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Original article

Contact sponge method: Performance of a promising tool for measuring the initial water absorption

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Abstract

Porous limestone and mortar are able to absorb large quantities of water. This phenomenon will accelerate the deterioration of the material. In such cases, the material might be treated with a hydrophobic product, which creates a superficial layer that hampers the penetration of water. In order to decide if such a treatment should be applied or not, the water absorbing behaviour of the material should be measured. With the same measuring technique the efficiency of the hydrophobic barrier can be evaluated. Moreover, it allows the monitoring of such barriers as a function of time. At the same time, the water absorption of porous stone material is an indication of the degree of deterioration and its sensitivity to future deterioration. Up to now, two different measuring techniques exist, but one can only be used in laboratory and the other, which can be operated in laboratory as well as *in situ*, is not always reliable for *in situ* analyses. This article proposes an alternative method: the contact sponge method. This recently developed method was tested on non-treated porous stone materials in a laboratory environment in order to evaluate its performance in comparison with the two existing methods.

Keywords: Porous stone materials; Water absorption; Contact sponge; Capillary; Karsten pipe

1. Research aims

Water inside cavities of porous stone materials such as limestone or mortar is one of the main causes of accelerated deterioration [1]. Therefore, the measurement of the initial water absorbing behaviour is a helpful tool in the estimation of the stone sensitivity to future deterioration. The porosity is also an indication of the degree of decay [2]. Moreover, such information allows the selection of a proper conservation treatment. For example, when a stone material is considered as too porous and therefore too prone to deterioration, a hydrophobic product can be applied to its surface. The measurement of the water absorbing behaviour can also be used to evaluate the efficiency of the treatment and to monitor the quality of the protective barrier as a function of time.

The pore network and the water transfer between the environment and the stone can be studied by combining several analytical techniques such as mercury porosimetry, Brunauer–Emmett–Teller (BET) measurements, 2D image analysis [3], open porosity and water absorption by capillary rise [4]. However, these techniques can only be used in a laboratory environment and therefore, they require a destructive sample

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preparation [5]. For precious and historic objects of art, the removal of samples is not always possible. The Karsten pipe method and the contact sponge method [6] are the most important non-destructive techniques that allow *in situ* measurements of the water absorption of porous stones. The Karsten pipe method was developed to evaluate the efficiency of water repellent treatments under heavy rainfall circumstances. The height of the water column of 98 mm in the pipe exerts a pressure on the surface on which it is attached, corresponding with rain drops hitting the wall with a static wind velocity of 140 km h^{-1}, perpendicular to the surface [7]. However, this technique does not measure the initial water absorption (i.e., the immediate water absorption within a small period of time by the superficial layer) because the first measurement with the Karsten pipe is only carried out after 5 min. Moreover, this technique has other disadvantages: the method is not easy to handle [8] and *in situ* measurements are seldom reproducible. In order to measure *in loco* the initial water uptake by porous materials, either treated or non-treated, Tiano and Pardini [6] developed the contact sponge method. This technique is fast, cheap, non-destructive, easy to use, it can be employed *in situ* as well as in laboratory and the preliminary results are very promising [6]. However, there is a lack of published results concerning this technique and so far, there is no standardized procedure available. This hampers the practical use of the contact sponge method. In this article, the contact sponge method is evaluated and compared with the capillary rise method and the Karsten pipe method. In this paper water absorption measurements were carried out in laboratory on non-treated materials, with homogeneous and heterogeneous porosity.

2. Theoretical background

The contact sponge method, the capillary rise method and Karsten pipe method can be used for measuring the absorption behaviour of porous stone materials. The three methods can be used to determine the water absorption per unit area. The principle of these three techniques will be described in this paragraph.

2.1. Contact sponge method

The contact sponge method consists of a 1034 Rodac® contact-plate (with 5.6 cm diameter) containing a humid sponge *Calypso natural make-up* from Spontex® (with approximately 7 g of water) which is thicker than the height of the borders of the contact-plate. The contact-plate can be pressed against a stone surface during a certain period of time (e.g. 90 s). When this method is employed manually, a maximum amount of pressure is applied, which is determined by the contact of the borders of the contact-plate with the examined surface. The pressure may also be controlled by using a dynamometric spring (DS) produced by SINT Technology (Fig. 1 – Left), in which depth levels vary from 1 to 4 corresponding to: 3.42×10^{-3}, 7.55×10^{-3}, 11.80×10^{-3} and 15.23×10^{-3} MPa, respectively (data converted from [6]). In this study, the contact-plate with sponge was placed on a table and the test material was pressed onto the sponge. Therefore, the water absorption by the sample was opposed to the gravity force, resembling the measurement circumstances of the capillary rise method (Fig. 1 – Right).

A balance measures the initial and final weight of the contact-plate containing the humid sponge before and after contact with a surface. The difference in weight corresponds with the amount of water which has been absorbed by the material surface under test. Results are then expressed by the mass difference as a function of area and time. In formula (1) Wa represents the water absorption per square meter and unit time, m_i the initial weight of the sponge inside the contact-plate, m_f the weight of the sponge inside the contact-plate after contact, A is the sponge area (0.002376 m^2) and t is the time.

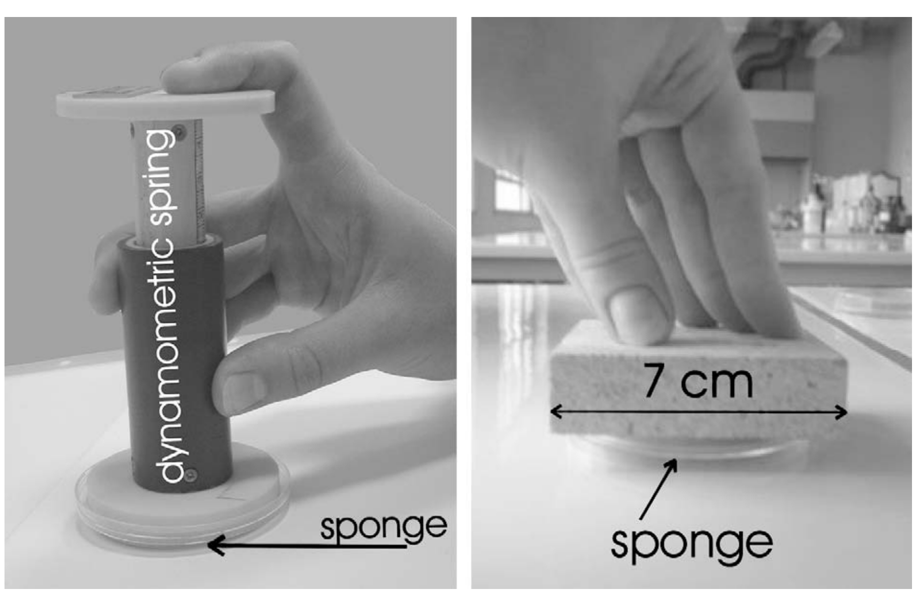

Fig. 1. Left – Pressure applied on contact sponge with a dynamometric spring. Right – Manual pressure applied on a stone sample, which absorbs water from the sponge by capillarity rise.

$$Wa\left(\mathrm{g/m^2\ s}\right) = \frac{\Delta m}{A \cdot t} = \frac{(m_i - m_f)}{A \cdot t} \quad (1)$$

2.2. Capillary rise

In the capillary rise method the porous stone material is placed on top of a pile of humid filter papers. The water from the filters is absorbed by the stone by means of the upward capillary force. Capillary rise tests were performed according to UNI/NORMAL 10859 [9] but only initial absorption times were considered (30, 60, 90 or 120 s) in order to compare the very first initial water uptake of this method with the contact sponge method. The aim was not to calculate the water absorption coefficient because only the most superficial layer of the material is being tested with the contact sponge. Results are calculated with Eq. (1). The surface A is equal to the contact area between the sample and the filter paper (stone sample area). The initial and final mass of the sample is respectively m_i, m_f.

2.3. Karsten pipe

The Karsten pipe method consists of vertical pipe filled with water, which is attached with plastiline to the surface of a porous stone material. The method was applied according to the RILEM II.4 recommendation [10]. The contact area A between the pipe and the stone is 0.000573 m^2. The water absorption by the stone after 90 s, 5 min, 10 min and 15 min is determined by measuring the reduction of the water volume in the graduated pipe. These values can be converted to the corresponding mass $m_{(90s)}$, $m_{(5min)}$, $m_{(10min)}$ and $m_{(15min)}$. The average water absorption $C_{KB10min}$ between 5 min and 15 min per unit area can be calculated with Eq. (2). In case all the water in the pipe was absorbed before 15 min, an extrapolation was made from the data points $m_{(90s)}$, $m_{(5min)}$ and $m_{(10min)}$. The water absorption during the first 90 s C_{KB90s} was also measured to be able to follow the initial water uptake and compare results with those of the contact sponge (Eq. (3)), where $m_{(90s)}$ is the volume of water absorbed after 90 s converted into mass and t_{90s} is 90 s time.

$$C_{KB10min}\left(\mathrm{g/m^2\ s}\right) = \frac{\left(m_{(15min)} - m_{(5min)}\right)}{A \cdot t_{10min}} \quad (2)$$

$$C_{KB90s}\left(\mathrm{g/m^2\ s}\right) = \frac{m_{90s}}{A \cdot t_{90s}} \quad (3)$$

Table 1
Overview of the recipes with which the mortars were made. The relative amounts of the type of aggregate, binder and water are given between parentheses.

Symbol	Type of aggregate	Type of binder	Water
R	Red chamotte (3)	Unilit Fen-XA hydraulic lime (1)	(0.5)
Z	White sand (2)	Unilit Fen-XA hydraulic lime (2/3) Portland-cement (1/3)	(1.0)
G	Expanded chamotte (2)	Boehm hydraulic lime (1)	(0.5)

Table 2
Average values of the real density and open porosity of the limestones and mortars [13–15].

Material	Symbol	Real density (kg m^{-3})	Open porosity (%)
Limestone			
Massangis Roche Claire	MC	2427 ± 5	10.7 ± 0.2
Massangis Roche Jaune	MJ	2400 ± 120	10.5 ± 0.9
Senonville	S	2388 ± 7	10.6 ± 0.2
Valanges	V	2300 ± 100	14 ± 2
Magny Doré	M	2290 ± 20	15.6 ± 0.8
Tercé	T	2060 ± 75	24 ± 5
Estaillades	E	1920 ± 20	29.3 ± 0.6
Mortar			
Mortar R	R	1744 ± 0.1	32.2 ± 0.7
Mortar Z	Z	2004 ± 3	37 ± 6
Mortar G	G	1270 ± 1	57 ± 6

3. Experimental

In order to evaluate the reproducibility and the sensitivity of the contact sponge method, the capillary rise method and the Karsten pipe method, measurements were performed on three series of test materials: thin layer chromatography plates (TLC), limestones and mortars. All tests were performed in a laboratory environment (average temperature of 20–22 °C and 55% relative humidity). For all methods, the mass of the samples in the dry state was obtained after they were dried in an oven at 60 ± 5 °C for two to three days and cooled down to room temperature in a desiccator. Water absorption measurements were carried out with distilled water. For the contact sponge method and the capillary rise method, the mass of respectively the sponge and the stone samples was measured with a balance with a precision of 0.001 g.

3.1. Thin layer chromatography plates (TLC)

The thin layer chromatography plates (DURASIL-25 produced by Macherey–Nagel®) were used to optimise the working procedure of the contact sponge method. The cavities have a pore diameter average of 6 nm and the surface of the cavities is coated with unmodified silica 60 [11]. This material was chosen because of its constant and well-known porosity. In this preliminary test the effect of the operator, the pressure on the sponge (pressure obtained with a dynamometric spring and manual pressure), the amount of water in the sponge, and the contact time on the measurements were evaluated. The TLC-plate was placed on the table and the humid sponge inside the contact-plate was superposed on the porous layer. Pressure was applied on the contact-plate (Fig. 1 – Left).

Table 3
Average water absorption (g/m^2 s) obtained by two types of springs used by two different operators. The listed uncertainties correspond to the standard deviation on the average values. Each measurement was carried out 5 times.

	Spring I	Spring II
Operator A	24.721 ± 2.033	25.119 ± 1.861
Operator B	24.165 ± 1.483	24.040 ± 0.894

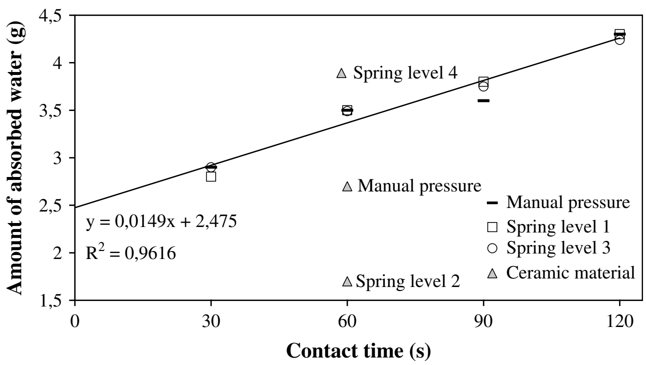

Fig. 2. Effect of contact time and pressure on the amount of absorbed water measured on TLC-plates. Literature data from [6] measured on a ceramic material (▲) with a contact time of 60 s and different pressures (manual and with a spring at levels 2 and 4).

3.2. Limestones and mortars

A series of limestones and mortars with different porosities were used to compare the contact sponge method with the capillary rise method and the Karsten pipe. The open porosity of the limestone and mortar series was determined according to the prEN 1936 [12] protocol. From the seven types of limestone and the three types of mortars, several samples ($7 \times 7 \times 2$ cm^3) were made. On each sample, the water absorption was measured with the three methods. The obtained data set was used to evaluate and compare the reproducibility and sensitivity of the three methods.

For the seven French limestones, the open porosity has been determined by the Belgium Building Research Institute (BBRI) [13–15]. For the three types of mortars, different aggregates and hydraulic lime materials were used to control their open porosity. The recipes used for their production are given in Table 1. The porosity of the mortars was measured according to the prEN 1936 protocol. An overview of the properties of the limestones and the mortars is summarized in Table 2.

4. Results and discussion

4.1. Working conditions of the contact sponge method

For the contact sponge method, the effect of the operator, the pressure on the sponge, the amount of water in the sponge, and the contact time were evaluated by using TLC-plates. These plates were used in order to reproduce the same experimental conditions used by Tiano and Pardini [6] for similar measurements.

In a first test, the water absorption of TLC-plates was measured by using an initial water amount in the sponge of ca. 7 ml and with a contact time of 60 s and a pressure by applying a dynamometric spring in level 1. The amount of water should be optimised for each type of material, depending on its porosity. In order to study the effect of the operator and the effect of the type of dynamometric spring, two operators performed exactly the same series of measurements. Each measurement was carried out 5 times. The results for the two operators and for the two dynamometric springs are summarized in Table 3. It can be concluded, that the type of spring and the operator did not have a relevant influence of the measured water absorption.

The evaluation of the impact of the contact time and the amount of pressure on the sponge was performed in a second series of tests on TLC-plates. The pressure was applied manually or with the spring using levels 1 and 3. For each amount of pressure, contact times of 30 s, 60 s, 90 s and 120 s were applied. All measurement circumstances were carried out four times. These repetitive measurements show that the variation coefficient (stdev/average $\times$ 100) was smaller than 7%. The average values of the measurements are summarized in Fig. 2 and it includes data from Tiano and Pardini [6] measured on a ceramic material. Fig. 2 suggests a linear relation between the contact time and the amount of absorbed water and a limited effect of the applied pressure on the amount of absorbed water for TLC-plates. Therefore, pressure has a negligible effect of the water absorption for TLC-plates.

The data of Tiano and Pardini [6] demonstrate a clear relation between the pressure and the amount of absorbed water. This could not be confirmed by our experiments. This might be explained by a difference in test material. In this research TLC-plates were employed; Tiano and Pardini used ceramic materials (ARS described in [16]). The properties of both types of materials are not the same. The ceramic material has 28% porosity and an average pore size diameter which is 1000 times larger than the TLC pores, which is ca. 6 nm. Due to the small pores of TLC-plates, the effect of the applied pressure is negligible compared to the high intermolecular forces between the liquid and the silica, which is responsible for the capillary effect. Consequently, only very high external pressures would lead to differences in the rate of water uptake.

Table 4
Average water absorption (g/m^2 s) for three limestone types (MC, T and E) with different open porosities. For each stone type, the average was calculated from 4 consecutive measurements on 5 different samples. For all average water absorption values the standard deviation is mentioned. The second column contains the variation coefficients.

	Massangis R. Claire (open porosity: 10.7%)		Tercé (open porosity: 24%)		Estaillades (open porosity: 29.3%)	
	(g/m^2 s)	(%)	(g/m^2 s)	(%)	(g/m^2 s)	(%)
Contact sponge 90 s	2.7 ± 0.2	7.1	20 ± 2	8	32 ± 2	5
Capillarity rise 90 s	2.1 ± 0.2	11.5	13 ± 1	9	30 ± 4	14
Karsten pipe 90 s	13 ± 5	42	114 ± 26	23	185 ± 115	62
Karsten pipe Δ 10 min	14 ± 6	41	134 ± 20	15	183 ± 109	59

Table 5
Average open porosity and water absorption (g/m^2 s) (average ± standard deviation) for 7 limestone types and for 3 mortar types. For each type, the average was calculated of 16 samples.

Stone type	Open porosity (%)	Contact sponge 90 s (g/m^2 s)	Capillary rise 90 s (g/m^2 s)	Karsten pipe 90 s (g/m^2 s)	Karsten pipe Δ 10 min (g/m^2 s)
Limestone					
MC	10.7	2.7 ± 0.2	2.3 ± 0.2	16 ± 5	8 ± 2
MJ	10.5	3 ± 0.8	2.8 ± 0.6	18 ± 7	8 ± 3
S	10.6	3.3 ± 0.4	2.9 ± 0.4	15 ± 5	12 ± 3
V	13.5	3.7 ± 0.3	2.6 ± 0.2	16 ± 6	11 ± 3
M	15.6	5.8 ± 1.5	6 ± 1.5	38 ± 26	16 ± 9
T	23.7	19 ± 2	13.2 ± 1.8	135 ± 24	112 ± 22
E	29.3	31 ± 3	32 ± 5	297 ± 98	309 ± 134
Mortar					
Z	37.3	7.0 ± 0.4	6.3 ± 0.4	63 ± 45	41 ± 36
R	32.2	10 ± 2	8 ± 2	72 ± 30	28 ± 14
G	57.1	18 ± 3	16 ± 2	139 ± 31	130 ± 28

4.2. Comparison of the three methods

The contact sponge method will only be employed in practice if its performance is at least as good as the capillary rise method and/or the Karsten pipe method. Therefore, the performance of these three methods will be compared with each other. For this, the reproducibility of measurements and sensitivity was studied in detail for the three methods. Two different measuring circumstances were employed for the Karsten pipe method.

4.2.1. Reproducibility

The water absorption has been determined for three limestone types (MC, T and E). This has been done for five samples of each type. For each sample, the water absorption has been measured with the three methods and for each method four consecutive measurements were carried out. The contact sponge method was applied manually during 90 s. For the capillary rise method a contact time of 90 s was employed; for the Karsten pipe a contact time was used according to Eqs. (2) and (3). Table 4 gives an overview of the average water absorption for each limestone type and for each method. The variation coefficient on these results is a measure of the reproducibility of the method. Contact sponge and capillary rise gave reproducible results on the tested materials (variation coefficient below 14%), whereas the pipe method was less reproducible (variation coefficient between 15 and 62%). This test indicates that a similar performance is obtained for the contact sponge method and for the capillary rise method.

4.2.2. Sensitivity

In order to estimate the sensitivity of the three techniques, 16 samples of seven limestone types and of three mortars were measured once. In Table 5 the average water absorption and its standard deviation for each set of 16 samples is summarized and compared with its open porosity. The data in Table 5 confirm the larger variation coefficients for the Karsten pipe method, as already noticed in Table 4. Moreover, the water absorption increases with the open porosity. This relation is shown in Fig. 3.

Although a relation between the open porosity and the average water absorption exists for limestone and for mortar types, this relation is clearly not the same. Due to the high pressure of the water exerted on the stone surface by the Karsten pipe method (9.2 MPa, data taken from [7]), this method measures the water absorption of deeper areas. The contact sponge method and the capillary rise method (within 90 s) measure the water absorption of a more superficial layer. Due to the carbonation process of mortars, the open porosity of this top layer is smaller than the open porosity of deeper regions [17]. Therefore, the relation between the average open porosity of the mortars and the corresponding water absorption is different.

In Fig. 3 the relation between the open porosity and the average water absorption is shown for the limestone types. The data obtained with the four methods could all be interpolated with an exponential function. The correlation coefficient was better than 0.96. The Karsten pipe method is clearly the most sensitive technique but its low precision makes it less reliable than the other methods. Moreover, it measures a much higher absorption at high porosities. This is in accordance with the behaviour of sound stones since the penetration of water in depth follows an exponential function [7]. Fig. 4 demonstrates a linear relation between the water absorption values obtained

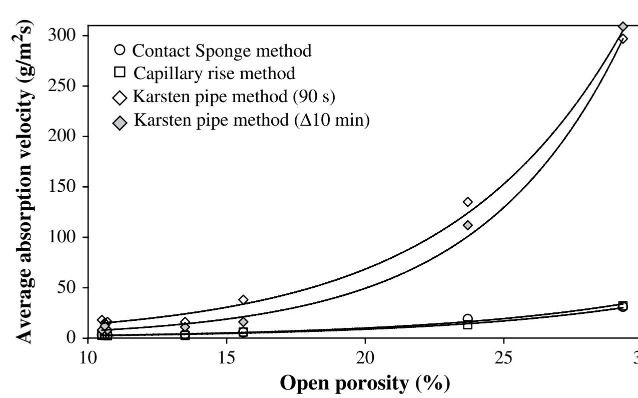

Fig. 3. Relation between the open porosity of limestone types and the average water absorption for all methods used.

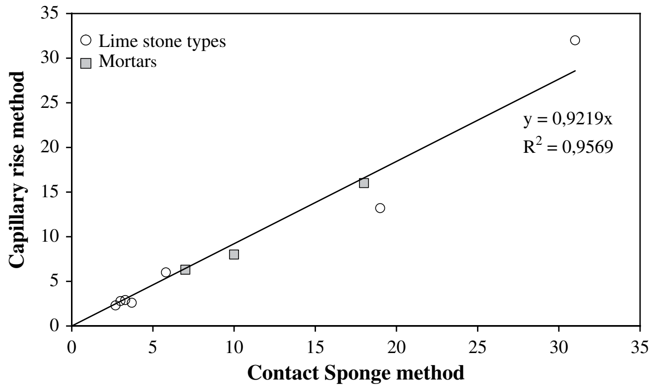

Fig. 4. Linear relation between the average water absorption measured with the contact sponge method and the capillary rise method.

with the contact sponge method and with the capillary rise method. Therefore, no significant difference in performance between these two techniques can be observed.

5. Conclusions

The performance of the contact sponge method, a recently developed measuring technique, was compared with two existing and well-known techniques: the capillary rise method and the Karsten pipe method. In this test, the Karsten pipe method appeared to be the most sensitive technique but its precision is rather low. The capillary rise method and the contact sponge method have a similar precision and sensitivity, but the last method has the additional advantage that it is non-destructive, easy to use, portable and it may be used in monitoring programmes with very low costs. These conclusions are based on a statistical study of laboratory measurements performed on 7 limestone types and 3 mortar types with different open porosities.

For the contact sponge method, it was noticed that the operator and the type of spring did not have any significant influence on the measured water absorption, whereas the contact time has a proportional effect on the absorbed water.

It should be remarked that the contact sponge method does not replace the capillary rise method (used in laboratory for general characterisation of sound, weathered and treated materials) nor the Karsten pipe (used *in situ* to determine if a surface is still water repellent) but should be considered as a complementary technique. The Karsten pipe method measures the water absorption of rather thick layers of stone, while the contact sponge method measures the same property for much thinner top layers. Moreover, the pipe method allows determining the water absorption coefficient of materials, which is not dependent of a time interval as the contact sponge method is, which in this last case can compromise the comparison of results among different authors. An important feature of the Karsten pipe method is the simulation of the pressure of driving rain, a pressure which is known to be able to ‘break’ temporarily the water repellent behaviour of a treated material (an effect that is typical for more porous stones, and not so much for compact materials such as concrete or marble). Nevertheless, for conservation/restoration purposes, the outmost superficial layer is much more important because deterioration processes, either of treated or non-treated stones, normally take place at the stone surface. Therefore, the contact sponge method is recommended to evaluate the water absorption of a material just below the surface. With this information, the conservator/restorer has more control in the evaluation of the need of a treatment and selection of the most appropriate product.

Given the extraordinary positive results in laboratory this method is being further tested *in situ*, on a complex system where variables as climate, stone deterioration and past conservation interventions play a decisive role on the handling and interpretation of results.

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