SWBSS2014
3^{rd} International Conference on Salt Weathering of Buildings and Stone Sculptures
14-16 October 2014

Monitoring of the salt mist aging test with DRMS

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Abstract

Salt mist test can be used to evaluate the durability of porous materials. Besides mass loss, the paper discusses the use of DRMS (drilling resistance measurement system) to monitor changes in strength due to salt crystallisation. Drilling resistance was used to characterize changes promoted in very different lithotypes, namely carbonate rocks and one variety of sandstone. The results show that this technique is able to identify fingerprints of the presence of salts on the profiles seen as increments in resistance. Damage zones can also be detected on the profiles of the aged samples, after desalination. The results clearly indicate the influence of moisture content on the drilling data. Although considered as a minimally invasive action, the method produces a small hole that has some influence on the ongoing decay process. In this paper illustrative examples are presented, some advantages and drawbacks are indicated in order to correctly evaluate the results when it is possible to use it to evaluate the action of salts.

Keywords: salt mist test, drilling resistance, natural stone, drill bit wear effect.

1 Introduction

Salts are considered as the most effective factor of decay, operating in very different climatic conditions, and are particularly relevant when repeated moisture and temperature cycles enhance the dynamism of their action. The evaluation of the durability of stone materials in face of this mechanism is a relevant research topic with implications in several domains, including stone conservation.

In laboratory, the behaviour of porous materials in salt crystallization tests has been extensively studied and results obtained in different stone types from all over the world are available in scientific journals and conference proceedings. *Salt crystallization test* has a long tradition of use to access the stone durability to salts. The original version was introduced by Brard in 1828 [1] to evaluate the susceptibility of stone to frost damage. According to it, stone samples are subjected to cycles of immersion in a salt solution followed by drying in an oven, using a concentrated solution of sodium sulphate. Now as then, the protocol of testing prepared by CEN uses a similar procedure [2] to determine the relative resistance to salt crystallisation of natural stones. The test results are expressed as “mass loss”. This test method is considered suitable for the evaluation of very porous stones (higher than 5% porosity) but it is also commonly used for low porosity materials evaluation. It is a very aggressive test, able to produce damage on porous materials very rapidly. To get information in a short period of time justifies the extreme conditions very often used in aging tests. However, this test is criticized due to the fact that the preconized conditions are extreme and far from reality.

Moreover, in particular when exposed to marine conditions, the materials are submitted to very different conditions from those considered above. Natural weathering due to salt crystallisation is quite often related with marine spray action. In the lab, the simulation of this action can be achieved by using a climatic chamber where the marine spray is simulated by the production of spray with the testing solution. As in the previous protocol, wet and dry cycles promote damage but softer action is expected for the salt spray. In this protocol, sodium chloride solutions are often used instead of sodium sulphate.

The equipment is able to create a salted and humid environment during the wet phase, followed by a period of drying created through the increase of temperature in the cabinet. The duration of each phase can be programmed and changed according to the objectives and always taking into account the characteristics of the materials to be tested. Besides a better similitude to reality, this test promotes smaller increments in decay between cycles and therefore it allows following the decay process in a better controlled way.

In both test procedures, the absorption of salt solutions into the void system of the materials is a determinant factor of decay. Successive cycles of wet and dry phases are responsible for decay, which is mainly attributed to the processes of physical disintegration of the materials as the result of salt crystallization pressure created inside the material.

2 Objectives

Both types of tests are currently considered in the European normative of stone testing, as previously mentioned. The two procedures use the quantification of damage expressed as a "mass loss" value determined after a certain number of cycles. Values are usually expressed as a single number per test.

In research studies, the results are usually expressed in a different way; the "mass loss value" is determined in each step to allow defining a trend line representing the overall behaviour of the stone during the test.

Besides "mass loss", which is considered a very relevant parameter, other physical properties can also be used to quantify physical changes. Porosity, water absorption and ultrasound velocity are examples of properties that can be successfully used to evaluate global mass modifications on stone materials, in particular when used in a comparative basis, before and after salt aging test.

Salt crystallization changes the original porous space configuration and new fissures, cracks or voids can be created whose presence is reflected in a lower mechanical strength. Visual damage can be identified as flaking, disaggregation, disintegration and rupture of the stone specimens. Quite surprisingly, the effect of the presence of salts inside the materials is not usually considered when analysing the test results. Though in some cases desalination is recommended, the operating conditions defined to achieve it hardly correspond to an effective desalination process. The presence of salts may induce damage but, depending on the materials, they can also artificially increase mechanical strength, or modify the ultrasound velocity usually used to assess degradation in a non-destructive way. Salt accumulation during aging test is already known in other stone types, namely on granites, where a very small amount of salts was detected using ultrasonic pulse velocity [3].

Natural weathering profiles can also be developed due to salt damage. The methods mentioned can hardly detect or identify the characteristics of the material in depth, although the final values can be skewed by their presence. Taking all these facts into consideration the *drilling resistance method* showed up as potentially interesting to investigate physical changes due to the presence of salts, especially when small changes of the resistance in depth are expected to occur.

This paper aims to support the use of drilling resistance measurements to control the changes induced during salt crystallisation tests, by presenting data taken from a salt mist test carried out in very different stone materials commonly used in Europe.

3 Materials and test conditions

Five lithotypes are considered in this work, including carbonate stones and sandstones. One marble (with heteroblastic fabric), three limestones (fine and coarse grained, more or less homogeneous) and one variety of sandstone with transverse anisotropy, tested in specimens (cut parallel to the bedding plane).

The marble samples (MC) are from Camaiore, in Italy. The fine grained limestone comes from Portland, UK (PW), and the coarse grained limestone from Vicenza (Va), Italy. Sander is an anisotropic sandstone, from Hassberge in Germany, (S). Some tests were also performed on a Portuguese limestone from Coimbra (Ti), a very soft and homogeneous stone, taken as a reference in the interpretation of drilling test data. Besides the wide range of chemical and mineral composition, these materials also present very different physical properties, including very low porosity materials, like marbles, as well as very high porosity ones, such as Vicenza (Va) and Portland stone (PW).

Porosity, water absorption and mechanical resistance properties of raw material used are presented in Table 1 for comparison.

Table 1: Physical properties of the raw materials

Lithotypes	Porosity accessible to water (%)	Maximum water content (%)	Water absorption by capillarity [Kg/(m^2*h $^{0.5}$)]	Compressive strength (MPa)	Drilling resistance force (N)
MC	0.4	0.1	3.9 - 4.2 x 10^{-2}	131.7	66 - 61
PW	21.1	9.9	8.5 - 9.4	53.2	18 - 25
Va	28.5	14.8	8.0 - 8.3	20.8	6 - 11
PW	21.1	9.9	8.5 - 9.4	53.2	18 - 25
S	17.8	8.2	1.5 - 1.9	51.8	11 - 14
Ti	19.0	8.7	nd	nd	6-8

Note: MC - marble; PW - limestone; Va - limestone; S - sandstone; Ti- limestone. For complementary information see text above

3.1 Salt mist test

The stone specimens were submitted to cycles of alternate conditions of salt mist and drying, in a climatic chamber capable of maintaining the temperature from lab conditions to about 40ºC. The salt mist consists of a solution of sodium chloride in deionized water at 1:20 in weight. Ten hours in a salt mist atmosphere (flowing rate of 2.5ml/h) under ambient temperature were followed by 38 hours of drying at 35ºC.

Prismatic specimens were used in this study. Lateral faces were coated with an epoxy resin in order to leave exposed only the two faces orientated in one direction. During the test the debris detached from the surface were carefully collected to help control the induced damage.

The samples were characterized to determine the original characteristics (T0) and photographed for visual inspection.

At the end of the test (or in specified intermediate control steps), the specimens were immersed in de-ionised water to remove salts before proceeding to characterisation. This is a very slow process and the water had to be changed periodically until total desalination. Then, specimens were dried, weighed and visually inspected. Mechanical characterisation was performed using non-destructive techniques selected from those currently in use in laboratories*. Drilling Resistance Measurement System, from SINT (Italy)* was used to evaluate resistance in depth.

In a preliminary test aiming at defining the protocol adequate to test the selected rock categories, two specimens of each rock type were used. The duration of drying and wetting phases, the possibility to test sequentially the same specimen in each step of the test are examples of questions then considered as relevant. The purpose of the present paper is to use the information obtained in that study to discuss the use of drilling data to monitor changes due to salt mist action. The wide range of materials object of analysis will also help to frame the range of validity of the suggested procedures.

The ultrasound methods allow a non-destructive monitoring of each individual sample throughout the test, consequently reducing the number of specimens required to test and avoiding the uncertainty induced by the comparison of results taken from different specimens at each step of the test. The use of drilling resistance to complement “ultrasound pulse velocity” data was also under analysis.

A total number of 50 cycles were performed; one phase of control was done between the 23 and the 25 cycles, in dry and wet phase. At the 25th cycle, in the begging of the drying phase, the specimens were cleaned for removal of detached particles and dried in a ventilated oven at 100ºC, for about 72hours. After 50 cycles the specimens were tested in wet and dry phases, cleaned and desalinated for 30 days to remove the salt from the interior. Dry mass loss was measured and final characterization of the material after 50 cycles was performed.

Considering the protocol used and summarized above, it is worth mention that EN 14147 protocol [4] follows a similar general philosophy. However, the concentration of the sodium chloride solution and the cycle conditions are different from those used in this test. A longer cycle and a more concentrated solution were considered to be more effective to test simultaneously so different stone materials.

3.2 Drilling resistance measurements

The drilling resistance measuring system (DRMS) is a power drill with constant feed and a force transducer that measures the thrust as a function of the drilling depth. It is considered a quasi-non-destructive technique due to the fact that a hole is produced during the test, typically a 5mm diameter hole. The equipment used was a prototype originally developed through the European HARDROCK project [5].

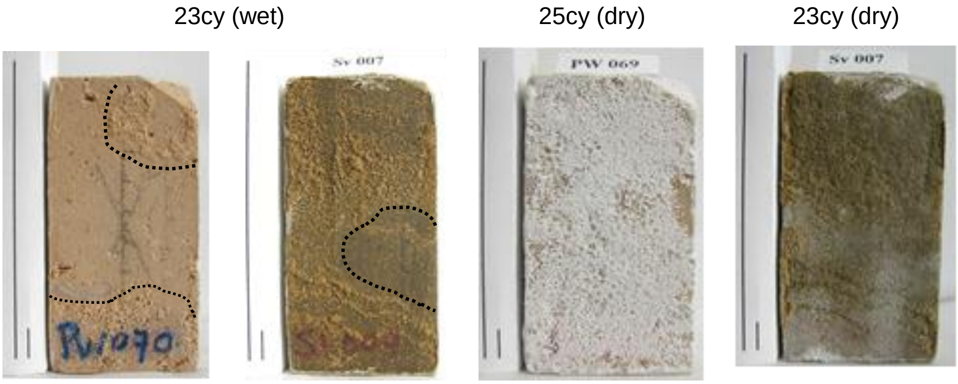
The drilling test has been used to characterize stone resistance in depth. To interpret the results several variables must be taken into consideration, namely grain size and homogeneity of the material or the water content, a variable that has not been very studied up to now. The presence of quartz grains is a serious drawback, in particular in very compact mineral textures, since it is responsible for the drill bit wear effect and a strong base noise in the drilling graphs. When drill bit wear exists, the correction of the original test data is absolutely mandatory [6].

4 Results and discussion

4.1 Salt damage patterns and their quantification with “mass loss“

Macroscopic observation can spot slight changes during the test, when they modify the original roughness of stone surfaces. Damage is more evident when the material is wet due to the fact that drying allows a formation of efflorescences, which are particularly evident in very porous materials able to absorb higher amounts of solution. Detachment of single grains or aggregates of grains (*disintegration*, *powdering*) or as thin scales (*flaking*) on Sander specimens (S) were evident deterioration signs.

Due to evaporation, salts formed a thin but hard crust on the surface of Vicenza stone specimens and fluffy efflorescences “randomly” dispersed on Sander specimens. Very often, deterioration patterns were not uniformly distributed. Original heterogeneities of the materials can justify differences on the level of damage observed in adjacent areas. Some examples are indicated in Figure 1.



23cy (wet) 25cy (dry) 23cy (dry)

PW - 6%* S - 5%* PW - 1%* S - 3%*

Figure 1: Heterogeneous distribution of damage during salt mist test. Macroscopic aspect of the surfaces during wet and dry phase at 23th cycle*; * the numbers (in %) represent the residual "salt solution content" in each phase, before drilling test*

Table 2 presents mass loss values after 50 cycles of salt mist and the quantity of solution absorbed in the wet phase. Figure 2 allows the comparison of different types of stone using *mass loss* values.

Table 2: Salt solution content estimation on wet phase and mass loss after 50 cycles

Stone types	MC	PW	Va	S	Ti
Mass loss (%)	0.07 - 0.11	0.75 - 0.97	0.37 - 0.82	1.69 - 2.22	0.33 - 0.46
Salt solution content (wet phase)	0.12 - 0.13	5.7 - 6.7	11.3 - 11.5	4.6 - 6.1	6.6 - 6.8

Note: MC - marble; PW - limestone; Va - limestone; S - sandstone; Ti- limestone.

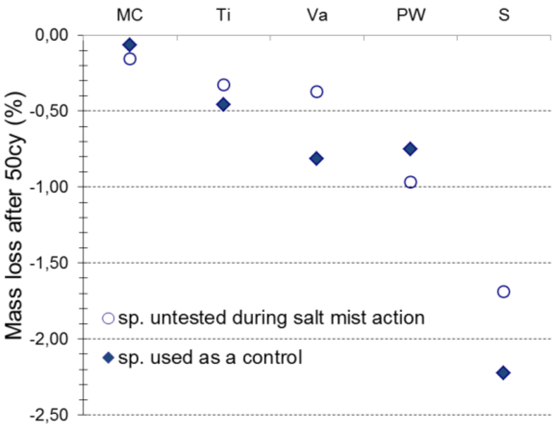


Figure 2: Mass loss after 50 cycles of salt mist test

Marble (MC) is the stone variety that was less affected by salt mist action. This is justified by its very low porosity and consequently by the reduced absorption of the salt solution.

Other lithotypes show different behaviours, which remain unexplained through these properties. Vicenza stone (Va), in spite of its high porosity, revealed a modest value of mass loss during this test. On the contrary, similar salt solution absorptions allowed different levels of damage in PW, S and Ti, as their mass loss values indicate, claiming for other suitable explanations.

4.2 Using drilling resistance to identify the damage due to salt mist action

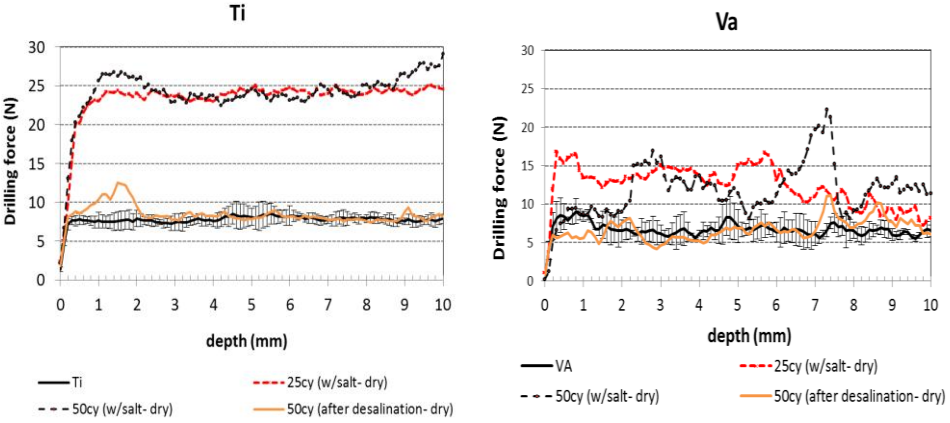
Drilling resistance test is a very sensitive test to evaluate resistance in depth. Concerning this application, the main raised question is related with the possible interference of the presence of salts on the results. The presence of water (as salt solution) inside the materials also influences the strength values, a fact that has to be taken into account, in particular if a well-defined reference for moisture condition cannot be guaranteed. Besides these specific questions, others aspects must be taken into account for a correct use of this technique, namely the influence of grain size and heterogeneity of the material, as well as the drill bit wear due to the presence of abrasive quartz grains, in particular in compact materials.

In the following graphs some examples of drilling test profiles are presented in order to illustrate what kind of information can be expected. For comparative purposes, in all the graphs T0 values are projected as the average values of at least five drillings (and their respective standard deviation), in black. All tests were performed using 600 rpm of rotation speed and the penetration of 10mm/min.

4.2.1 Soft and non-abrasive stones

Good results are usually obtained on very homogeneous materials, in particular if they are non-abrasive like the Portuguese limestone (Ti). Concerning salt mist test, the presence of the salt increases the resistance of the material, as shown in Figure 3a, graphs “25cy-dry” and “50cy-dry”. In this case, the tested length is fully “impregnated” with salt and the force resistance more than doubles the original value. Desalination removes the salt and consequently lowers the drilling resistance. However, desalination was not complete since a peak is still detected near the surface (1-2mm) thus illustrating how sensitive the method is to minimum amounts of salt. Damage was limited to scarce powder detachments and has no fingerprints in the drilling profile.

In this type of stone it is very common to have drilling graphs showing some artificial increase of force in depth due to the accumulation of fine dust during the drilling process, as seen in Ti specimen at the 50th cycle with salt (Figure 3a). To limit the occurrence of this packing effect, the hole-in-hole procedure can be adopted when small size specimens (till 4cm thickness) are used [7].



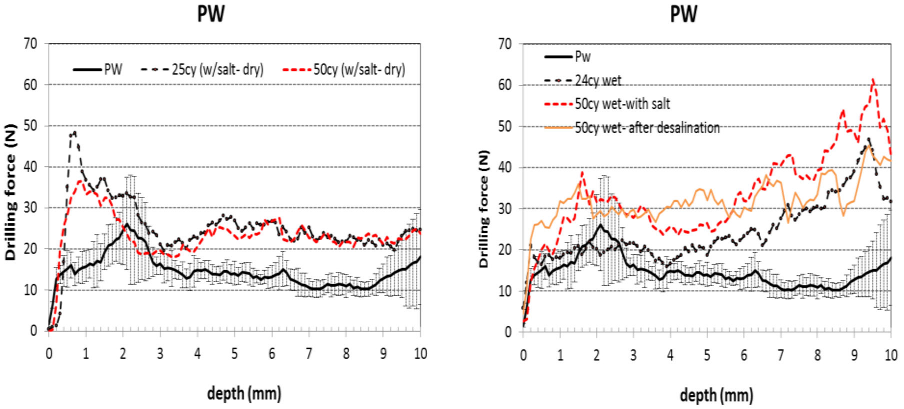
a) Portuguese homogeneous limestone (Ti) b) Vicenza heterogeneous limestone (Va)

Figure 3: Drilling tests on limestones.

A very different situation is obtained on Vicenza stone (Figure 3b) due to its marked heterogeneity. Graphs are much rougher, but the presence of salt is still detected by the increase of force values in the salinated condition.

Portland stone (PW) is a harder and very heterogeneous material (Figure 4). In spite of this fact, the hardness profiles clearly demonstrate the presence of salt, more pronounced on the two first millimetres while less in depth, from 4-9 mm (Figure 4a).

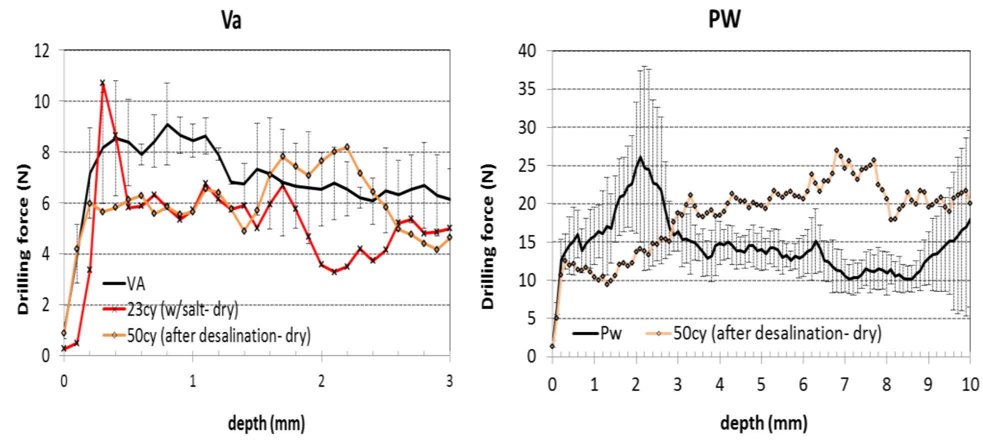
The effect of moisture to promote packing in depth is also clear in this stone when it is wet (Figure 4b). Packing, produced by dust accumulation and favoured by the presence of moisture, can be overcome by using the pilot-hole technique, as indicated before.



a) drying phases b) wetting phases

Figure 4: Drilling tests on heterogeneous Portland limestone (PW); dry and wet phases

Vicenza stone (Va) shows a surface damage in the first 2mm, where previously a peak of salt accumulation was detected (Figure 5a).



a) Vicenza limestone (Va)

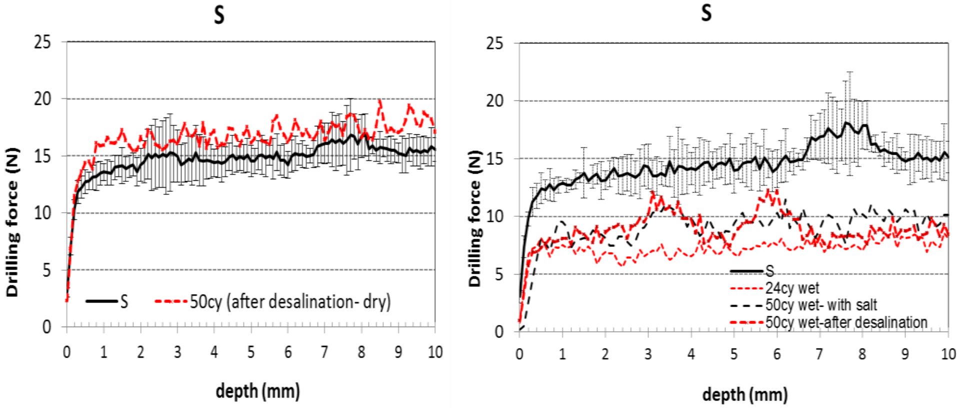
b) Portland limestone (PW)

Figure 5: Drilling tests on heterogeneous limestones varieties after salt mist, in desalinated specimens

In other cases, some salt can still remain in depth (Figure 5b), although in the first three millimetres near the surface a decrease of resistance is registered and considered to be the result of salt damage. In fact, on Portland limestone, the desalination was not fully achieved when it was tested but the profile shows some surface damage in the first millimetres.

4.2.2 Hard and abrasive materials

When the stone is abrasive, like Sander sandstone (S), drilling tests must be carried out using a specific protocol to determine the abrasive law and to ultimately correct the drilling resistance data against the drill bit wear [6]. In this study, drilling data were obtained before the correction methodology was entirely developed and therefore corrections had to be made in a slightly different way, with the corrections being supported in drilling tests carried out in the To reference samples above defined.



a) after desalination

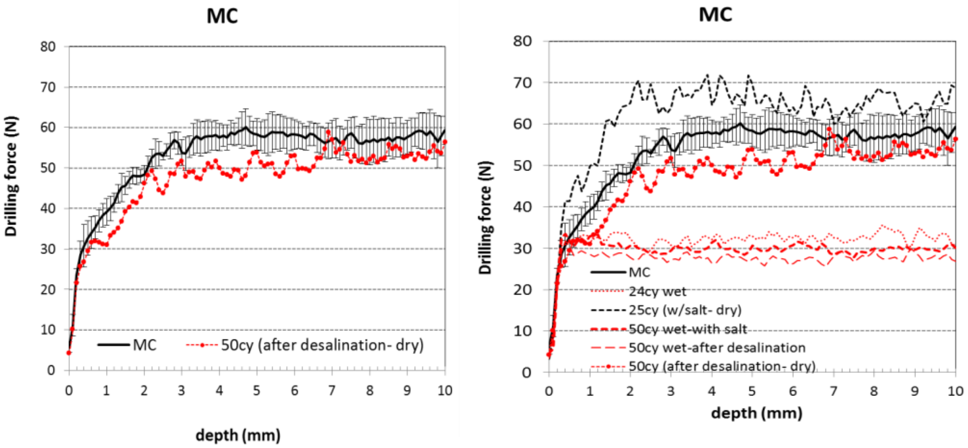
b) wetting phase
Note: “water” content estimation on 24cy-5% and 50cy- 4-6%

Figure 6: Drilling tests on abrasive Sander sandstone (S)

In this type of stone the presence of moisture is particularly relevant and it is responsible for a decrease of the resistance values (Figure 6). After salt mist action, no evident signs of damage or accumulation of salt were detected. The slight increase of force compared to non-decayed values (represented by “S” in Figure 6b) must be considered in the range of error introduced by the correction process in this particular case.

Marbles can be also slightly abrasive. However, in the present case, the values of forces were corrected due to the fact that only one drill bit was used to test all the materials. The wear effect promoted by Sander sandstone was responsible for the artificial increase of the values obtained on marble and justifies the need for the correction.

The drilling tests on marbles did not detect significant changes after salt mist action (Figure 7a) but the influence of moisture must also be considered in this stone variety. Even in small amounts, the force values decreased dramatically, as it is evident in the holes drilled during wetting and drying phases of the cycle (Figure 7b).



a) after desalination

b) wetting and drying phases

Figure 7: Drilling tests performed on marble (MC)

5 Conclusions

The use of drilling resistance on salt mist samples allow to confirm the sensitivity of the method for stone rock profiles characterization.

In fact, salt mist action promotes slight changes, hardly detectable after test. Great part of the damage is removed due to the fact that sand disintegration or small flakes produced are removed in each cycle.

However, the experience allows considering that it can be successfully used to detect the presence of salt accumulations or the damage promoted by salt damage. The slight decrease of mechanical resistance near the evaporitic zone on the profile is, in this case, considered the result of the action of salts.

From the results obtained an influence of the moisture on the drilling profiles is noticed. In most of the cases it reduces the force, and this means that stone hardness decreases due to this fact. Actually, this finding confirms the influence of the presence of water in the lowering of mechanical resistance of materials.

Acknowledgement

This work was carried out in the framework of the project “Effects of the weathering on stone materials: assessment of their mechanical durability” (McDUR), contract G6RD-2000-00266.

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