable, i.e. not suitable for capillary imbibitions. The six thermodynamic conditions tested here are represented as stars on the phase diagram (Fig. 1): 5%/5 °C; 5%/RT; 5%/50 °C; 12%/RT; 12%/50 °C; 25%/50 °C.

As can be seen on the figure, these conditions correspond to various saturation conditions of the sodium sulphate brine. This saturation ranges from 0 for pure water to 1 for full saturation. The closer the solution is to the full saturation conditions, the faster it will reach supersaturation hence weathering of the sample. This will happen after fewer imbibitions for a higher saturation (Flatt, 2002; Coussy, 2006). Here are the six saturation conditions tested in this study:

$T = 5\text{ °C}$	5% in weight – around 0.85 saturation
$T = 25\text{ °C}$	5% in weight – around 0.25 saturation
	12% in weight – around 0.60 saturation
$T = 50\text{ °C}$	5% in weight – around 0.17 saturation
	12% in weight – around 0.40 saturation
	25% in weight – around 0.83 saturation

These tests are composed of cycles which have a duration of 24 h. The cycles are made of three different stages: imbibition at an ambient temperature; drying; return to initial ambient temperature. Timing for each stage is modified from the EN 12370 standard about stone resistance to crystallization of salts in pores.

Let us describe in more details the three steps:

- (i) 2 h imbibition with a brine (concentration of Na₂SO₄ is fixed by the 6 thermodynamic conditions) is realized for the three different ambient temperature conditions. Note that temperatures of imbibition solution are the same as ambient temperatures, i.e. respectively 5 °C, RT and 50 °C. Samples soak in a container with 1 cm height of solution. The level of solution is maintained constant during all stages. Imbibition solution has been prepared with sodium sulphate decahydrate and demineralised water. The cycles start at 16 h in the afternoon, the weighing at the beginning of this step will thus be called 16 h weighing.
- (ii) Drying: All the samples are placed in a drying oven at 105 °C for 16 h. The weighing performed at the beginning of this step will be called the 18 h weighing. This step ends at 10 h in the morning.
- (iii) Return to initial ambient temperature, i.e. 6 h of cooling from 105 °C to respectively 5 °C, RT and 50 °C. The weighing performed at the beginning of this step is the 10 h weighing.

Sixteen full cycles have been performed during which all the samples have been weighed three times per cycle (before imbibition, after imbibition, and after drying). The parts of the stones which fell during the experiments with size from approximately 0.5 mm were collected and weighed every cycle after drying. They will be referred to as “remains” until the end of the article. The only remains which were not collected consist in powder resulting from crumbling.

A cooled incubator is used for the 5 °C ambient environment. 50 °C ambient temperature is obtained using a drying oven and drying stage takes place in a drying oven. Temperature is set up with a precision of ± 0.1 °C.

3. Results

3.1. General observations

The first thing to notice is that all the samples present a different decay from one thermodynamic condition to the other. Some pictures of each sample that are characteristic of the decay are presented in Fig. 2. Future references to this figure will refer precisely to the experimental condition and the number of the cycle.

At 50 °C, no damage has been noticed on none of the 6 samples, only harmless efflorescences on the sides of the samples (Fig. 2; 50 °C, cycle 15). At 5 °C, the damage starts directly as soon as the samples contain salt, i.e. during the second cycle. After this cycle, few signs of very thin contour scaling appear (a few tenths of millimeters). During the third cycle, the thin contour scaling almost ends and is followed by crumbling. This crumbling is very regular throughout the cycles until the end of the test, and the particles lost form a very fine powder.

At RT, the decay processes are a little different. During the 5–6 first cycles, the samples are slowly damaged. Efflorescences are observed as well as a decay of the lower parts of the samples, those who are immersed during the tests. The efflorescences are unexpectedly more important on the sample saturated with the 5% brine, i.e. with the lowest salt concentration. But at this point no major difference is observed between the two concentrations of salts (5 and 12%). From approximately the 6th cycle, contour scaling starts to appear on all samples, with the detached part thicker for 12% than for 5%. In fact,

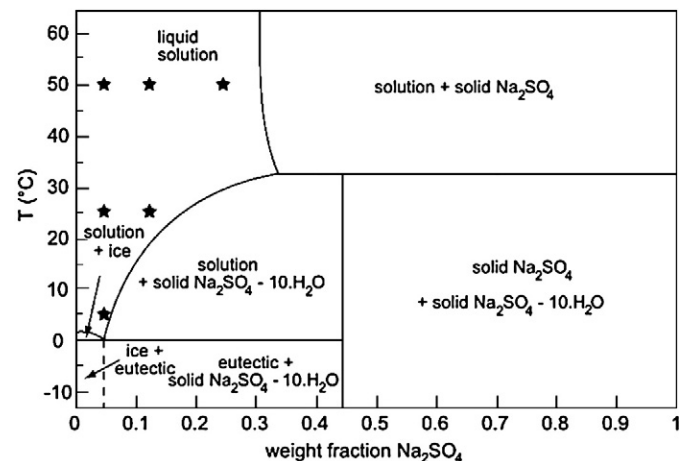


Fig. 1. Sodium sulphate phase diagram (Hougen et al., 1954). The black stars represent the six thermodynamic conditions studied.

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