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Evaluation and monitoring of water diffusion into stone porous materials by means of innovative IR thermography techniques

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Abstract

This paper shows the application of two innovative infrared thermography (IRT) methods for the evaluation of water transport phenomena through the outer layers of porous stone materials. An infrared camera monitored: a) the moisture stain due to the water absorption after having spilled a drop on the stone surface and b) the "Moisture ring" around the damp surface after the contact between a soaked round sponge and the stone surface. The time of spreading and the geometric shape of the damped area depend on the porosity of the stone material and are useful to characterize the physical stone features. Monitoring the evolution of the moisture ring by IRT allows to implement and optimize the data coming from the water repellent products protocols with data coming from the "contact sponge measures". Moreover, moisture ring test links IRT and laboratory tests characterizing the diffusion phenomena of liquid and water vapour in porous building materials. 30 specimens of marble, calcarenite and sandstone were tested with steady methods (dry index and water absorption by capillarity), that validated the data provided by the two innovative IRT techniques.

I. Introduction

The cycle of water-vapour exchange between air and masonry materials is mostly influenced by their chemical and mineralogical composition, hygroscopicity of mineral components, pores amount, their size distribution and shape and by the surface hydrophilicity, as well known by the scientific literature [1, 2, 3, 4]. Water enters into a porous material either as vapour and liquid; as vapour is retained as a function of hygroscopicity, and it could cause the local phenomena of condensation; as a liquid it could be transported through capillary network. In its path, liquid water dissolves soluble salts which precipitates on the outer surfaces or in the layer underneath in functions of their solubility, giving raise to the efflorescence or sub-efflorescence phenomena respectively. These latter are considered among the most damaging effects generally present in building porous materials.

The presence of liquid water in the stone bulk is typically due to water absorption by capillarity; on the contrary, surface condensation involves the stone surface and the outer layers.

As well known, mechanisms and kinetics of decay are strictly depending on the stone characteristics, especially the surface and the outer layers, until a few centimetres of depth. In scientific literature the deterioration of porous materials is extensively discussed [5, 6].

The study of the exterior layers of the material is crucial in evaluating the decay progress due to the water exchange and the changes in the surface hydrophilicity due to the application of consolidants and water-repellents [6]. The most updated literature [7, 8, 9] indicate that a complete material characterization requires the measurements of the porosity, the absorption by capillarity (CA) and the Drying Index. The results of standard tests serve as assessment and comparison to the results of the innovative tests that the authors propose in this paper, to evaluate their reliability and effectiveness. In previous papers the authors analysed the differences of hygroscopic performances of plaster surfaces due to the roughness and compactness of mortar finishes [11]. In this paper the research focus on water exchange properties of three different stones without any sampling them, with the aim to integrate traditional standardized techniques with not destructive IRT.

II. Materials and methods.

2.1 Stones characteristics

The tests went on three different kinds of natural stones which have been chosen with different physical features in order to asses a variety of behaviour regarding water absorption. Specimens sized 50x50x20 mm have been used as indicated by the Italian standard UNI 10859 [12].

Noto Calcarenite is a porous yellowish fossil-bearing Miocene calcarenite quarried in Sicily. Its micro-structure is well known and described in literature [13]. Dorata Sandstone (Arenaria di Manciano) is a medium porosity Miocene sandstone quarried in South-Tuscany with sparry calcitic cement and feldspar-quartz clasts. Carbonate content is quite high, reaching 67% at the maximum.

A dolomitic fine-grained marble has been used, extracted from the quarries of Mermeren Kombinat, near the town of Prilep in Macedonia; it bears a dolomite content of over 99% with a saccharoid structure [14].

2.2. Thermographic test

The Spilling Drops test (SD) test permits to measure the surface characteristic of absorption and diffusion of liquid water by the thermographic visualization of the dumped areas. During 10 minutes of shooting, one drop of distilled water (0.03 ml) is spilled on the specimen. Thermal images show the drop spreading on the surface according to the physical features of the material. In this case the area of the damped surface cooled by the evaporation of water is the parameter leading to a possible evaluation of the porosity.

The Moisture Ring (MR) test records the water spreading and its evaporation during 10 minutes after the application of the sponge with a thermal camera. It consists in the implementation of the steady test of the contact sponge [15-16- 17]. Wet surface appears as circular area because of the sponge circle shape. Both tests use cooling caused by the evaporation of water and detect the thermal contrast between the wet and dry areas. Shooting thermal images for a time lapse of 10 minutes allowed to complete the tests with a temporal parameter. The two procedures permit to observe the differences of the water spreading on different stone surfaces.

III. Results

3.1. Open and total porosity

Measurements of open and total porosity were carried out by mercury intrusion porosimetry (Thermo Scientific low pressure Pascal 140 – high pressure Pascal 240); data expressed in volume (V%) are in table 1 [18].

	Noto calcarenite	Dorata sandstone	Prilep Marble
Megapores %	12.20	25.88	24.42
Macropores %	86.80	66.99	72.96
Mesopores %	1	7.122	2.63

Table 1 – distribution of porosity percentage in the specimens under tests

3.2 Contact sponge test

This test was recently suggested as a rapid tool to highlight water absorption capacities on building surfaces [17-18]. As a consequence, it is considered an useful tool for an empiric in-situ evaluation of stone degradation without sampling. The repetition of the quantitative measurements of the absorbed water on 10 specimens, showed that the test is precise and accurate: water absorption ranged from 1.21 g (Noto), to 0.078 g (Dorata) and 0.017 g (marble) with standard deviations expressed in percentage respectively of 18.8%, 17.7% and 17.6% (table 2).

3.3 Water Asorption by capillarity.

The test has been carried out following UNI 10859. Q_f is the water amount absorbed after 72h per surface unit (Table 1); in the linear part of Fig. 1, capillary absorption coefficient defined as

$$CA = Q_{30}/t^{30} \quad (1)$$

allowed to calculate the slope of the curve, where Q_{30} is the water absorbed by the specimen after 30 minutes per unit surface [19]. Noto calcarenite' curve reached an asymptotic value quite immediately after only 30 minutes, while Dorata sandstone' curve took more or less 8 hours to reach its asymptote.

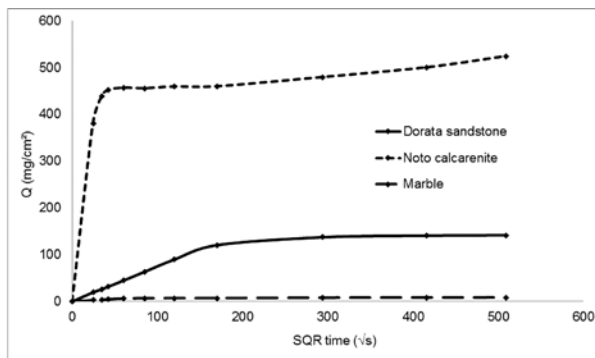


Fig. 1 Water absorption by capillarity (Q) of Dorata sandstone, Noto calcarenite, Prilep marble. Data are averages of 5 measurements on 5 specimens

3.4 Evaporation flux measurement

In Fig. 2, the graphic of the evaporation flux on Noto calcarenite displayed values of a higher evaporation flux than marble and Dorata sandstone, and the values are constant with a water content up to 9-10% of WC absolute

value (corresponding in the graph to 50% or relative WC). The plots of Dorata sandstone and marble are similar: the flux has high value only for relative WC near to 100%, then suddenly decreases.

Stone	Open porosity	WA (g/cm ² *min)	WA St. Dev.	Q_f (mg/cm ²)	CA (mg/cm ² s ^{-1/2})	Drying Index (DI)
Prilep Marble	0,57	0,00056	9,7 10 ⁻⁵	8,513 ± 3.34	0,115±	0,328
Dorata Sandstone	10,70	0,00257	4,55 10 ⁻⁴	141,769±11 . 62	0,752±	0,504
Noto Calcarenite	36,20	0,03935	7,373 10 ⁻³	524,161±15 . 50	10,654±	0,442

Table 2 – Physical characteristics of the three stones (average on 10 specimens). WA = water absorption, CA= capillary absorption coefficient, Q_f = water absorbed in 72h

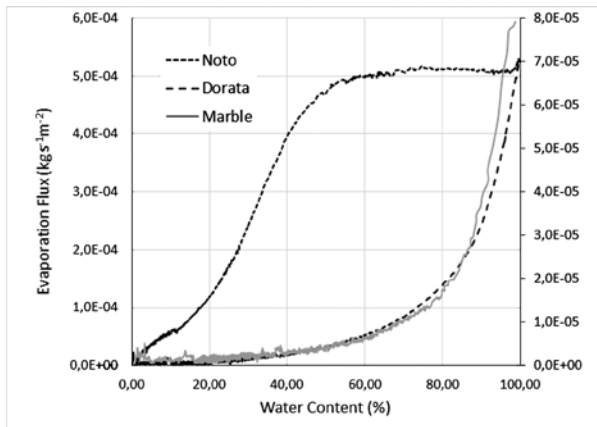


Fig. 2 Evaporation fluxes of three stones under controlled environmental conditions ($T=25^{\circ}\text{C}$; $\text{RH}=50\%$) versus relative water content. The graphic has different scales of Noto stone (on the left) and of Dorata and marble (right)

All the tests permitted to distinguish the water exchange properties of the different stone surfaces. In addition, they allowed to verify that the exterior layer of the materials surface really affects water absorption, spreading and evaporation mechanisms between ambient and stone. Table 2 presents the data coming from measurements of total

open porosity, water absorption (WA) from contact sponge method, Qf and CA from capillarity test and DI.

3.5 Thermographic tests

MR and SD allow to observe the water spreading on the surface and then its evaporation. The results of laboratory tests show a clear correlation among porosity, water absorption by capillarity, water spreading inside the microstructure of the external layers of the material and evaporation flux (Drying Index). The cooling down of the damp areas on the specimen's surface is the effect of the water (both in liquid and vapour phase) transport phenomena. Thermal images drop test provide the measurement of the change of temperature due to these effects (Figure 3). On the Noto calcarenite's surface the wet area is wider than the one on the marble's surface specimen, and it has a regular circular shape too; this shape is due to the liquid which regularly fills up all the open pores at disposal. The widest area is due to the rapid absorption of the drop by the surface, that is rich in macropores, where water capillary spreading is faster. On the Dorata sandstone the wet area has undergone the largest increase during the observation time, nevertheless the shape becomes irregular and the contour line is so unreadable and vague that it is difficult to measure. This behaviour can be attributed to the megapore prevalence with respect to the Noto stone. Regarding the Moisture ring test the authors stressed that after having dampened the stone surfaces by contact sponges, the dynamic measurement of temperature permitted to evaluate the extension of the water

diffusion on the surface. The extension and shape of the wet moisture ring changed for any tested materials, depending on their surface characteristics (Table 3). The authors selected the thermograms of the widest area and the longest perimeter to evaluate their differences in the three stones (Fig. 4).

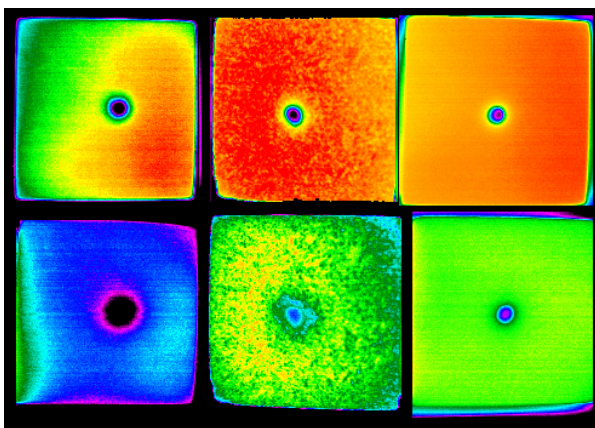


Fig. 3 Thermal images shot at the initial moment of spilling the drop on the stone surface (top line) and 10 minutes after or when the drop geometry became stable (bottom line). Range of temperature $25.0-32.0^{\circ}\text{C}$; emissivity ranging between 0.89 (Dorata and Noto) and 0.95 (Prilep Marble).

Stone	Average area of the drop, starting point (px)	Average area of the drop, maximum extension (px)	Percentage of the increase (drop areas)	Moisture ring area (px)	Moisture ring perimeter (px)
Prilep Marble	14,98	17,41	16%	1083	118
Dorata Sandstone	16,04	37,99	137%	991	113
Noto Calcarenite	47,17	60,57	28%	1274	127

Table 3 – Measurements of the spilling drop and moisture ring: areas and perimeter in pixels; average on ten specimens.

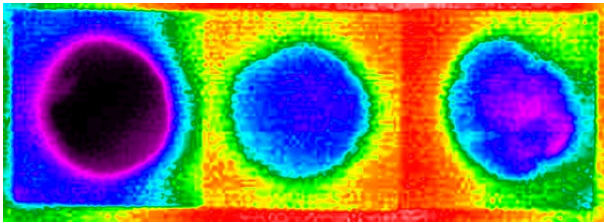


Fig. 4 Moisture ring after the contact with the soaked sponge, from left to right Noto calcarenite, Dorata sandstone, and Prilep Marble, Temperature ranging between 26 and 29°C

IV. Discussion

The tests of MR and SD allowed to distinguish the different kinds of porosity: in fact, Dorata sandstone has a higher variety of pore sizes than marble and Noto calcarenite, as fig. 2 shows. The distributions of pores in Marble and Noto Calcarenite are similar, without mesopores (diameter between 2 and 50 nm) whilst the pores distribution in Dorata Stone shows a plateau in the central values (diameter in the range 0,1-10 μm). MR results of Dorata Stone (tab. 3) are not linear with the total open porosity as observed in the other stones: this suggests an influence of the presence of mesopores (the least considered) [16, 17] that contributes to water spreading into the bulk but not to the surface evaporation. The present evaluation and discussion of results takes into account also the size of the area where evaporation occurs, comparing the extension (see table 3). Nevertheless, at the present step of the research some factors affect the measurement of the area of interest and its delimitation, the spatial resolution of thermograms, the background temperature. WA has also a direct correlation with the coefficient of water absorption by capillarity (CA) ($R^2 > 0.999$).

V. Conclusions

The presented techniques allowed to measure different characteristics of the exterior layer of stone building materials. They derive from three different fields of investigation, and the results congruence shows their integrability. Laboratory weighing tests validated the thermography results, and the latter resulted suitable and effective tests to quantitatively evaluate the absorption capability and the evaporation of liquid water. In this way IRT by passive approach showed good results to indicate the variation of surface microstructure by measuring the changes of the surface temperatures due to absorption, diffusion and evaporation of water. Particularly, the integration of IRT and the test of water absorption by contact sponge allowed to ascertain the origin of the factors which could have a major role to identify the risk conditions, like the pores distribution and size in the very exterior

layer of the materials, where the water exchanges occur much more frequently than a few cm inside. Moreover, these results could provide fruitful considerations in foreseeing the soluble salts movements when the formation of efflorescences are concerned. The authors qualitatively showed the sensitivity of the presented methods in distinguishing the different porosity of materials. A further step of the research will be the application of the water drop and moisture ring methods to stones before and after the application of water repellent restoration products, to evaluate the changes of liquid water absorption.

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