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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 measured: 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. In addition, monitoring the evolution of the moisture ring by IRT allows implementing and optimize the data coming from protocols of water repellent products with data coming from the "contact sponge measures". Moreover, moisture ring test links IRT and laboratory tests characterizing the transport phenomena of liquid and water vapour in porous building materials. Thirty 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.

**Keywords:** evaporation, porosity, IR thermography, contact sponge, moisture ring, spilling drop

## **1. 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 as either vapour or 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 transport phenomena in between stone microstructure and environment; moreover, stone surface hydrophilicity could be dramatically changed after the application of consolidants and water-repellents [6]. Current standard and 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 [10]. Capillary absorption test is an effective method to measure the penetration of water into the bulk of stone materials. The used Standard [11] determines by gravimetry the water absorption of a specimen sized  $50 \times 50 \times 20$  mm, kept in contact with water through a multiple layer of filter papers; the measurements have been carried out at regular time steps intervals; the absorption referred to surface unit ( $\text{mg}/\text{cm}^2$ ) is normally reported in a graph vs  $t$  ( $\text{s}^{1/2}$ ).

It is possible to measure the Drying Index after having carried out the Total Immersion test [12]. The drying index is defined as the ratio of the integral of the drying curve and the initial water content [13], which have been measured gravimetrically, at the end of Total Immersion Test. 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 and finishes [14]. In this paper, a strong innovative approach has been pursued focusing on water exchange properties of three different stones without any sampling, with the aim to integrate traditional standardized techniques with not destructive IRT (infrared thermography).

## **2. Materials and methods**

### **2.1 Stones characteristics**

The tests were carried out on three different kinds of rocky materials, which have different physical features to assess a variety of behaviour regarding water absorption. Specimens sized  $50 \times 50 \times 20$  mm

have been used as indicated by the Italian standard UNI 10859 [11]; data are averages of 5 measurements on 5 specimens.

Noto Calcarenite is a porous yellowish fossil-bearing Miocene calcarenite quarried in Sicily. Its micro-structure is well known and described in literature [15, 16]. 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. The third kind of stone is a dolomitic fine-grained marble, 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 [17].

## 2.2. Thermographic test

The test of Spilling Drops consists on the evaluation of the absorption of one measured drop spilled on the stone surface.

The Spilling Drops (SD) test permits to measure the surface characteristic of absorption and diffusion of liquid water by the thermographic visualization of the damped areas induced by a drop of water: one drop of distilled water (0.03 ml) is spilled on the specimen, then thermographic shooting is carried out for 10 minutes. 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. Analogously the Moisture Ring (MR) test records the water spreading and its evaporation during 10 minutes after the application of a sponge wet with a measured amount of water, with a thermal camera [18 and 19]. It consists in the implementation of the steady test of the contact sponge [20]. Wet surface appears as a circular area because of the sponge circle shape. Both tests use the cooling effect 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 materials.

## 3. 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); Tables 1 and 2 summarize the data regarding the distribution of porosity percentage and Total Open Porosity respectively (data expressed in volume -V%).

	Noto calcarenite	Dorata sandstone	Prilep Marble
Megapores %	12.20	25.88	24.42
Macropores %	86.80	66.99	72.96
Mesopores %	1.00	7.12	2.63

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

IUPAC (International Union of Pure and Applied Chemistry) defined mesopores ranging between 2 and 50 nm, macropores between 50 and 7500 nm, while megapores have width larger than 7500 nm [21 and 22].

Noto calcarenite is characterized by a high porosity (36.2%), due to a large quantity of macro-pores, although also mega-pores are present, the pore diameter is prevalently (about 70%) around 0.5-5  $\mu\text{m}$ , in fig. 1. These characteristics induce the stone to have a high capillarity absorption rate<sup>i</sup>. This variety probably contains ink bottle section pores [23], because the intrusion/extrusion curves obtained by mercury intrusion porosimetry showed that the absorbed mercury is retained inside the materials.

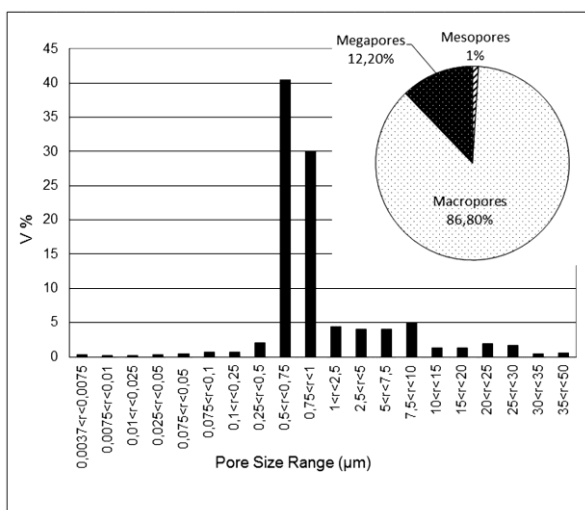


Fig. 1 Noto calcarenite, percentage of total volume (V%) as function of ranges of pores radius.

Dorata sandstone is characterized by a medium porosity (10.7 %) made up of meso, macro and mega-pores; the pore size diameter is distributed over a wide range between 0.01-5  $\mu\text{m}$ . This characteristic gives a high capillary absorption rate also to this stone. This variety probably contains ink bottle section too, since the porosimeter curves shows that the absorbed mercury is held inside the materials.

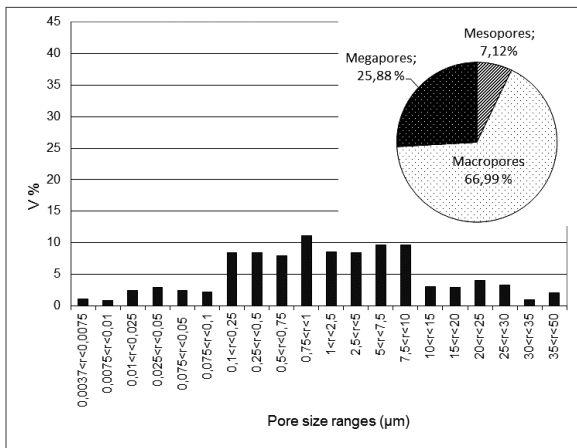


Fig. 2 Dorata sandstone, percentage of total volume (V%) as function of ranges of pores radius.

### 3.2 Contact sponge test

This test was recently suggested as a rapid tool to highlight water absorption capacities on building surfaces. Consequently, it is considered a useful tool for an empiric on-site evaluation of stone degradation without sampling; moreover, it is a valid tool to compare the effectiveness of different water repellent treatments. 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 Absorption by capillarity.

The test has been carried out following UNI 10859.  $Q_f$  is the water amount absorbed at the final step of the test, in our case after 72h, per surface unit (Table 2); 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 [20]. 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.

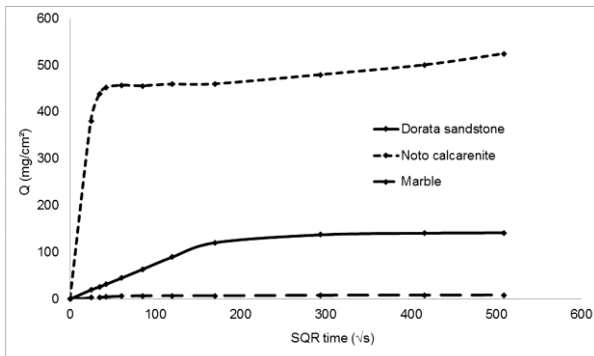


Fig. 3 Water absorption by capillarity (Q) of Dorata sandstone, Noto calcarenite, Prilep marble.

### 3.4 Evaporation flux measurement

In Fig. 4, 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 till a decreasing water content down to 9-10% of WC absolute value (corresponding in the graph to 50% or relative WC).

The results of Dorata sandstone and marble showed similar trends: initial high value of flux, while in the second step the evaporation flux value became progressively similar to the evaporation flux showed in the Noto specimens for very low values of WC.

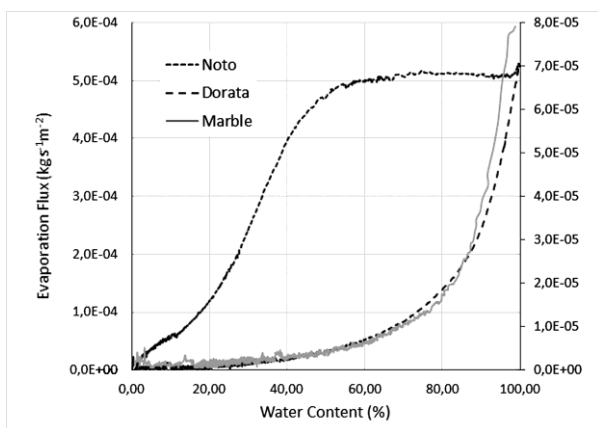


Fig. 4 Evaporation fluxes of three stones under controlled environmental conditions ( $T=25^{\circ}\text{C}$ ;  $\text{RH}=50\%$ ) versus relative water content. The graphic has different scales of Noto stone (on the left) and of Dorata and marble (right)

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 summarizes the data coming from measurements of total open porosity, water absorption (WA) from contact sponge method, Qf and CA from capillarity test and DI.

Stone	Open porosity	WA (g/cm <sup>2</sup> *min)	WA St. Dev.	Q <sub>f</sub> (mg/cm <sup>2</sup> )	CA (mg/cm <sup>2</sup> s <sup>-1/2</sup> )	(DI)
Prilep Marble	0,57	0,00056	9,7 10 <sup>-5</sup>	8,513 ± 3.34	0,115±0.05	0,328
Dorata Sandstone	10,70	0,00257	4,55 10 <sup>-4</sup>	141,769±11.62	0,752±0.16	0,504
Noto Calcarenite	36,20	0,03935	7,373 10 <sup>-3</sup>	524,161±15.50	10,654±0.37	0,442

Table 2 – Physical characteristics of the three stones (average on 10 specimens). WA = water absorption, CA= capillary absorption coefficient, Q<sub>f</sub> = water absorbed in 72h, DI = Drying Index

### 3.5 Optical test

The authors utilised a NIR (Near Infrared) Camera (Jade Camera Cedip, sensitive in the peak of maximum absorption of liquid water at 1.934 μm) to map precisely the liquid water stains on the surface [24]. The images (fig. 6) allowed to distinguish between heat diffusion (cooling due to the evaporation) and water spreading on the surface specimens. Taking into account the high difference between the heat diffusion (measured in m<sup>2</sup> s<sup>-1</sup>) for stone and the velocity of capillary rising the authors' hypothesis is that the water spreading in such materials is faster than the heat diffusion. The NIR shooting procedure is the same used for thermal tests.

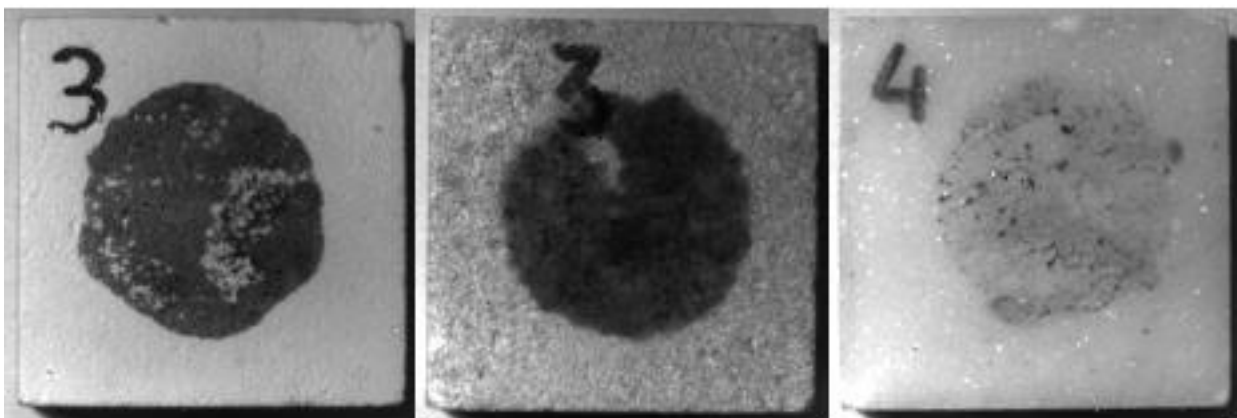


Fig. 5 Images of Noto calcarenite, Dorata sandstone and Prilep marble (from the left) after the application of the contact sponge, shot in the near infrared band



### 3.6 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' 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 6). On the Noto calcarenite surface the wet area is wider than the one on the marble's surface specimen; on both kind of stone materials, the wet area has also a regular circular shape; 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, which 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. 7).

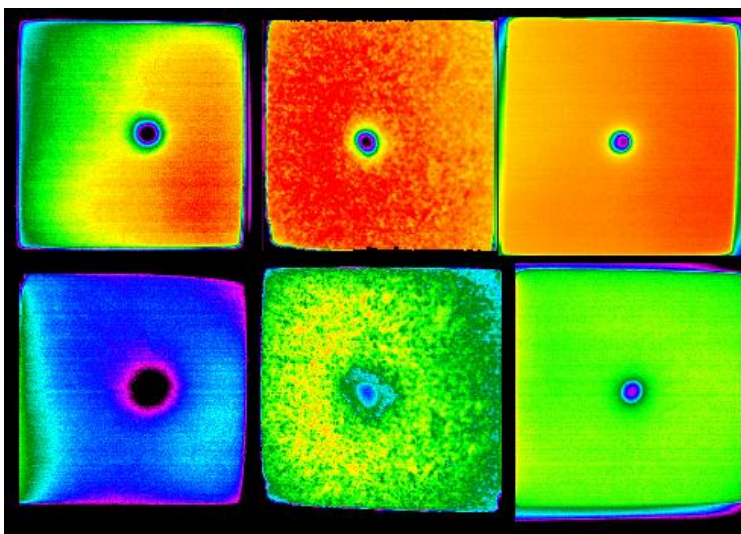


Fig. 6 Thermal images shot at the initial moment of spilling the drop on the stone surface (top line) and 10 minutes after or when the drop geometry became stable (bottom line). From left to right: Noto calcarenite, Dorata Sandstone and marble. Range of temperature 25.0-32.0°C; emissivity ranging between 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	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.

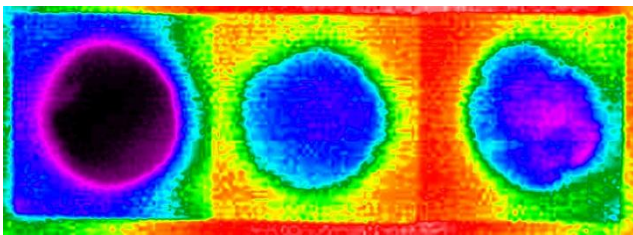


Fig. 7 Moisture ring after the contact with the soaked sponge, from left to right Noto calcarenite, Dorata sandstone, and Prilep Marble, Temperature ranging between 26 and 29°C

#### 4. Discussion

In the MR test the marble surface, characterized by low absorption and low porosity, shows that the wet area has the same size of the sponge and the contour lines are irregular with a thin cooled ring. The effects can be clearly visualized in the thermograms where the moisture ring appears small and irregular if compared to the ones of the other stones.

Noto calcarenite is characterized by a high water absorption capability and a high total porosity, consequently there is the highest evaporation rate. This wet area appears well defined in thermal images with a neat contour line of the ring. The contour line in the image at 1.9 micron (fig. 5) clearly splits the area where the pores are filled of water and the area where the pores are empty.

In Dorata sandstone, the two experimental techniques (MR and SD) tested the difference of water transport behaviours: on Dorata sandstone, the drops extensively spread. In fact, the increment of the area is the highest (+137%) whereas liquid water from the sponge has a lower absorption

(0.0025 g/cm<sup>2</sup>\*min) and consequently a low spreading; optical test confirmed the trend of the lower absorption. The authors hypothesized that the water content in the drop behaves as a sort of “reservoir” compared to the water amount contained in the soaked sponge. In this latter case, the water is distributed between the sponge and the stone pores as a function of the dynamic equilibrium due to the difference of the amount (and size) of pores in the sponge and in the stone itself. As already discussed in the paragraph of optical tests, the hypothesis is that the pores on the Dorata sandstone’s surface are half-filled with water or are visible only the small pores with a smaller evaporative surface. This provides a lower evaporation rate, hence a lower cooling. The cooling effect (highlighted by the MR and SD tests) is the smallest among all specimens, and in thermal images the contour line of the colder zone is totally irregular.

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 these areas as the delimitation of the area of interest, the size of the pixel of thermograms, the shape of the pixel, the threshold of the background temperature. The determination and study of each of these factors will be a focus of the next part of the research.

Finally, figure 5 shows the trend of WA versus total open porosity. WA has also a direct correlation with the coefficient of water absorption by capillarity (CA) ( $R^2 > 0.999$ ).

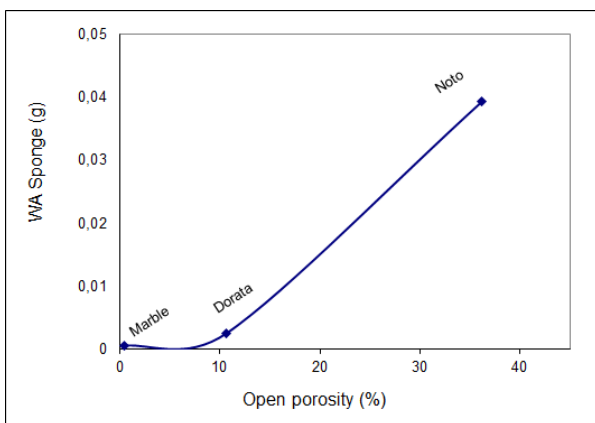


Fig. 8 Water absorption versus total porosity of the specimens

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 an increase for 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 smallest considered) that contributes to water spreading into the bulk but not to the surface evaporation.

## **5. 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. Thermography procedures are innovative, and data crossing with standardized procedures shows their validity. In particular, 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 texture 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 for the best conservation of the material, 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 [19]. The experimental phase showed some limits of the application on site that the authors plan to overcome using the experience gathered with this work. For example, the low temperature of the surface and the environment, the location/orientation of the surfaces to test that could prevent evaporation, and in case of damaged surfaces (presence of black crusts, deposits, cracks, blistering, flakes, etc), a preliminary cleaning is strictly required.

The contact sponge test dates to 2003; the method was recently standardized by the working group UNI Cultural Heritage. The improvement due to the standardization of the test procedure will bring

advantages also to the moisture ring tests, especially for spreading and validating the application of water by contact sponge in the field: a standardised procedure for wetting the surface will eliminate the difficulties in the evaluation of thermal images regarding evaporation phenomena on stones.

#### Acknowledgements

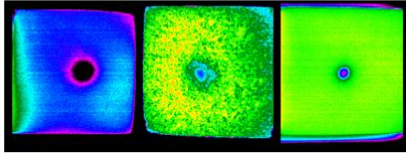
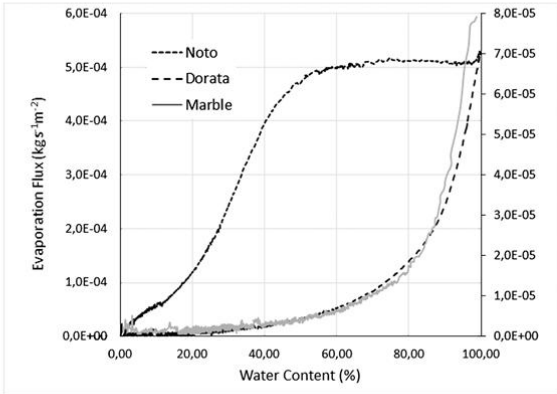
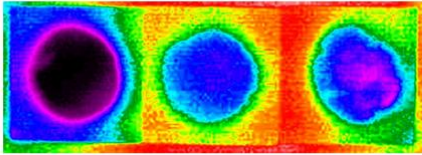
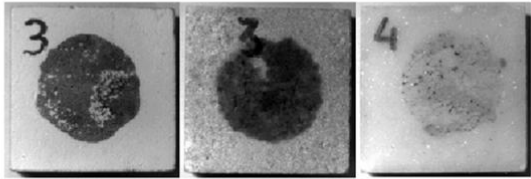
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Graphical abstract



**Innovative IRT application to detect differences in microstructure of stone surfaces**

## Highlights

1. The tests of Moisture Rings (MR) and Spilling Drop (SD) allowed to distinguish the different kinds of porosity
  2. Typology of pores influences the shape of spilling water drops, apparent at IR
  3. Prevalence of macropores induce regular shape in thermal image
  4. Prevalence of megapores produce irregularity in thermal images
  5. Mesopores are supposed to contribute to water spreading in the bulk instead of surface evaporation
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