Geological Society, London, Special Publications

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Geological Society, London, Special Publications 2010; v. 333; p. 111-118
doi:10.1144/SP333.11

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Evaluation of the compatibility of building limestones from salt crystallization experiments

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Abstract: Salt absorption in porous building stones contributes greatly to the degradation of monuments. Two French porous limestones with similar main characteristics (total porosity, densities and mechanical resistance) were studied: white Tuffeau and Sebastopol stone. Accelerated ageing (weathering) tests were carried out by applying immersion-drying cycles with water containing sodium sulphate or sodium chloride. Samples of the two stones were tested separately and then in sets containing both rock varieties. The results facilitated interpretation of the observed and measured responses of these two limestones to the cycling salt crystallization. The durability of studied stones was evaluated by determining the normalized weight changes during the applied cycles. The Sebastopol stone amplified the amount of salt stored in the Tuffeau with increasing number of cycles performed, inducing its more rapid degradation. Water retention and water transfer in the pore space were found to be two main factors controlling the rate and the type of stone decay due to the salt crystallization.

Limestones used in monuments gradually deteriorate with time due to several physical, chemical and biological complex processes. It is well known that water plays a key role in the phenomena of stone deterioration. Indeed, water can act directly with freezing–thawing or dissolution of some minerals such as calcite. Water can also act indirectly by the transport of soluble salts which are major agents of the deterioration of building stones. Sodium sulphate and sodium chloride are the most frequently found salts and the most damaging to historical monuments (Goudie & Parker 1998; Rodriguez-Navarro & Doehne 1999; Benavente *et al.* 2001; Angeli *et al.* 2006). Salts can originate from various sources: air pollution, soil, sea spray, inappropriate chemical treatment or interaction between building materials such as mortars which may contain salts (Price 1995).

The stone degradation is closely related to its geomechanical properties and weathering agent. The petrophysical properties that characterize the durability of a stone are mainly the tensile strength and the pore space (porosity and pore size distribution). Nevertheless, part of the degradation of monuments built with limestones originates from an incompatible association between the original construction stone and the stone of replacement in restoration works. The replacement of the damaged stones is a crucial issue in the restoration of historical monuments. In fact, the choice of compatible stones constitutes a major difficulty in France because some quarries of original stones are now exhausted or unidentified. The aim is to find a new stone similar to and compatible with the old stones if it is impossible to use exactly the same. The aim of this work is to study the compatibility between stones gathered during ageing tests. The behaviour of each stone was previously studied during the same ageing test.

The durability of the stones is studied through the measurement of the stone resistance to salt crystallization by means of accelerated ageing tests. Stone which retains its original size, shape and appearance over an extensive period of time during these tests is considered to have a high durability. These tests are carried out by applying immersion-drying cycles using water polluted by salts (sodium sulphate Na_2SO_4 and sodium chloride NaCl) in accordance with the EN 12370 European standard test. Two French building limestones with a similar high total porosity (around 45 vol.%) are tested in this study: the white Tuffeau and Sebastopol stone. The compatibility of the two stones is analysed with the observation of reaction to the ageing test for two types of samples: isolated stones and stones linked together (Tuffeau and Sebastopol stone). A comparison of the behaviour of the two stones under the same accelerated weathering test allows the compatibility these stones to be determined.

Materials and methods

Materials

The studied limestones were characterized using different complementary techniques. Mineralogical

From: PŘIKRYL, R. & TÖRÖK, Á. (eds) *Natural Stone Resources for Historical Monuments*. Geological Society, London, Special Publications, **333**, 111–118.
DOI: 10.1144/SP333.11 0305-8719/10/$15.00 © The Geological Society of London 2010.

Table 1. *Main physical properties of two tested stones*

	Tuffeau stone	Sebastopol stone
Mineralogical composition	Calcite ≈50% Quartz ≈10% Opal CT ≈30% Clays and micas ≈10%	Calcite ≈80% Quartz ≈20%
Skeletal density (g cm^{-3})	2.55	2.71
Bulk dry density (g cm^{-3})	1.31	1.58
Porosity (vol.%)	48	42
Median pore access diameter (μm)	2.7	17.9
Tensile strength at saturated state (MPa)	0.45	0.59
Compressive strength at saturated state (MPa)	4.5	5.8

composition was determined by X-ray diffraction, thermogravimetric analyses, infra-red spectrometry and scanning electron microscopy. The characterization of pore space was carried out by mercury intrusion porosimetry. Tensile strength was calculated from the results of the Brazilian splitting method with an Instron 4485 press. The main physical characteristics of stones are summarized in Table 1. The two limestones studied (Tuffeau and Sebastopol stone) were selected because they have several close characteristics: total porosity of about 45 vol.% and a compressive strength and a tensile strength at the saturated state of the order 5 MPa and 0.5 MPa, respectively. Mechanical properties (compressive and tensile strengths) were measured in the saturated state since mechanical strength is much weaker than dry stone strength (West 1994; Beck *et al.* 2007).

White Tuffeau is commonly used in most monuments built along the Loire valley such as the famous Loire Castles (Beck *et al.* 2003) and the Sebastopol stone is used near Paris. The major minerals of these limestones are calcite (i.e. $CaCO_3$) and quartz (i.e. SiO_2). Moreover, in the white Tuffeau, predominantly clayey minerals and opal cristobalite/tridymite are found. The size and shape of grains in white Tuffeau vary significantly and consist of large grains of clay (glauconite), quartz, sparitic calcite and mica arranged with several types of fine grains such as clay (smectite), micritic calcite, small fossil shells and spherules of CT-opal (Beck *et al.* 2003). The Sebastopol stone comprises calcite and quartz grains that are coarser with a more uniform size (Beck & Al-Mukhtar 2005). The white Tuffeau is a relatively light building material. It has an apparent density of 1.31 g cm^{-3} and a total porosity of 48 vol.% under total dry conditions (Table 1). Results of mercury porosimetry demonstrated the multiple porosity nature of white Tuffeau stone with a very wide pore-size distribution (from 6 nm to 20 μm) with a median pore access diameter of 2.7 μm. The pore-size distribution of Sebastopol stone is mainly unimodal (from 1–40 μm) with a median pore access diameter of 17.9 μm and predominant pore access diameter of about 20 μm (Beck & Al-Mukhtar 2005).

Hydraulic properties of these stones are also very different. Indeed, water transfer properties are related to the porosity and to the porous network characteristics constituted by pore sizes, pore shapes and connectivity of the porous structure. Both stones used in this study have relatively high permeabilities and high capillary kinetics. Nevertheless, capillary coefficients of Sebastopol stone are about twice as large as those of Tuffeau and the permeability coefficient is about 15 times greater. This confirms the influence of pore size distribution on the main hydraulic properties (Beck & Al-Mukhtar 2005). Moreover, water absorption is higher in the case of the Tuffeau stone and this limestone absorbs very large amounts of moisture in an environment of high humidity. Indeed, the degree of saturation of Tuffeau is around 20% in a wet atmosphere (98% of relative humidity) while that of Sebastopol stone is about 2% in the same conditions (Table 2). The Tuffeau stone exhibits a very high water retention capacity. Such behaviour can be attributed to the presence of clays and the fine pore space with nanometric pores (Table 1).

Experimental method

Two types of salts were used for the aging experiment: sodium chloride which crystallize to form the halite (NaCl) and sodium sulphate which can crystallize to form the mirabilite $Na_2SO_4 \cdot 10H_2O$ at high humidity or the thenardite Na_2SO_4 at low humidity (Flatt 2002). The experimental material consisted of two different arrangements of specimens: (1) single isolated cubes having dimensions $5 \times 5 \times 5$ cm (three specimens for each rock type), and (2) a specimen prepared by joining together (with a high quality hemp cord) one cube of Tuffeau and one cube of Sebastopole stones.

Table 2. *Main hydraulic properties of the two tested stones*

	Tuffeau stone	Sebastopol stone
Permeability in mD (m s^{-1})	100 (10^{-6})	1450 (14.5 10^{-6})
Capillary coefficients:		
Mass uptake coefficient (g cm^{-2} $min^{-1/2}$)	0.39	0.70
Capillary front coefficient (cm $min^{-1/2}$)	1.04	2.36
Absorption (%)	89	84
Water retention as a degree of saturation (%) at:		
12% RH	1.43	0.15
44% RH	3.11	0.34
76% RH	6.81	0.48
98% RH	22.89	2.37

Experiments have been performed according to the EN 12370 (1999) standard concerning the resistance to salt crystallization in the pore space. Each test comprises 15 cycles of immersion/drying and each cycle consists of the following stages:

- 2 hours of immersion in a salt solution of sodium sulphate or sodium chloride with concentration equal to 14% by weight (temperature 20 °C);
- 20 hours of drying at 105 °C in the oven; and
- 2 hours of cooling at room temperature (20 °C, 40–50% relative humidity–RH) before the next immersion in salt solution.

Samples were weighed at the end of immersion (stage 1) and at the end of drying (stage 2) in order to follow the evolution of the dry and saturated weight according to the imposed cycle.

Results and discussion

Results obtained are presented in terms of normalized weight (weight of the sample at the measured stage/initial dry weight) versus the number of applied cycles of immersion/drying. For each cycle, measurements are composed of a 'saturated' weight (end of immersion stage) and a dry weight (end of drying stage) for the test in the case of the two salts. Results are presented in Figures 1 and 2 for Tuffeau stone and Sebastopol stone, respectively. The normalized weights for the set of Tuffeau and Sebastopol linked together are presented in Figure 3. Figure 4 shows photographs of fresh stones before the ageing test and after the 15th cycle for each type of sample.

In the case of the isolated Tuffeau stone samples, the dry weight and the saturated weight increase regularly with the number of cycles until the 4th cycle. This increase in weight is due to storage of salt in the pore space and on the surfaces of the stone (efflorescence). The saturated normalized weight is identical for the two tests because the salt concentration in mass is the same (14% in weight). Nevertheless, it is noticed that the dry normalized weight is higher with NaCl salt even if the molar mass of sodium chloride is lower than that of the sodium sulphate. The Tuffeau stone therefore tends to store NaCl more easily than Na_2SO_4. From the 4th cycle, the difference between the saturated and dry weights becomes gradually much lower in the case of the cycles with NaCl and less efflorescences display on the surface of the stone.

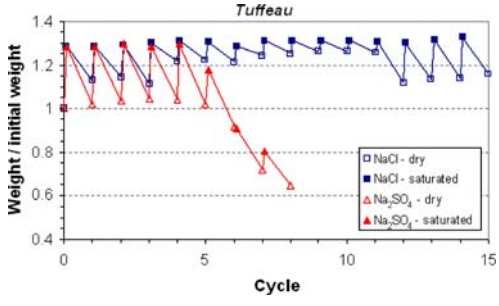

Fig. 1. Normalized weights evolution during the 15 cycles for Tuffeau stone.

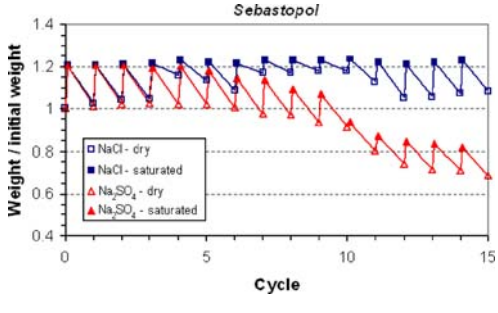

Fig. 2. Normalized weights evolution during the accelerated weathering test on Sebastopol stone.

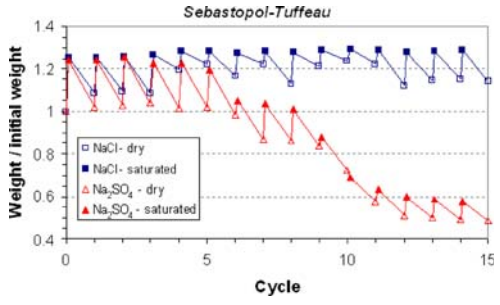

Fig. 3. Normalized weights evolution during the 15 cycles for Sebastopol/Tuffeau linked together with a high quality hemp cord.

The dry weight demonstrates an increase while the saturated weight remains constant. There is more NaCl salt stored in the Tuffeau stone, which has difficulty in drying completely. Indeed, salt is transported near the surface in the drying stage. The crystallized salt can form a barrier in this zone inducing the obstruction of part of the porous network. The salt present in the pore space therefore reduces the evaporation kinetics and the migration of the fluids in the drying stage (duration fixed at 20 hours). This behaviour corresponds to the tendency of NaCl to block pores as demonstrated by Espinosa-Marzal & Scherer (2008).

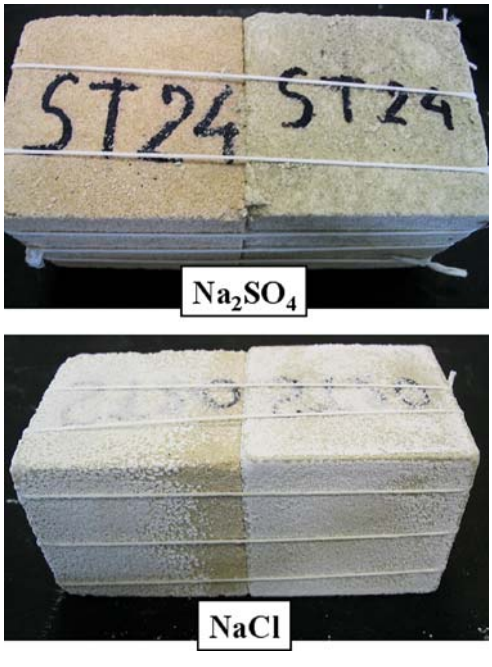

Fig. 4. 'Sebastopol/Tuffeau samples linked together' at the fresh state and after 15 cycles of immersion/drying in the case of Na_2SO_4 and NaCl crystallization.

Table 3 shows the normalized weights of all studied samples at the end of the drying stage of the 15th cycle and of an exceptionally dry state (after one month in the drying oven at 105 °C). The Tuffeau stone was not strictly dry at the end of the final-cycle drying because its normalized weight is 1.160 compared to 1.121 in a dry state. From the 12th cycle, the dry weight decreases, increasing the difference between dry and saturated weight. Little efflorescence is observed. There was no significant matter loss from the stone except for an increase in the roughness of the surface and an erosion of the edges (Fig. 4). It is possible that, during the immersion stage, part of the salt stored near the surface is dissolved. This induces a reduction in the salt content in the stone before drying.

The action of Na_2SO_4 on the Tuffeau stone is different as it generates salt crystallization pressures with a thenardite/mirabilite phase transition (Flatt 2002). The resulting damage is more important. The evolution of the normalized weight with the number of cycles therefore follows an expected behaviour: the weight increases with the storage of salt. The beginning of the deterioration can be observed with a competition between the salt supply and the matter loss (stone damage). Such behaviour has been observed by Angeli *et al.* (2007) on various stones. Lastly, the weight decreases quickly with the stone damage. In the case of Tuffeau, the deterioration is very fast (Fig. 4). The high loss of matter corresponds to an erosion of the edges (5th cycle), then with important crackings (7th cycle) and finally where the sample is completely damaged (8th cycle).

In the case of the isolated Sebastopol stone samples immersed in NaCl solution, the behaviour is practically the same as for the Tuffeau. The only difference is that the Sebastopol stone absorbs less salt solution than the Tuffeau stone in the first cycle: normalized weight is 1.2 compared to 1.3. The phenomena must be similar with a progressive storage of salt and the formation of a barrier slowing down evaporation. Nevertheless, contrary to the Tuffeau, the normalized dry weight is the same for NaCl as for Na_2SO_4. The Sebastopol stone therefore stores less NaCl in its porous structure and does not demonstrate any damage. Indeed, at the end of 15 cycles, the normalized dry weight with NaCl is 1.082 compared to 1.121 for the Tuffeau. In addition, this stone dries more easily than the Tuffeau because it has a lower water retention capacity. There is almost no water stored at the end of the drying stage of the 15th cycle.

The principal difference between the two stones can be shown with the Na_2SO_4 action. In contrast to Tuffeau stone, Sebastopol stone did not show

Table 3. *Normalized weights at the end of the ageing test (weight loss corresponds to normalized weight <1)*

	NaCl			Na_2SO_4		
	Tuf	Seb	Seb-Tuf	Tuf	Seb	Seb-Tuf
Normalized weight at the drying stage of the 15th cycle	1.160	1.083	1.140	–	0.688	0.486
Normalized weight at the dry state (1 month at 105 °C)	1.121	1.082	1.112 Tuf 1.086 Seb 1.131	–	0.688	0.478 Tuf 0.208 Seb 0.785

visible rupture. Degradation is gradual with a homogeneous matter (particles) loss on the surface (Fig. 4). This causes a progressive weight loss (Fig. 2) which in a dry state' is approximately 30%. The total porosity and the tensile strengths, which evaluate cohesion between the particles, are almost the same for the two stones (approximately 45 vol.% and 0.5 MPa respectively). Even if the tensile strength is somewhat higher for the Sebastopol stone, the difference in the deterioration process is mainly due to the large difference in the water transfer properties such as capillarity, permeability and water retention. These parameters are controlled by the structure of pore space, the size and shape of pores and connectivity of the porous network (Beck & Al-Mukhtar 2005). The Tuffeau stone contains about 10% of clay minerals and has an important fraction of very small pores which can retain water at low relative humidity. This could generate high salt crystallization pressure in the pore space and induce the rapid decay of the Tuffeau stone. On the contrary, Sebastopol stone has a very low water retention capacity, a higher permeability and most of its pore spaces are larger compared to those of Tuffeau. Hence, the salt can fill pore spaces of the Sebastopol stone without generating high crystallization pressures.

In case of the samples constituted by a set of Tuffeau and Sebastopol linked together, the behaviour seems to cumulate the properties of the two stones tested separately (Figs 1 and 2). Generally, the weight increases slightly because of the storage of NaCl salt. However, during immersion, some of salts accumulated near the surface can be dissolved and so the total amount of salt may reduce during one to two cycles and increase for the following cycles. At the end of the aging test (after 15 cycles with NaCl salt solution), a slight alteration can be observed to the Tuffeau cubes as some granular disintegration on the surfaces (Fig. 4). It must be mentioned that Tuffeau samples seem to maintain some water at the end of the drying cycle. The drying time (20 hours) is too short for the Tuffeau to dry completely, particularly in the case of NaCl solution.

The compatible-incompatible characteristics of the stones can be observed through the interaction between the blocks of stones linked together. Figure 5 shows the photographs of the stone blocks linked together for the 4th cycle. The stones demonstrate high efflorescence in the case of the cycles carried out with NaCl. Nevertheless, for Sebastopol stone, a zone of approximately 1 cm thickness near the Tuffeau does not have any efflorescence and seems to be humid. This behaviour is due to the interaction between the two stones. Since the pores of the Tuffeau are smaller, the capillary suction generated tends to absorb the solution in the Sebastopol stone. Tuffeau therefore absorbs more salt when it is in contact with Sebastopol stone during the first cycles. From the 5th cycle, the behaviour of the two stones with NaCl solution begins to change because the NaCl salt content becomes enough high to slow water transfer, mainly in the case of Tuffeau. Indeed, Tuffeau has a high water retention capacity and Sebastopol stone has a high water capillarity and a high permeability (Table 2). Sebastopol stone can therefore drain water whereas water transfer is lower within Tuffeau.

Moreover, the Sebastopol stone dries faster than Tuffeau stone. After the drying of Sebastopol is almost complete, part of the moisture within the pore space of the Tuffeau can be absorbed by the capillary action into the Sebastopol stone. Therefore, even if capillary suction is higher in the Tuffeau (suction generated by smaller pores), water can be transferred from the Tuffeau stone to the Sebastopol stone during the drying stage via the contact surface, since the Tuffeau is never completely dry (Table 3). Water absorbed by Sebatopol contains NaCl salts, which results in the reduction of salts in Tuffeau. This is confirmed by the measurements of salt content after the 15th cycle; the NaCl content is higher in the Sebastopol stone than in the Tuffeau (1.131 compared to 1.086) and this figure is even higher for the isolated case (1.131 compared to 1.082).

Regarding deterioration by Na_2SO_4, the Tuffeau disaggregates very quickly during the 8th cycle. The remnant of the Tuffeau cube is maintained with the cube of Sebatopol stone by the strings of connection. The Sebastopol stone continues to disaggregate uniformly on the surface (Fig. 4). After

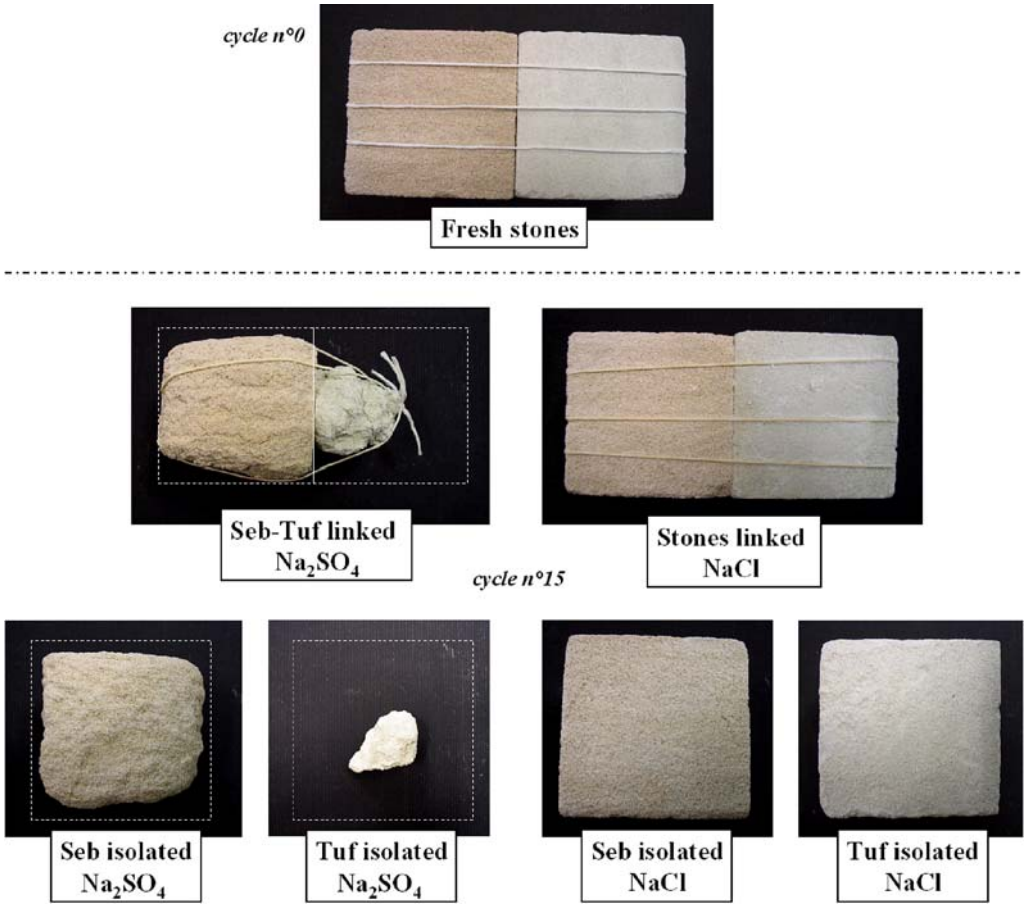

Fig. 5. 'Sebastopol/Tuffeau samples linked together' after 4 cycles of immersion/drying in the case of Na_2SO_4 and NaCl crystallization.

15 cycles, the weight loss of the Sebastopol stone linked to the Tuffeau (normalized weight: 0.785) is less than for the isolated Sebastopol stone (normalized weight: 0.688). The stone therefore deteriorates less when it is in contact with the Tuffeau. Indeed, according to Figure 4, the erosion of the surface close to the face in contact with the Tuffeau is less than for the remaining parts of the sample. The alteration of the Tuffeau is somewhat slowed and that of the Sebastopol is slightly reduced. The replacement of Tuffeau by Sebastopol can be advantageous to construction as the latter is more resistant to deterioration processes including sodium sulphate or sodium chloride.

Concluding remarks

The aim of this study was to analyse the behaviour and evaluate the durability of two limestones subjected to an accelerated aging test. As the normalized aging test is conducted with sodium sulphate (Na_2SO_4), we carried out a similar accelerated test with sodium chloride NaCl. Finally, the two previous tests were also carried out on a set of two stones (Tuffeau and Sebastopol) in order to determine the compatibility between the two stones. The main result is that the quantity of sodium chloride salt stored in the Tuffeau is higher than that of sodium sulphate. This stone, however, appears to withstand sodium chloride cycles. The durability of the Tuffeau stone face to sodium sulphate is limited as samples were completely damaged during the 8th cycle. This behaviour can be attributed to salt crystallization pressure and thenardite/mirabilite phase transition generated by sodium sulphate during immersion-drying cycles. An additional property of sodium chloride can also explain the reaction of stone: indeed' the water absorption capacity of the NaCl salt is higher than

that of the Na_2SO_4. During the drying cycle, samples are not completely dry and the crystallization pressure is lower than that for sodium sulphate. A drying stage of 20 hours duration is clearly insufficient in the case of the Tuffeau, in particular with sodium chloride.

Sodium chloride salt was accumulated more than sodium sulphate in the Sebastopol stone. However, durability of Sebastopol stone seems to be better than that of Tuffeau with the two salts. Less damage was observed with sodium sulphate. This behaviour can be explained by the difference between the two stones in the mineralogical composition and in the pore space distribution, inducing different water transfer properties (permeability, capillarity and water retention). Sebastopol stone samples are always completely dry at the end of drying cycle which is not the case for the Tuffeau samples. This demonstrates that water transfer in Sebastopol stone is much faster than that in Tuffeau (Beck & Al-Mukhtar 2005). Moreover, pores in the Sebastopol stone are large and so harder to block (obstruct) by the salt. Therefore, at the end of a drying cycle, the pores of the Sebastopol stone are practically dry while the pores in Tuffeau stone are still humid.

The normalized test (EN 12370 standard) seems to be very destructive for the soft limestones over a small numbers of cycles (about only five in the case of Tuffeau stone); it therefore cannot be used to reveal the behaviour under realistic environmental conditions. The same test carried out with NaCl allows us to track more accurately the response of the stone throughout the test. Moreover, the alteration type during these ageing tests using salt crystallization is always granular disintegration. This is a natural (*in situ*) type of alteration of these stones, but not the unique type for the case of Tuffeau stone (which also experiences exfoliation, flaking, fissuring and alveolar).

The ageing tests carried out on the two types of stone gives some indication of compatibility. The use of sodium sulphate quickly induces an important alteration in the set, mainly displaying granular disintegration in Tuffeau. It is therefore not easy to study the compatibility with this normalized ageing test. The use of NaCl attenuates, or reduces slightly, the alteration of the Tuffeau stone. This can be attributed to water transfer in Sebastopol stone which is very quick. Through the contact surface between the stones, water and salt solution can be transferred from Tuffeau to Sebastopol and then ejected from the set (efflorescences and subflorescences are observed). It can be stated that replacing Tuffeau stone with Sebastopol stone is advantageous as the durability of the set is improved. However, in construction the contact between stones is usually realized by a mortar joint. If the water transfer properties of the mortar are similar to that of the stone, the conclusions presented in this study are also valid. It is necessary to consider the physicochemical compatibility between the mortar and the stone for the restoration works, however (Beck & Al-Mukhtar 2008).

Because the damage depends on the type and the quantities of salts in the stone as well as the water transfer properties (permeability, capillarity and water retention), other accelerated weathering tests must be developed to evaluate the localization of salts during immersion-drying cycles (Topal & Sozmen 2003; Angeli *et al.* 2007), imbibition-drying (Benavente *et al.* 2001; Van *et al.* 2007*a*) and relative humidity cycles (Van *et al.* 2007*b*).

More characterization tests must be planned such as X-ray diffraction, mercury intrusion porosimetry and scanning electronic microscope in order to determine the mineralogical and pore space changes during ageing tests. The microstructure and rock fabric of the two stones strongly affect the durability and the nature of the deterioration, and can influence the phenomenon of compatibility.

The authors would like to express their thanks to the quarry Lucet (St-Cyr-en-Bourg, France) for providing white Tuffeau and the quarry Rocamat (Saint-Maximin, France) for providing Sebastopol stone. Thanks also to Sami Al-Mukhtar who conducted some of experiments presented in this paper.

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