RESEARCH PAPER

Assessment the stones compatibility based on salt weathering tests

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A B S T R A C T:
The current study aims to assess the compatibility between tuffeau and Richemont stone exposed to salt weathering, the most frequent physical-mechanical weathering process. Different experimental tests, both in macro- and in micro- scale, were performed on the aged stone samples underwent 20 wetting-drying cycles using salt solution of sodium chloride. The results of the aged samples were then compared with the already results belonged to the fresh samples. In the accelerated ageing tests the stone samples were situated in two different methods: the isolated stone samples and the pair of stone samples, i.e. one tuffeau sample linked to one Richemont sample. The linked test method was adopted to simulate the in-situ situation of the stones. The main results show that the microstructural characteristics of the stone (pore size distribution, water transfer properties and tensile strength) strongly reflect the stone resistance versus the salt weathering. Moreover, the results indicate that the integrity of tuffeau stone samples increased when linked to the Richemont stone samples referring to the presence, in partial way, the compatibility between the two stones. However, the limited conditions of the salt crystallization test (i.e. only 20 wetting-drying cycles) were not sufficient to detect the complete behavior of the two stones towards the salt weathering. Therefore, the current results confirm that the compatibility assessment between the stones cannot be judged based on the results presented in this study.

KEY WORDS: Compatibility; salt weathering; porous limestone; tuffeau; Richemont.
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INTRODUCTION:

Recently, people witness a sophisticated and complex life that is producing many different kinds of pollutants and negatively affects the daily life, not only on the living organisms, but on all that the earth contains. With regard to the study of built heritage, including both the archaeological sites and the historical buildings, the different types of pollution, no doubt, it will affect the continuity of the beauty and the integrity of these buildings and pose a great threat. In this point of view, the preservation, the restoration and the maintenance of ancient buildings are the fundamental and the inevitable demand, especially as these landmarks are of the utmost importance and of the significant resource for the economies all over the world.

The attention to the built heritage required many methodologies that can be included in the five sequenced stages: assessment the building condition, diagnosing damage state of the buildings, searching for the causes to the building damages, finding solutions to the current problems
and finally the possibility of creating a platform of the prevention for future problems. Off course, each stage represents a major challenge for the scientists whose are interested in the field of restoration works. Concerning the "finding solutions to the current problems" stage, where the treatment become difficult to be addressed for the building materials that suffer from too badly cases such as failure or breakage, the new alternative building materials are usually used. Herein, highlights should be given to the importance of the term compatibility between building materials. The restoration and even the reconstruction of the built heritage require the search for new building materials that are compatible with the original ones and should, at least, not add additional future problems. In the last two decades, the researchers have addressed the concept of the compatibility between the building materials and they tried to explain its principles, fundamentals and requirements (Jefferson et al., 2006; Groot and Maurenbrecher 2016). However, some studies have also examined the possibility of achieving the compatibility between building materials (Schueremans et al., 2011; De Kock et al., 2015; Borsoi et al., 2017). Furthermore, other researchers have highlighted to the problems resulting from the use of incompatible building materials (López-Arce et al., 2009).

The presence of salts inside the stones can be sourced by: salts from the ground that are transferred via the capillarity, the preexist salts found in the stones during the geological formation of these stones, the salts coming from the marine salt spray for coastal regions, the salts remaining on the stone surfaces by the droppings of birds and bats, environmental pollution in urbanized cities. Thus, it is well accepted that the salt weathering plays an important role in the degradation of the stones (Cardell et al., 2003; Silva and Simão 2009; Sun and Zhang 2009).

The salt produces damage to the stones by one of the following three main mechanisms: salt crystallization (Zedef et al., 2007; Ludovic-Marcques and Chastre 2012; Modestou et al., 2015), hydration pressure (Winkler and Wilhelm 1970; Charola and Weber 1992), and thermal expansion (Wang and An 2016; Gonçalves and Brito 2016). In this research work the accelerating salt weathering test was performed on two porous stones. This was done in order to study the compatibility between these two stones. The Chambord Castle (XVI century AD, located in the Loire Valley-center region of France) represents the studied case adopted in this paper. The castle suffers from deterioration in its main construction building material, tuffeau stones, as stone detachment in the form of stone spalling. At the beginning of the 20th century, the Chambord Castle was restored, and the Richemont stone was used as an alternative stone instead of the original ones. Later, the castle has undergone numerous restoration works and has been included on the UNESCO’s list since 2000. The current research aims to assess the compatibility between tuffeau and Richemont stone based on the accelerated salt weathering tests. This is done in an attempt to investigate the possibility of using the Richemont stone in the future restoration work.

1. MATERIALS

Two calcareous stones widely used as traditional building materials in the architectural heritage-central of France were considered in this study; tuffeau and Richemont stone. Tuffeau stone was well studied by (Beck 2006). Tuffeau is of Turonian age from quarry of Usseau in Vienne close to the Loire River (NW France). This stone formation is very soft and highly porous limestone, thus, in Latin etymology, it could be called as "tofus" referring to spongy meaning. Tuffeau, with white color, is a siliceous fine-grained limestone formed by different mineral phases (calcite, quartz, opal, and clay minerals). In one quarry, and due to the different condition of the geological deposition, tuffeau stones could be varied in their chemical composition, physical features, and even in color.

Richemont stone is more recently studied stone (Al-Omari 2014). The Fresh quarried blocks of Richemont stone were obtained from the Charente-Maritime quarry in France (Rocamat Company). Like tuffeau, this limestone is of Turonian age but whitish-beige in color and with better mechanical properties than tuffeau. It is a fine-grained bio-pelmicrite limestone (a nonomineral calcitic). Macroscopically, numerous shell fragments in form of fine irregular short white lines can be observed.

Both stones were already characterized in complementary multi-scale physical, chemical and mechanical approaches [Beck 2006; Al-Omari....]
2014). Tab.1 summarizes the main characterization of these two stones. Based on the stones' characterization it can be said that the two stones are varied in their physico-chemical and mechanical properties. The results in Tab.1 can help us to interpret the results of the current study in relation to the role of salt crystallization in the deterioration of stones and, therefore, evaluating the compatibility of the stones, which is the objective of this research.

2. METHODS

2.1 Salt crystallization cycles

In this study, the European standard (EN 12370, 1999) was followed to evaluate the stone resistance against salt crystallization. Sodium chloride was selected in the preparation of saline solution. During the experiment the cubic fresh stone samples (4 cm side) were subjected to 20 wetting-drying cycles according to three sequential stages: (1) immersing the stone samples inside the saline solution with a concentration of 14% by weight of sodium chloride for two-hour; (2) drying the samples using an oven with 105 °C for 20 hours; (3) cooling the samples at room temperature for 2 hours, Fig.1. During the test, two different methods of the stone samples situation were followed: the first one is the traditional method of testing the stone samples individually; the second one is the new method of testing the pair of stone samples, i.e. one tuffeau sample linked to one Richemont sample. This second method was chosen to represent the situation of the stones in the field (the original tuffeau adjacent to the alternative Richemont stone). For each stone type and for the two test methods, the crystallization cycles were performed three times in order to verify the validity of the results obtained. During the test, the visual appearance of the stone samples was monitored periodically at the end of each cycle; the external surface of the sample was imaged using a digital camera. In addition, in each cycle the stone samples weights were measured twice: after the immersion stage with the saline solution and after the drying stage. This was done in order to follow the change (gain or lose) in weight of the stone samples during the experiment. A digital balance with precision of 0.01 grams was used, while the employed compact digital camera has an optical sensor resolution of 16 Mpix. At the end of the crystallization test, the aged stone samples were underwent the set of complementary tests: x-ray diffraction, salt content by washing, ionic chromatography (IC), and mercury intrusion porosimetry (MIP). This was done in order to provide a full characterization of the aged stone samples. During these complementary tests and for the test method including the pair of stone samples, small pieces were cut at the position near to the linked surface. In this case, the stone samples have been given legends as "linked samples".

2.2 Mineralogical observations

The mineralogical composition of the aged samples was determined by X-ray diffraction (XRD) test using Philips Type PW 1830 Diffractometer. In this test, the analysis conditions were: radiation Cu Kα (λ = 1.5406 Å), 40 kV voltage and 30 mA current intensity. The explored angle area was between 4º and 60º (2θ) with a step of 0.025º and a counting duration of six seconds at each step.

2.3 Salt content measurements

The measurements of salt content of the aged samples were achieved by two different methods: (1) the traditional method by weighing the small pieces of the aged samples before and after the washing process with distilled water; (2) the more precise method by Ion Chromatography (IC) test. In the traditional method, at the beginning the small piece of the aged samples were weighed, then they were desalinated using distilled water until the electrical conductivity of the filtered water was less than 0.5 μS/cm followed by drying at 105 °C and weighed again. The difference in the weights gives the salt content.

In the (IC) test, the ion contents coming from the soluble salts contained in the aged stone samples can be determined. This test was performed by following the Italian standard (NORMAL, 1983). Prior to test, the solutions were prepared by diluting 200 mg of powder in de-ionized water to a concentration of 10 g/l. At room temperature...
condition the solutions were shook continuously for 72 h and followed to the filtration stage using a 0.45 μm membrane. For anions, the eluent was 50 μL EGC-KOH Gradient 10 min with a flow rate of 1.0ml/min at pressure about 1800 Psi. Finally, the quantitative analysis of the soluble salts was performed on a Dionex ICS 900 equipped with AS17 (4x250 mm) and AG17 (4x50 mm) separator column.

2.4 Porous system investigations

The porous system of stone is one of the most important things to investigate when studying the effect of salt crystallization. To accomplish this objective in our study, the characteristics of the porous system for aged samples both of tuffeau and of Richemont stone were detected by the help of the mercury intrusion porosimetry technique using an AutoPore IV 9520 (Micrometrics) porosimeter. The test was performed to measure the pore size diameter in the range of 0.003 - 450 μm by applying the thrusting pressure in the range of 2.76(10)^3 - 415 MPa. The test were carried out on three samples with nearly the same size and weight in order to achieve the standardize testing, the minimize errors, and the insure reproductivity.

3. RESULTS AND DISCUSSION

The first results of this study are related to the measured normalized weights for tuffeau and Richemont stone samples during the salt crystallization test in two different conditions (isolated and linked samples) both in dry and in saturated states, Fig.2. The term of normalized weight refers to the weight ratio between the sample weight at the given stage and the initial dry weight of the stone sample. Fig.3 shows the visual appearance of the aged stone samples during the test. Concerning the isolated test condition both of tuffeau and of Richemont stone, it seems there is an increase in the dry normalized weight up to the 5th cycle. In next another ten cycles there is a fluctuation in this normalized weight. Indeed, during the first five cycles the increase in the dry normalized weights is attributed to the deposition of crystalline salt inside the stone pores and to the efflorescence generation on the sample surface. Nevertheless, between the 5th and 15th cycles, the stone samples exhibit a competition between, on one hand, the loss in weights by powdering for tuffeau and by superficial granular disintegration for Richemont stone, (see Fig.3), and on the other hand, the gain in weights by salt supply. At the test end, the results analysis refers to the significant increase in the dry normalized weight for tuffeau about 27 %. This is not the case for Richemont stone, where throughout the whole test period the gain in weights of the stone samples nearly equal to the loss in its weights as matter loss, Fig.2. At the test end, Richemont stone show decrease in the dry normalized weight ≈ 3 %. This behavior is strongly attributed to the microstructural characteristics of the stone itself: Richemont stone with very low water retention capacity compared to tuffeau (Al-Omari 2014). Here, the drying process is easier and faster and thus the more efficiency of salt crystallization in deteriorating the stone samples (more matter loss).

The results show also that, in case of the isolated tuffeau samples, after the 5th cycle till the test end the difference between the saturated normalized weight and the dry normalized weight remain constant about 10%. This is because tuffeau contains considerable amount of micropores (Beck 2006) which cannot be filled with crystalline salt even at 20th cycle (i.e. the salt supply still in progress). While for the isolated Richemont stone samples, this difference in the normalized weight was about 5% between the 5th cycle and the 15th cycle. Then the difference become less and constant about 2.5% till the last cycle. This means that most the pores contained in Richemont stone have been filled with the crystalline salt (i.e. the early finish of the salt supply). In relation to the second test condition (the pair of the stone samples: tuffeau cube linked to Richemont cube), from logic point of view the behavior of the normalized weight, for both the dry and the saturated conditions, represents the mid-state for the isolated stone samples. The results of the normalized weight, therefore, did not provide important evidence for the difference in the behavior of the stone samples during the two test conditions adopted in this study (i.e. the compatibility characteristics are not verified). However, the micro behavior of the stone samples at these two test conditions will be detected through analyzing the result of the other tests (X-ray diffraction, MIP, and IC) as presented in the following paragraphs.
Examining the existence of the sodium chloride salt (halite) in the aged stone samples was done using the X-ray diffraction test. The data refer that the halite is contained in all the aged stone samples. Also, the X-ray diffraction test was employed to examine the washed stone samples free from the halite. The analysis of results confirms that there is no halite in the washed stone samples. This is in turn revealing to the completion of the washing process and confirms the tests of the electrical conductivity referred to earlier. Therefore, the method of salt washing is applicable to investigate the salt content in the aged stone samples.

The salt content was also obtained from the IC test. This was done by carrying out simple calculations involving the molecular weights both of chlorine ions and of compounds produced by crystalline salinity (i.e. the halite). The concentrations of chloride ions obtained from the IC test for the aged stone samples, expressed in mg/l were as follows: (tuffeau-isolated 1162.2; tuffeau linked 979.8; Richemont-isolated 335.5; and Richemont-linked 395.6). It should be noted that the percentage of halite content discovered either by washing method or by IC test method were very similar, which confirms the validity of these results. The calculated percentages of halite by washing method are: (20.03% for tuffeau-isolated; 17.52% for tuffeau-linked; 5.60% for Richemont-isolated; and 5.91% for Richemont-linked). By using the IC test method, while, these percentages were found 19.16, 16.15, 5.53 and 6.52 % for the aged stone samples of tuffeau-isolated, tuffeau-linked, Richemont-isolated and Richemont-linked, respectively.

The results of MIP test for the aged stone samples, examined at two different test conditions (isolated stone samples and the pair of linked stone samples), as well as for the fresh stone samples were presented in Fig.4. Moreover, Tab.2 lists the data of the pore size distribution. It should keep in the mind that the results of MIP test for the fresh samples were taken from the previous studies (Beck 2006; Al-Omari 2014). The results refer the fresh stones have different pore size distribution. Tuffeau has a bimodal pore distribution with the wide range of pore diameter sizes from 20 μm to less than 0.01 μm. More than half of the observed pores are in the range (1.0-10) μm. This can be attributed to the homogeneous size of the main grains (opal in the form of spherules about 10 μm in diameter (Beck 2006). Richemont stone has the largest pores with a submodal distribution of pore size. Although, most of the pores discovered in this stone are located between 0.1-10 μm, Richemont stone like tuffeau contains the same proportion of the pores in the range 1.0-10 μm (Al-Omari 2014). In general, the data in Fig.4 revealed that there is no difference in the behavior (shape and orientation) of the curves both for the aged stone samples and the fresh ones. Again, Tab.2 lists the data of the pore size distribution for the aged stone samples both in the isolated state and in the linked of pair samples state. These data can be compared with already data of the pore size distribution for the fresh stone samples. It is clear that there are significant changes in the proportions of pore size distribution for the tuffeau stone samples in pre- and post-crystallization tests. While, the Richemont stone samples showed no significant differences. These results confirm the data presented in Fig.4 where the greatest differences in the incremental pore volume occurred for tuffeau stone samples rather than Richemont ones.

One of the key aims of this study is to find the compatibility, if any, between tuffeau and Richemont stone. Thus, in the rest of this study attempts are paid to make a comparison in the results of the pore size distribution for the aged stone samples underwent two different test conditions adopted herein (situation of the stone samples whether isolated or linked). This is will done through the calculation of the crystallization pressure and the salt susceptibility index.

In the crystallization pressure calculation, the methodology presented by La Iglesia et al., (1997) was followed and the crystallization pressure is calculated using Eq.(1):

$$P_s = 2\sigma \left(\frac{1}{r} - \frac{1}{R}\right)$$ (1)

Where σ is the interfacial tension of the salt-solution, r is the smaller pore, and R is the larger pore. For only one brine composition salt-solution interfacial tension so the crystallization pressure could be compared for the different aged stone samples. Based on the pore size distribution for obtaining r and R, and 40 N/m for σ (NaCl), the
calculated crystallization pressures expressed in MPa are: Tuff.-isol. 27.8; Tuff.-linked 15.7; Rich.-isol. 4.3; and Rich.-linked 4.5. It is obvious that tuffeau stone sample is enhanced (with lower crystallization pressure) when linked with Richemont stone.

The role of pore size distribution in obtaining the SSI was investigated by applying the procedure innovative by Yu and Oguchi (2010), Eq.(2):

$$SSI = (I_{PC} + I_{PM0.1})(\frac{P_{m5}}{P_c})$$

Where $I_{PC}$ is the index of total connected porosity, $I_{PM0.1}$ is the index of microporosity of pores smaller than 0.1 μm in radius, $P_c$ is the total connected porosity, and $P_{m5}$ is the microporosity of pores smaller than 5 μm in radius. Returning to the data of mercury intrusion porosimetry test, both the microporosities $P_{m0.1}$ and $P_{m5}$ can be calculated, the indices of these microporosities can be obtained from the related tables (Yu and Oguchi 2010). Accordingly, the aged stone samples: Tuff.-isol, Tuff.-linked, Rich.-isol and Rich.-linked have the SSI values as 11.9, 10.7, 7.7 and 8.7, respectively. Again, the linked tuffeau behave more positively against the salt susceptibility compared with the isolated one.

Tab.3 gives a simple way to compare the stone sustainability against salt weathering at two different test conditions, for isolated/linked stone samples situations. For all the investigated parameters, the stone samples of isolated tuffeau are less resistance to salt damage than the linked ones. In the case of Richemont stone, the stone samples did not show differences in the alteration degree when they are being in the situation of isolated or of linked. Exceptions in the weight loss and appearance: the stone samples of tuffeau, whatever the isolated or the linked, showed the same visual appearance. At this end, the crucial question arises: why the resistance of the tuffeau stone samples enhanced, partially, when linked to the Richemont stone sample? To give the answer: it is, firstly, required to define the three stages through which the stone samples pass during the salt crystallization test (Angeli et al., 2007): stage I, the increase in the samples weight due to salt supply; stage II, the fluctuation in the samples weight resulted by the competition in salt supply and matter loss; stage III, the excessive decrease in the samples weight against the salt supply. Backing to our stones, tuffeau is characterized with very high porosity ≈ 45%, it is with high water retention capacity (Beck 2006), thus the first stage could continue for long time, starts at the 1st cycle and not finished up to the test end. This phenomenon is applicable for both the isolated and the linked stone samples. In case of Richemont stone the story is different. The salt supply stage, considering Richemont stone with the lower porosity ≈ 27%, was completed between the 5th to 9th cycles and followed the second stage till the test end, Fig.2. Therefore, in this study, the salt crystallization test that lasted only 20 cycles was not sufficient to reach, at least for the Richemont stone, the third stage, as well as the lack of access to both the second and third stages in the case of tuffeau. Therefore, it is recommended that the salt crystallization test extend for more courses to gather the whole information about the role of salt weathering in the deterioration of these stones. On the other hand, the experimental measurements revealed that tuffeau stone samples did not reach the full dry weight during the dry stage, i.e. the only 20 h is insufficient for full drying. This means the remained salt solution within the tuffeau stone samples will transfer to the adjacent Richemont stone samples during the drying stage. The process of salt solution transformation is well accepted by taking into account the fact the tuffeau has the higher water transfer properties (see Tab.1). This illustration gives another explanation why the linked tuffeau stone samples behave more positively against the salt weathering than the isolated stone samples.

4. CONCLUSIONS

This study focused in finding a suitable method to simulate the stones situation in the field when exposed to salt weathering. The experimental work includes preforming the salt crystallization tests on the porous stone by following two different test conditions based on the situation of the stone during the test: the individual test method, and the linked test method. The aged stone samples were passed through different tests both in macro- and in micro-scale. The results were well discussed to find the compatibility between the stones. In the following the conclusions are summarized:
1. Applying two different methods in the salt crystallization tests leads to obtain different results with regard to the behavior of stone samples against salt weathering. Consequently, these proposed test methods enable for assessing the compatibility between the stones.

2. The fine analysis of the results obtained both from the macro-scale tests (weight measurements and visual appearance) and from the micro-scale tests (XRD, IC and MIP) refer to the presence of the compatibility, partially, between the examined stones.

3. The crystallization pressure (Ps) and the salt susceptibility index (SSI) are the two parameters predicted from the pore size distribution, they could be employed to assess the compatibility between the stones.

As the salt crystallization tests has lasted only 20 wetting-drying cycles, unfortunately, it was insufficient to observe the three stages already described by (Angeli et al., 2007). Therefore, the main conclusion can be drawn: the compatibility between tuffeau and Richemont stone cannot assert. It is strongly recommended to carry out other weathering tests in wide range of cycles including other salt solutions of Na\textsubscript{2}SO\textsubscript{4} CaSO\textsubscript{4} in addition to NaCl.

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| Table 1. The characterization of the stones, n: total porosity (%); ρs & ρa: skeletal & apparent density (g/cm\textsuperscript{3}); Ca : Capillary absorption coefficient, (g/m\textsuperscript{2}/min\textsuperscript{1/2}); σt : indirect tensile strength, (MPa). |
|---|---|---|---|---|---|---|---|
| Stone type | Mineral /% | Porosity & densities |
| | | n | ρs | ρa | Ca | σt |
| Tuff. | Calcite / 50 | 45.0 | 2.4 | 1. | 0.3 | 6 | 11.67 |
| | Opal / 30 | | | | | | |
| | Quartz / 10 | 3 | 7 | 35 | | |
| | Clay / 10 | | | | | | |

| Table 2. Results of the MIP test |
|---|---|---|---|
| Stone type | Pore size distribution, (μm) | |
| | Small pores | Medium pores | Large pores |
| Tuff. fresh | < 0.01 | 0.01-0.1 | 0.1-1.0 | 1.0-10 | >10 |
| Tuff.-isol | 6.22 | 12.55 | 20.29 | 52.02 | 8.92 |
| Tuff.-linked | 9.99 | 23.86 | 23.87 | 34.21 | 8.07 |
| Rich. fresh | 6.54 | 22.76 | 25.35 | 35.04 | 10.31 |
| Rich.-isol | 0 | 2.96 | 42.17 | 51.58 | 3.29 |
| Rich.-linked | 0 | 2.37 | 40.87 | 54.08 | 2.68 |

| Table 3. Comparison of the sustainability degree for the aged stone samples. Dark gray indicates the least sustainable samples, light gray intermediate, white the highest. |
|---|---|---|---|---|---|---|---|
| Stone type | Weight loss & appearance | Salt content | Salt susceptibility index, (SSI) | Crystallization pressure, (P) |
| Tuff. | isolated | isolated | isolated | isolated |
| | linked | linked | linked | linked |
| Rich. | isolated | isolated | isolated | isolated |
| | linked | linked | linked | linked |
Figure 1. Schematic of crystallization cycles

Figure 2. Normalized weights of the aged stone samples

Figure 3. Visual Appearance of the aged stone samples

Figure 4. Pore size distribution of the aged stone samples

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