

Experimental characterization of the deterioration of masonry materials due to wet and dry and salt crystallization cycles

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Abstract. Considering that by 2050 the effects of climate change around the world will cause extreme conditions for the built environment, particularly for historic masonry constructions, it is important to understand in more detail the effect of different environmental conditions on the durability of masonry of stone. In this work, an experimental characterization of the deterioration process of the stone masonry submitted to wet and dry and salt crystallization cycles is presented and the results are discussed. For this, the effect of these conditions on the mechanical behavior under compression of stone masonry was evaluated. For the tests, samples composed of two and three units respectively were adopted. The experimental campaign was divided into two phases: (1) water absorption cycles by capillarity (clean water and water with a concentration of 3% and 10% sodium chloride) and drying cycles in samples at 60°C. Sodium chloride was selected because it is often found in old buildings subject to cycles of humidity. For this, a special setup was designed to perform the immersion and drying of several samples at the same time, controlled by moisture and temperature sensors; (2) mechanical characterization of material and masonry assemblages under uniaxial compression tests after the cycles. From these experimental tests it was possible to evaluate the effect of the exposure conditions on the mechanical properties of the materials and masonry assemblages.

Keywords: weathering, characterization, stone masonry, wet and dry cycles, salt crystallization.

1 Introduction

Stone is a material traditionally used in construction due to properties such as its hardness, strength, durability and aesthetic value. The behavior can change with time due to weathering, which influences the petrographic characteristics, texture and porosity [1]. The same trend can be caused by the adaptation of internal elements to the atmospheric conditions under the action of physical and chemical factors, which can go against the physical integrity and the mechanical characteristics of the material and the structure.

In the scope of the preservation of the built heritage, it is important to understand the influence of these actions on the behavior of stone masonry as a system composed of units and mortar. It is necessary to consider that the degradation is mainly due to the decomposition of the material that means the alteration of its internal structure, which can result from the gradual disaggregation. This process can be delayed with restoration interventions and with the maintenance of the buildings, but that cannot be avoided. The deterioration of the masonry may be due to the presence of minor minerals in the composition of the material that negatively influence its resistance

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to alteration. But it is the porosity, which is certainly the most important intrinsic factor against the variation, since the pores are able to transfer fluids and it is in the solid-fluid contacts where reactions as harmful as the crystallization of hygroscopic salts take place. These salts enter the porous matrix and precipitate when the water evaporates due to the increase in temperature. The problem arises when the salt crystallizes and increase in volume and causes a great internal tension that often exceeds the mechanical resistance of the material, generating destructive action such as the weakening of the internal structure and the separation of its parts. This process called crypto-florescence causes cracking, crumbling and pulverization in historical construction [2]. Furthermore, when the liquid flow evaporates outer the surface of the material and transports the salt out of the element, the efflorescence process takes place, characterized by the accumulation of a whitish powder or filaments that not only aesthetically damage the building but can also cause the detachment of the material surface [3]. This phenomenon has been progressively recognized at a scientific level as a process of damage and is currently considered one of the most powerful mechanisms of deterioration of porous materials in masonry constructions [4].

An important example is the deterioration of calcarenite stones in the temple called Casa de Venus, located in Morocco, a case where halite (NaCl) and gypsum are the main identified salts, as they can be found in almost all environments: urban, rural and coastal [5]. Halite (NaCl) is mainly related to seawater, marine aerosols and deicing salts. Gypsum is mainly associated with air pollution [6], biological colonization or with incompatible cleaning and preservation products, mortars and plasters. The most relevant salts in the deterioration process are those with sulphate, nitrate, potassium, sodium, magnesium and chloride ions [7]. Chloride is considered the greatest cause of decay when the monument is in coastal proximity.

The present investigation has as the main objective to evaluate the influence of wet and dry and salt crystallization cycles on the physical and mechanical properties of masonry materials and masonry assemblages. To this goal, specimens of granite joined by lime mortar were constructed, which were physically and mechanically characterized at their original state and then subjected to a deterioration process associated with wet and dry and salt crystallization tests. Finally, the influence of the induced damage on the mechanical properties of lime mortar, granite and granite masonry under compression was analyzed.

2 Materials and testing methods

2.1 Introduction

The first part of the investigation was based on the experimental mechanical characterization of stone materials characteristic of stone masonry commonly used at northern region of Portugal, namely granite and lime mortar. A first step was to characterize the masonry materials before submitting them to the effect of wet and dry or salt crystallization tests. From this experimental characterization, mechanical properties were found to be compared to the ones obtained in specimens submitted to dry or salt crystallization tests. The lime mortar selected was a

commercial premixed mortar, composed of natural white lime, hydraulic binder and classified sands. The granite is from Minho region of northern Portugal, being the most used material in the construction of old buildings of monumental or vernacular architecture. It is a granite of two micas with a porphyritic tendency [8] due to the presence of Feldspar crystals in a background mass constituted by quartz, feldspars and micas. It is a fine to medium grain size granite and the yellow color indicates certain degree of weathering.

Masonry materials were submitted to different tests for physical and mechanical characterization. Capillary water absorption and porosity tests were carried out in mortar prisms of $160 \times 40 \times 40 \text{ mm}^3$ and in granite cubes of $70 \times 70 \times 70 \text{ mm}^3$ in order to evaluate their susceptibility to water, which represent one of the main sources of stone deterioration. In addition, ultrasonic pulse velocity (UPV) was measured to evaluate and correlate physical characteristics and mechanical properties [8]. The mechanical characterization of the lime mortar was carried out based on flexural and compression tests at different ages, namely 7 and 28 days. In the case of granite compression tests were carried out on cubic granite samples from which it was possible to obtain the compression strength.

The experimental campaign included also compression tests on masonry prisms built with granite stone and lime mortar previously described. The masonry prisms were built with three granite blocks joined by a 10 to 15 mm joint of lime mortar with dimension $200 \times 150 \times 480 \text{ mm}^3$. The specimens were tested under uniaxial compression to obtain the reference values of the compressive strength and elastic modulus and compare them with the values obtained in masonry prisms submitted to wet and dry and salt crystallization tests. The physical and mechanical properties of granite and mortar are summarized in Table 1. In addition, the reference of the European standards used in the experimental testing are also shown. From the results, it is possible to observe the physical properties of mortar are in line with the values provided by the manufacturer, but the compressive resistance is higher than the values provided. The granite revealed to a porous physical structure with low resistance to compression, reflected in a low value of ultrasonic pulse velocity and a high variability, when compared to a close granite studied by Vasconcelos (2005) [9].

Table 1 – Values of the physical and mechanical characterization of lime mortar and granite

	Standard	Lime mortar	Standard	Granite
Capillarity coefficient	EN 1015-18 (2002)	0,40 kg/(m ² .min ^{0.5})	NP EN 1925 (2000)	0.373 g/cm ² .h ^{0.5}
% Porosity	EN 1015-10 (1999)	22.4%	EN 1936 (2006)	8,9%
UPV (m/s)	ASTM C597 (2002)	2762 m/s	ASTM D 2845 (2005)	2331.69 m/s
compressive strength	EN 1015-11 (1999)	10.92 MPa	EN 1926 (2000)	36 MPa
flexural strength	EN 1015-11 (1999)	3.25 MPa	-	-

2.2 Wet and dry and salt crystallization tests

The wet and dry cycles and the salt crystallization tests were carried following some suggestions of the European standard NP EN 12370 (2001) for obtaining the resistance to the crystallization of salts in stones with more than 5% porosity. The wet and dry and salt crystallization tests were carried out on cubic granites, prismatic

specimens of lime mortar and on the masonry prims. For this, besides the clean water, it was decided to dissolve sodium chloride in two concentration, namely 3% (close to the salinity of the water sea) and 10%, clearly higher than the salt concentration of the sea water. The decision for this type of salt was that it is commonly seen in efflorescence in ancient stone constructions and is easily available. Figure 1 shows the setup used for the wet and dry and salt crystallization tests.

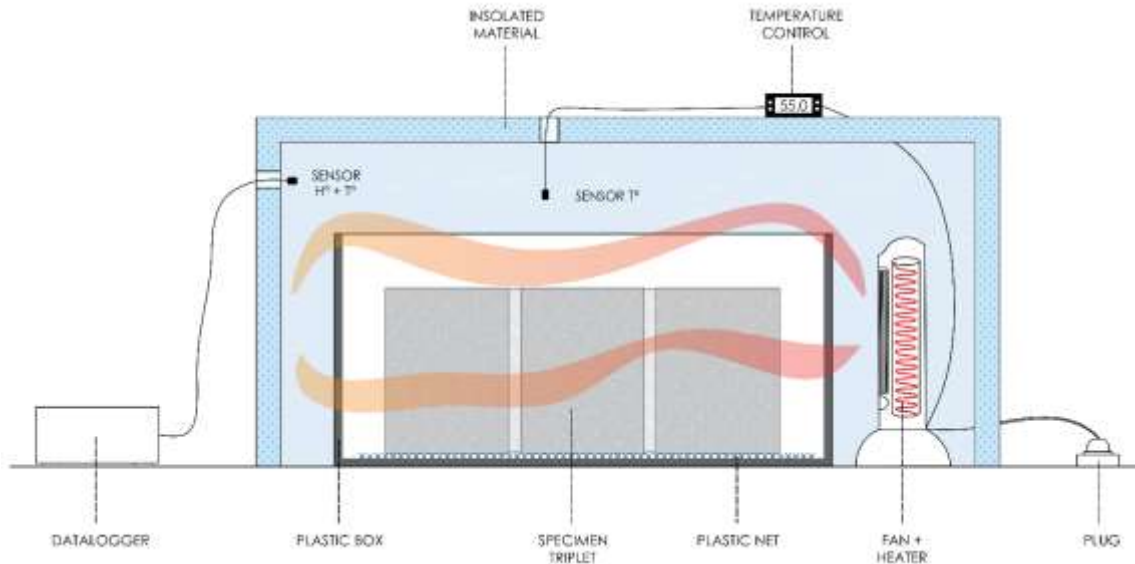


Figure 1. Setup of the wet and dry and salt crystallization tests

The wet and dry cycles were composed by two phases: (1) wetting phase: 3 boxes have the tests specimens immersed in a 2cm height solution that has salt concentrations of 0%, 3% and 10% respectively. The initial value of the temperature was set at 23 ° C while the ambient humidity was $68 \pm 2\%$. During this 8-hour phase, the specimens absorbed the solution by capillary, meaning that the salt ions are transported in the diluted solution and can be introduced into the materials and circulate through their pores and cavities. At the end of the immersion, the water was removed from each container and then the containers were cleaned with a cloth, avoiding the accumulation of salt; (2) drying phase: each group of test specimens is contained by small greenhouse of $900 \times 1500 \times 700 \text{mm}^3$ that was designed in expanded polystyrene and fulfilled the function of an oven. The temperature of the inner environment of the greenhouse was controlled due to the installation of heating devices. An air heater, consisting of a fan and a coil homogenize the internal environment temperature at around 60 ° C since it was controlled by a sensor located at the center of the greenhouse. From this sensor, it is possible to automatically turn off and on the fan depending on the temperature variation. In parallel, another sensor connected to a datalogger, recorded the temperature and humidity of each stove simultaneously during the 32 hours of this phase, reaching to register a decrease in relative humidity to 20%. The internal environment control system is turned off with the aid of a timer to start the cooling stage of the test specimens during the following 6 hours in order to prevent the temperature change from affecting the process of the next cycle. Each cycle lasted 46 hours, making a total of 276 hours after which the specimens were ready to be tested destructively.

3 Results and discussion

After the durability cycles, the visual inspection of the specimens was performed. In general terms, during the wetting phase, it was noted that the capillary uptake of water in masonry occurred with greater speed in the lime mortar joint than in the stone blocks, which may be due to the difference between the absorption coefficients by capillarity. A slight detachment of binder was also observed in all the specimens, as a slightly dense whitish liquid on the surfaces. Halite tends to precipitate at the air-solution interface, resulting in efflorescence on the outer surface. Based on past experiments, it was observed that this salt shows a slow rate of precipitation and growth, which results in lower crystallization pressures, that is, lower damage capacity of sodium chloride when compared with more harmful salts such as sulfates [10]. This does not mean that sodium chloride does not cause damage, because the rapid evaporation of water by the change of temperature can lead to the growth of subflorescence, since it tends to occur beyond the drying front (in depth) promoting a greater supersaturation and therefore, higher crystallization pressures.

3.1 Visual assessment: efflorescence formation

3.1.1 Efflorescence in prisms of lime mortar of 40x40x160mm

The samples of lime mortar after wet and drying cycles present a change in hue, ranging from a light cream color to a darker and yellowish color for all three concentrations studied. For the samples placed in 3% and 10% NaCl solutions, the efflorescence was visible from the third cycle as a thin and discontinuous layer of white particles, which evolved throughout the six cycles to become settlements with appearance of "fluff" without compaction, fragile to the touch, see Figure 2. This characteristic shape grows from an almost dry surface, which receives the solution in a punctual manner through the substrate forming very thin and elongated acicular crystals called "whiskers" [11].



Figure 1. Lime mortar specimen subjected to 6 cycles in water with 10% NaCl concentration

Another important consequence is the presence of micro cracks. In reference to the subflorescence, Figure 3 shows a comparison at two different scales under the lens of the microscope, the inside of the mortar specimens affected by the solution of higher salt concentration (10% NaCl) after the 6 cycles. The image amplified by 100x shows traces of salt crystals nested in the pores with greater dimension, and the image amplified by 400x shows the existence of internal "whiskers" on a layer of smooth and crystalline appearance.

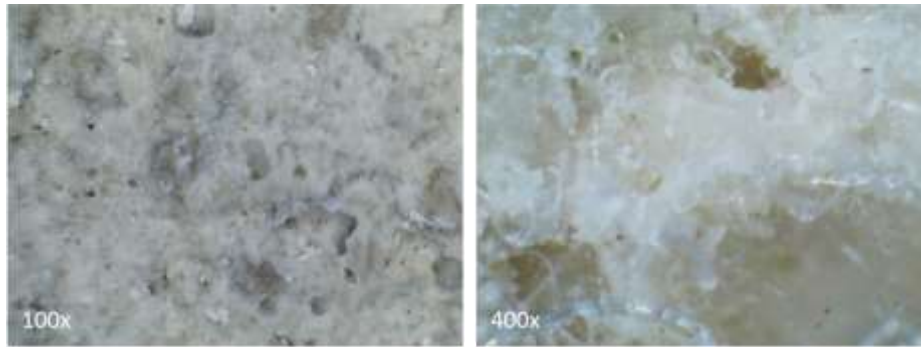


Figure 2. Amplified images of the interior of the lime mortar subjected to 6 cycles in water with 10% NaCl concentration

3.1.2 Efflorescence in masonry specimens

The masonry specimens placed in the solution with 3% NaCl showed a slow advance of precipitations evident from the third cycle, as spots existing in the upper two thirds of the block. Regarding the specimens placed in 10% NaCl water concentration, the first evidences of efflorescence were seen at the end of the first cycle (46 hours). Their evolution on the specimen shows the consolidation in several layers of the efflorescence located in greater concentration in the center of the specimen between 7 and 10 cm in height in the ascending direction. In addition, a concentration of precipitations was recorded in the mortar-block interface and a slight wear of it, as shown in Figure 4.



Figure 3. Visual aspect of masonry after the crystallization cycles on 10% NaCl water concentration; (a) duplet specimens; (b) triplet specimens

As for the triplets of $200 \times 150 \times 480 \text{ mm}^3$, the efflorescence was present in the 3% NaCl samples at the end of the fourth cycle, while in the 10% NaCl samples a slight layer of salt was observed finishing the second cycle. Figure 5 shows that at the end of the sixth cycle in the samples at 10% NaCl, a less dense but homogeneous layer was formed on the top of the specimens, probably due to the size of the sample as it increased the distance to the front of precipitation.

3.2 Effects of cycles: mechanical properties

3.2.1 Mortar

After the wet and dry cycles, it was observed a mass loss of 3.22% in the 0% NaCl mortar specimens while the specimens immersed in 3% saline solutions and 10% NaCl gained between 2% and 9% mass respectively.

The capillary coefficient increased by about 65% for the test pieces with 3% and 10% NaCl content, while the UPV values decreased as the NaCl concentration solution increased, which appears to reveal a greater deterioration of its internal structure. Regarding the mechanical resistance to bending, a 30% decrease was observed for the three types of salt content, while the compressive strength decreased by 15% in the specimens tested in pure water and by 30% in the specimens tested concentrations of 3% and 10% NaCl.

3.2.2 Stone

The analysis of results on the uniaxial compression tests carried out on cubic specimens shows that the compressive strength for the concentration of 3% NaCl decreased by 32%, while for the concentration of 10% NaCl increased by 8%. Regarding the modulus of elasticity (E) there was a decrease of 60% for the samples to 3% NaCl and of 40% for the samples to 10% NaCl. This phenomenon can be justified by the fact that the granite used in the tests showed a coefficient of variation of between 50 and 60%, since an inverse relationship was expected between the increase in salt content and the reduction of the mechanical properties of the granite.

3.2.3 Masonry

The masonry prisms tested in uniaxial compression [8] show a reduction in the mechanical properties, namely compressive strength and modulus of elasticity with respect to the reference specimens. The data shown in Figure 5a shows a decrease on the compressive strength in masonry prims for increasing salt concentration. A reduction of about 25% was recorded in the masonry specimens placed in the 10%NaCl concentration solution. Regarding the variation of the mean values of the modulus of elasticity (Figure 5b), it is seen that a general decrease is observed for the three groups of test pieces compared to the reference module of between 15 to 20%. It is observed that the test pieces subjected to wet and dry cycles in 0% NaCl showed a significant decrease on the elastic modulus when compared to the reference masonry prisms. The masonry prims subjected to wet and dry cycles in salty concentration exhibit also a reduction on the elastic modulus but slightly lower when compared to the previous specimens. The salt concentration appears also not to have a significant influence on the modulus of elasticity.

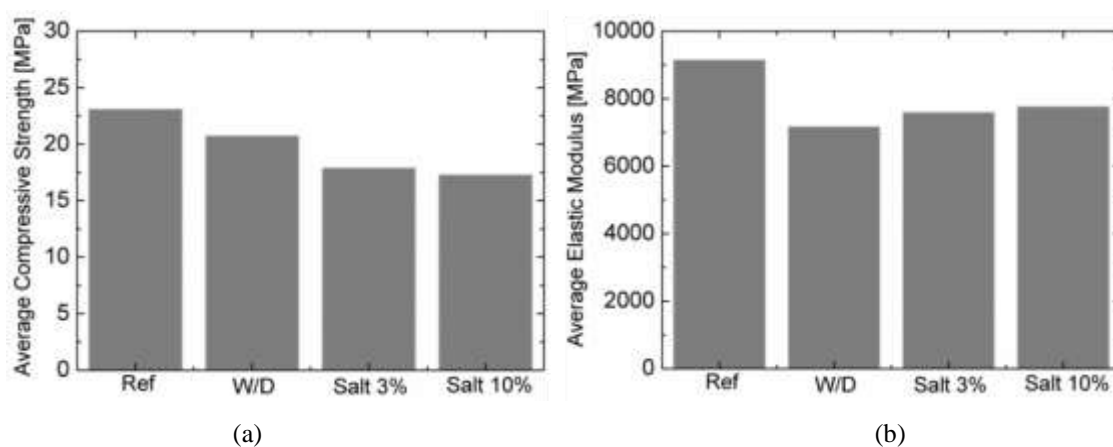


Figure 4. Comparison of mean values of the mechanical characteristics of the masonry prism before and after cycles
(a) Compressive strength (b) Elastic Modulus

4 Conclusions

Cyclic wet and dry and salt crystallization tests carried on masonry materials and masonry assemblages were designed to evaluate the influence on the wet and dry and salt crystallization on physical and mechanical properties of materials. A test setup was designed to simulate the uptake of three concentrations of sodium chloride in water, namely: (i) water with 0% NaCl, (ii) water with 3% NaCl and (iii) water with 10% NaCl. After 6 cycles of wet and dry of different material specimens it was observed the growth of salt clusters inside the material that precipitated on the surface forming efflorescence usually located in the upper part of the specimens (evaporation front). It could be seen that the degree of damage to the stone caused by the internal pressure of the salts is due in part to the relationship between the supersaturation of the solution and the area where the crystallization occurs. It was observed that after 6 cycles, the mortar showed a reduction in the compressive and bending strength and an increase on the physical properties (capillarity, porosity) as a consequence of the deterioration due to wet and dry cycles. The masonry prisms also showed a reduction on the compressive strength and elastic modulus after the wet and dry crystallization tests.

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6 References

- [1] Alonso, F., Esbert, R., & Ordaz, J. (2006). Análisis del deterioro en los materiales pétreos de edificación. *Recopar Revista electrónica* n°3, 2-10.
- [2] Andriani, G. F. (2007). The effects of wetting and drying, and marine salt crystallization on calcarenite rocks used as building material in historic monuments. *Geological society*, 271, 179, 180.
- [3] Gentilini, C., Franzoni, E., Bandini, S., & Nobile, L. (2012). Effect of salt crystallization on the shear behaviour of masonry walls: An experimental study. *Construction and Building Materials* 37, 181-189.
- [4] Gonçalves, T. C. (2007). Salt crystallization in plastered or rendered walls. Lisboa, Portugal: Universidade Técnica de Lisboa.
- [5] Zaouia, N., Elwartiti, M., & Baghdad, B. (2005). Superficial alteration and soluble salts in the calcarenite weathering. Case study of almohade monuments in Rabat: Morocco. *Environmental Geology* 48, 742-747
- [6] Beck, K., & Al-Mukhtar, M. (2010). Weathering effects in an urban environment: a case study of tuffeau, a French porous limestone. *Geological Society, London, Special Publications* 331, 103-111.
- [7] Maurício, A., Pacheco, A., Brito, P., Castro, B., Figueredo, C., & Aires-Barros, I. (2005). An ionic conductivity-based methodology for monitoring salt systems in monuments stones. *Journal of cultural heritage*, 6, 287, 288.
- [8] Graus, S. (2017). Weathering of stone masonry due to wet - dry and salt crystallization cycles, Master thesis, University of Minho, 2017.
- [9] Vasconcelos, G. (2005). Experimental investigations on the mechanics of stone masonry: Characterization of granites and behavior of ancient masonry shear walls. Guimarães, Portugal: Tese de Doutoramento em Engenharia Civil, Universidade do Minho.
- [10] Alves, C., Vasconcelos, G., & Ferreira, R. (2014). Salts in stone and masonry. *Advanced Masters in Structural Analysis of Monuments and Historical Constructions, SA6: Restoration and conservation materials*, págs. 2-50.
- [11] Arnold, A., & Zehnder, K. (1990). Salt weathering on monuments. *First International Symposium on the Conservation of Monuments in the Mediterranean Basin*, 31-58.