New Procedure for Performing Moisture Absorption Tests

A highly accurate, automatic procedure for absorption tests allows comprehensive quantification of processes including immersion, vapor absorption, evaporation and capillary absorption.

María J. Mosquera, Rodrigo Alcántara and Joaquín Martín
Departamento de Química-Física
University of Cádiz, Cádiz, Spain

The decisive role water plays in the deterioration of historical buildings is well-known and widely documented. Therefore, characterizing the different types of moisture transport involved (water absorption by capillarity and immersion, water vapor absorption or hygroscopy, and drying behavior) is relevant for studies of the alteration of porous material.

To quantify these absorption processes, various working procedures have been proposed in the current standards (RILEM, CNR-ICR, ASTM, ISRM), depending on the solid–water interaction process concerned. However, all are characterized by the same general principle: measuring the evolution of the sample mass at pre-set time intervals, which requires interrupting the process being studied.

To resolve the disadvantages presented by interrupting the absorption process, our research team devised an earlier, automatic procedure for performing absorption tests by capillarity. This test uses the principle of continuous measurement of the kinetic process. Recently published initial results indicate that the new procedure is suitable for this purpose, and represents an important advance over existing procedures.

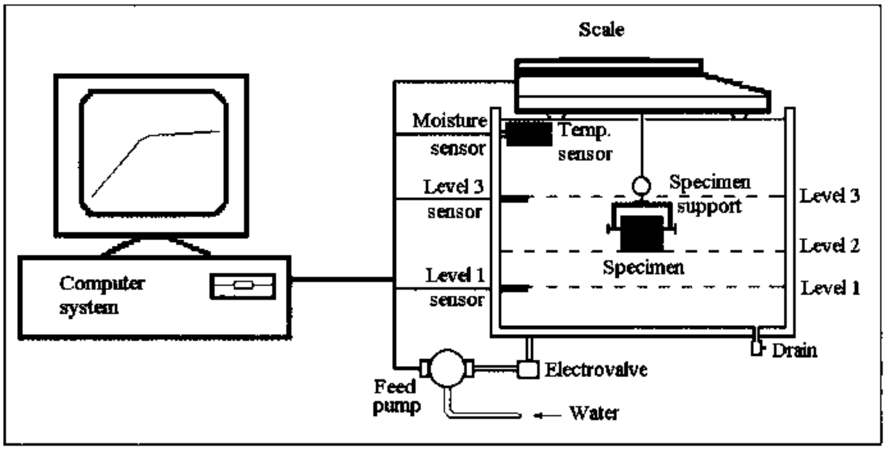
Moreover, the application of the new methodology has proved to be essential for samples with small dimensions, and for highly porous materials characterized by high absorption rates and friability, for which the use of an existing standard protocol is not possible.

However, our experience has enabled us to detect slight errors in absorption profiles, basically associated with the dynamic instability of the water level. In the last year, this automatic procedure has been substantially modified to provide more accurate data sets and to permit the complete quantification of other types of absorption processes: immersion, vapor absorption and evaporation. To fulfill these requirements, the new device's design incorporates the following improvements:

- Automatic control of the interaction between the water level and the sample;
- Automatic inspection of environmental conditions during test performance;
- Specific inelastic specimen-support that allows total contact between water and specimen while holding the specimen in the horizontal position.

Instrument Specifications

- Specimen chamber: The water–sample interaction is performed in a thermally isolated receptacle where a stable environment is maintained (deviations were <5% in all tests performed). An environment sensor interfaces with the computer system to monitor temperature and relative humidity parameters.
- Computerized weighing system: A scale capable of weighing by suspension and that can be interfaced with the computer system is used.
- Specimen support system: The specimen-holder used has two parts—the actual



General schematic diagram of the instrumentation used to perform the procedure proposed in this article.

support that holds it by lateral fixings so the solid–water contact is complete; and an inelastic suspender that prevents alterations in the specimen's level during the test. The parts are attached to each other by magnets, which allows adequate control of the specimen horizontality.

- Computerized water control system: The water level in the chamber's interior should be different for each type of test. Water can be regulated to required levels through a computer program that adjusts the water inflow using a low-flow electrical pump and a narrow-gauge electrovalve. At the start of the experiment, the software operates the filling system, opening the electrovalve and switching on the electrical pump. It provides a slow water inflow into the specimen chamber, avoiding water surface fluctuations. The software controls the switching-off of the filling system in different ways, depending on the type of absorption test being performed:
  - Water vapor absorption and evaporation: The water level should be set below the specimen, without any physical contact with it. A sensor located at this position detects the required level.
  - Water absorption by capillarity: For this type of test, the water level must coincide exactly with the lower surface of the sample. The required level is detected by the variation in the sample weight when the first contact occurs. In this way, the water surface is always stabilized at the correct level independent of sample length, minimizing errors associated with the effects of floatability and surface tension.
  - Water absorption by immersion: The specimen must be fully immersed in the water. A sensor located over the specimen monitors the required level.
- Computer system: A computer system with software written especially for this procedure is used to meet two objectives—the control of water–sample interaction, and the continuous recording of the data on mass and environmental parameters.

As soon as the correct water level is attained in the specimen chamber, data are recorded. In the specific case of water vapor absorption and evaporation tests, the computer system offers two options for beginning data recording: begin recording as soon as the correct water level is attained, or begin recording when specific environmental parameters are attained.

Experimental Procedure

To prove the efficiency of the new methodology, four different absorption tests (water vapor absorption, evaporation, capillarity and immersion) were carried out on a series of selected stone materials. The absorption tests were performed according to specifications, complying with the following rules:

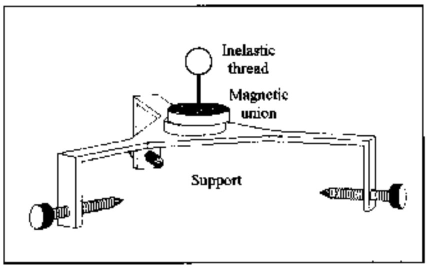
1. Five samples consisting of a 3-cm cube of each variety of stone were tested in every absorption test.
2. Prior to the performance of the absorption tests, all the samples were dried at 60° until constant mass was recorded.
3. In the water vapor absorption and evaporation tests, data recording began as soon as the required water level was attained. Respectively, 98 and 60% of the moisture content was achieved in the absorption and evaporation test.
4. All the tests were finished when saturation was attained, except for the hygroscopy test that lasted for eight days.
5. The evaporation test was performed as soon as the water vapor absorption test was concluded, without modifying the position of the specimen.
6. Since stone materials are usually anisotropic, capillarity absorption profiles are orientation-dependent. In this case, attention is restricted to mean values for measurements perpendicular to and parallel to the direction of stratification.

In relation to the results, absorption rates were expressed in %water absorbed/min, except for the capillary test. In that test, the absorption rate was defined as the flux to a surface. Therefore, it was expressed in $g/(cm^2\cdot min)$.

Stone Materials

Three rock types widely used in construction that are significantly different in mineralogical composition, including granulometry and porosity, were tested using the new procedure:

- Biocalcarenite from a quarry in El Puerto de Santa María (Cádiz, Spain) is composed of calcite (>50%) and quartz (10–30%);
- White limestone from the Estepa quarry (Sevilla, Spain) is composed of calcite (100%). This variety has a medium porosity of 10–12% and a



Schematic diagram of the specimen support specifically designed for obtaining a complete contact specimen-water.

high homogeneity in pore radii (60% of the pores show a size between 130 and 240 nm).

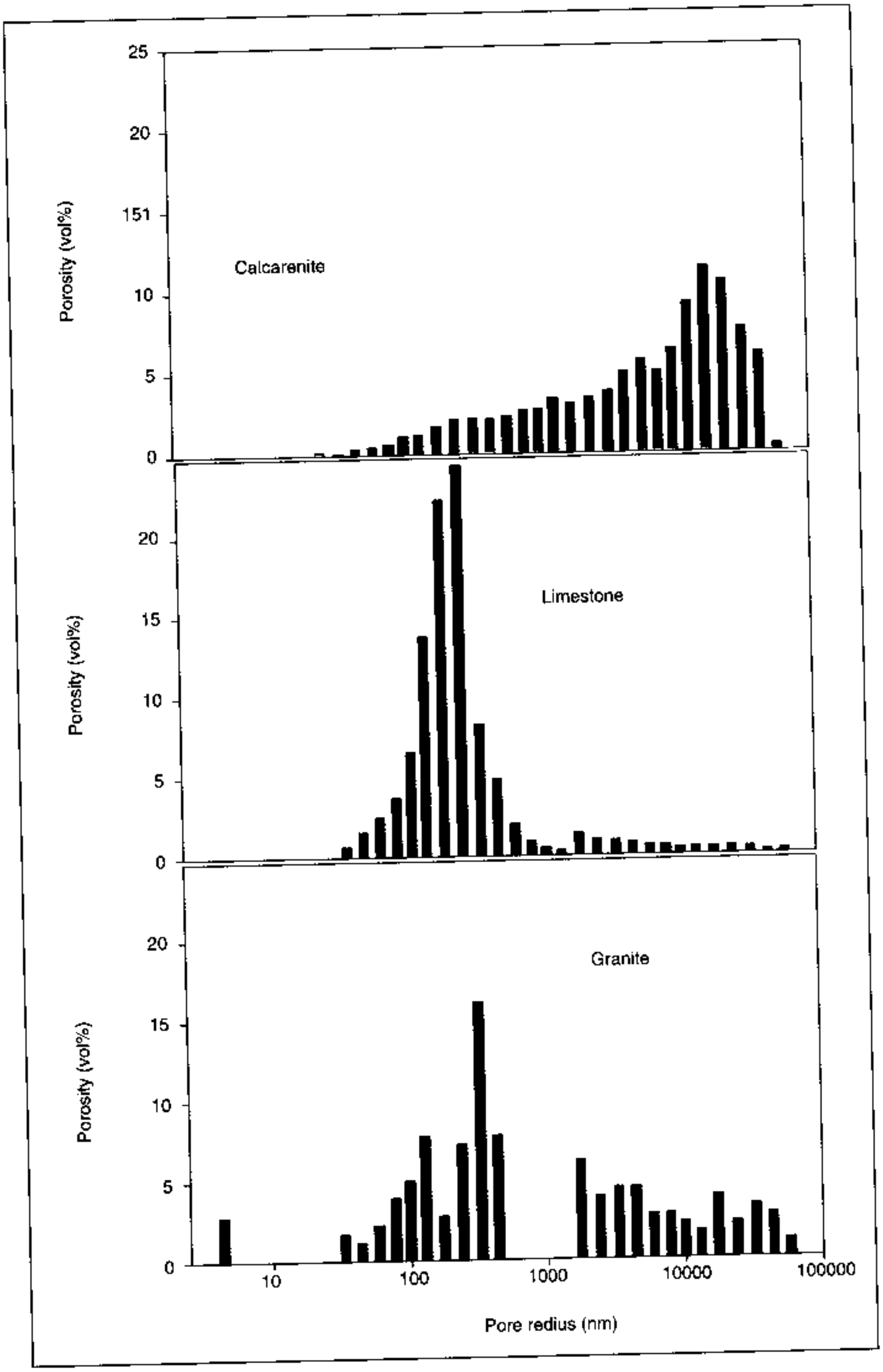
- Granite from the Roan quarry (La Coruña, Spain) is composed of quartz (44%) and is fairly homogeneous, with no apparent orientation. It presents a low open porosity (2%) due to plate fissures.

Preliminary assays were carried out to characterize the stone materials:

- Petrographic (X-ray-diffractometry and microscopic analysis of thin sections);
- Porosity (RILEM procedure (1980) and mercury intrusion porosimetry (MIP)).

Results

The new protocol presented here was performed without problems on all the selected materials, confirming the methodology's wide possibilities. The proposed protocol still is based on the same fundamental concept as our first methodology: detecting changes in the sample mass without removing it from the experimental environment, enabling the efficient, exhaustive and continuous monitoring of the entire process. Thus, all advantages listed in earlier articles are common to the present procedure and applicable to the different types of tests that can be conducted.



Pore radii distribution obtained by mercury porosimetry.

On the other hand, our protocol shows an important advantage over the continuous measure procedures exclusively designed for capillary absorption: the performance, using one single device and one single protocol, of every type of water absorption process in both the liquid and vapor phases.

In fact, this article presents data obtained from four different tests (hygroscopy, evaporation, capillarity and absorption by immersion) that have been performed without making any modification to the experimental protocol or the equipment. According to the experimental results, specific advantages for each absorption test were obtained.

Water Vapor Absorption, Evaporation

Under current standard procedures, kinetic study of sedimentary material is difficult, and, in the case of granite, impossible. In granite, the alterations resulting from the removal of the specimen are greater than those resulting from the absorption of water. However, using present protocol allows the absorption and the subsequent evaporation rates to be fitted to a kinetic dependent on the square root of the time ($r > 0.99$) for the three selected materials.

Although calcarenite (28%) has a significantly greater porosity than limestone (10%), the absorption–desorption rates are similar for both materials. Limestone pores are smaller than calcarenite pores, creating a large specific surface to which water vapor molecules can adhere.

Capillary Absorption Test

The new water level control system overcomes all the disadvantages of our earlier protocol. In our first method, the dynamic water level control system generated vibrations in the water surface that were transmitted to the weighing scale, producing slight fluctuations in the absorption profiles.

In the new procedure, once contact is established between sample and water, the filling system stops, eliminating this factor as a cause of surface vibrations. Further, the automatic control of specimen–water contact detected by sample mass variation gives a satisfactory reproducibility of the test results, minimizing floating or surface tension effects.

In our first procedure, the water–sample contact, attained by mechanical means from the vertical displacement of the measuring tank, caused water level adjustments to be inaccurate in a series of experiments.

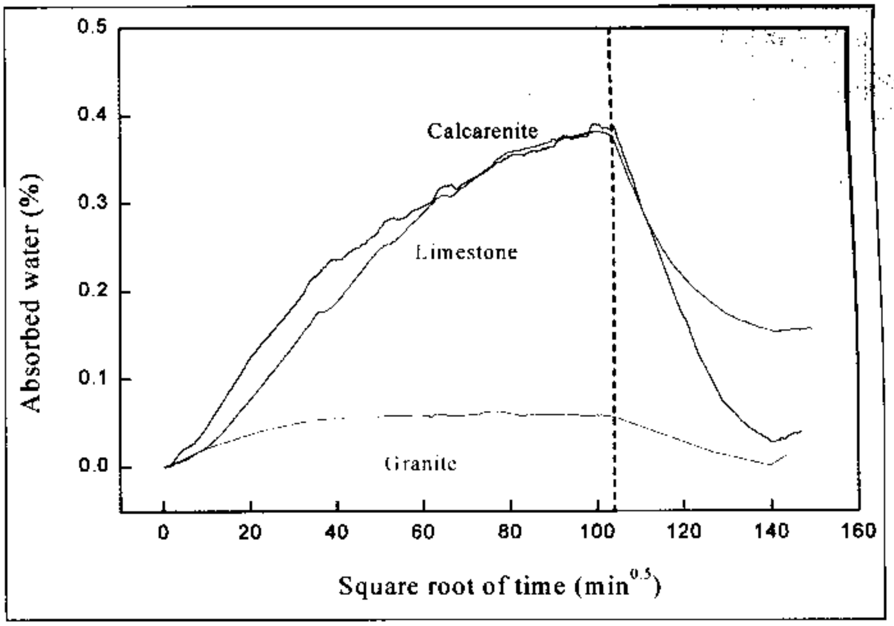
On this same point, the elimination of the water recirculation system suggests that alterations of the sample mass could occur because of the fall in the water level in contact with the specimen during the absorption phase.

To quantify this possible effect, we developed a control test using a nonporous material. The assay consisted of determining the alteration in the mass specimen when the water level falls, then gradually removing a certain volume of water from the specimen chamber. The linear effect observed permits the correction of the absorption profiles by means of a simple function ($y = 9.09 \times 10^{-4} + 0.025 \times r = 0.9993$).

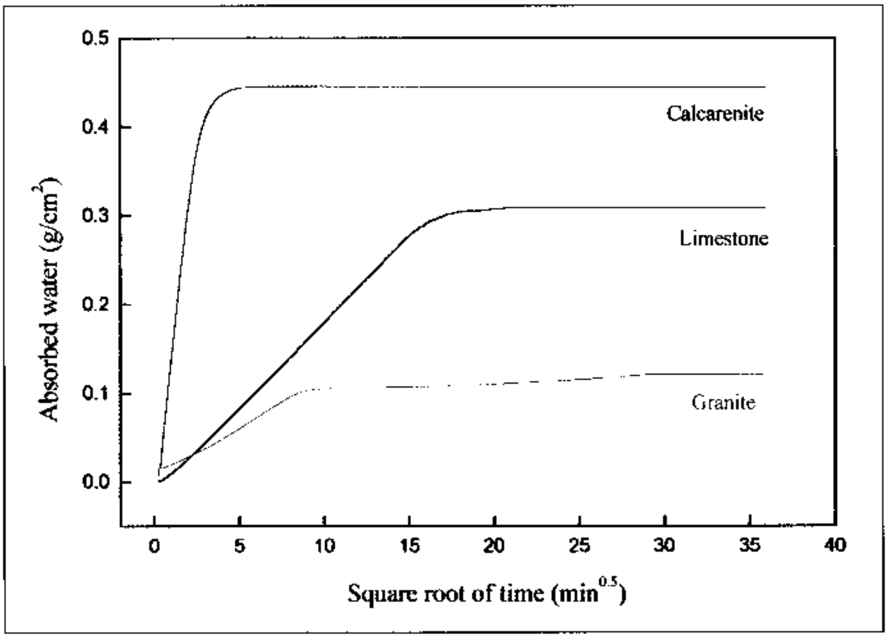
However, a calculation of the maximum amount of water absorbed by capillary action according to experimental data obtained (~3.5 g, corresponding to the most porous material) establishes a maximum water level fall of 0.05 mm in our specimen chamber (surface = 754 cm^2). The alterations in the absorption profiles due to this cause (<0.012 g) are insignificant compared to the increase of total mass, and therefore may be safely ignored.

Thus, to assure water level stability during the experiment without having to use a complicated water recirculation system, specimen chamber dimensions must not fall below a minimum.

The capillary absorption kinetics for the three selected materials can be fitted satisfactorily to a model dependent on the relevent square root ($r > 0.99$).



Water vapor absorption plots and the subsequent evaporation curves.

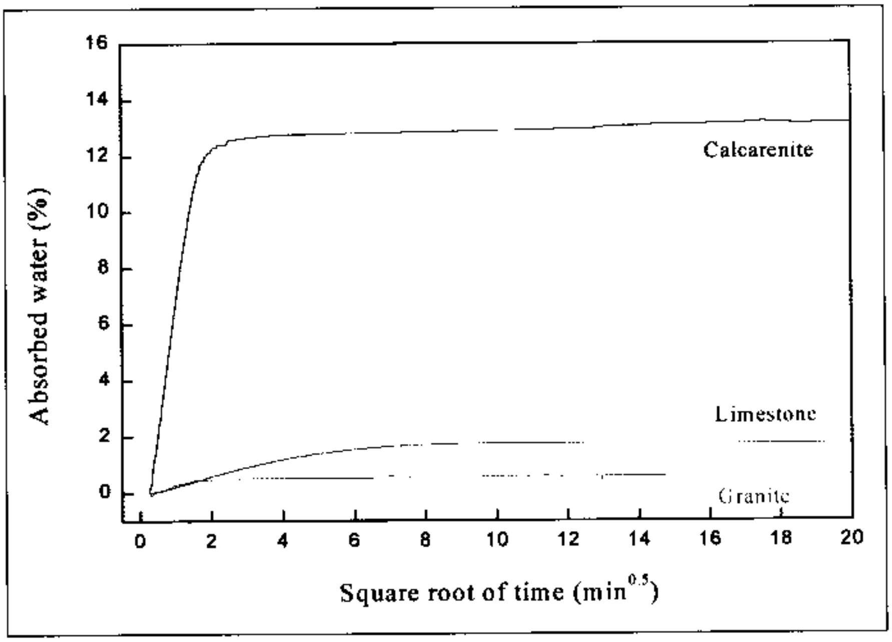


Water absorption profiles obtained by capillary action.

These results coincide with the theoretical model of capillary rise in which the gravity effect is discounted. Obviously, the height of our specimens (3 cm), and in general of any laboratory-scale specimen, permits the gravity effect on the absorption profiles to be discounted.

We need to emphasize that the exponential kinetic model suggested by Hoffman et al. (1990) is concerned exclusively with absorption profiles where the gravity effect is substantial, as in the examples proposed by these authors (walls of considerable heights).

Limestone shows the best correlation coefficients ($r > 0.999$) owing to its high homogeneity in pore distribution. This confirms the close correla-



Water absorption profiles obtained by immersion test.

tion between absorption profiles and pore distribution. Both of the other stone materials present lower correlation coefficients ($r > 0.99$), corresponding to a lower homogeneity in pore distribution. Specifically, if the low homogeneity in pore distribution of the calcarenite is accounted for, the coefficient obtained is surprisingly good.

Immersion Absorption Test

The absorption profiles obtained in the whole specimen tests show an initial phase with a high absorption rate during the first few minutes (<3 min for the calcarenite and granite specimens).

Despite the importance of this initial absorption phase, in which the percentage of total water absorbed is high (95% of the total for calcarenite), performing the test under the existing standard protocol makes data recording during these short periods impossible. As a result, interpretations of the kinetic for immersion found in the literature are not accurate in respect to this initial absorption.

Given these experimental results, it seems safe to conclude that the standard protocols do not permit a realistic kinetic interpretation of absorption by immersion. Using our methodology, the absorption rates in the initial stage can be fitted satisfactorily to a square-root-dependent kinetic.

Again, we observed a close correlation between absorption rate and pore radii distribution. Therefore, granite with a low porosity (2%) shows absorption rates close to limestone's (10% porosity), because of its larger pore size.

Advantages

Absorption tests on porous materials were performed using an automatic procedure characterized by both the continuous detection of the process evolution and by the automatic means controlling the entire system and the data obtained. Using this new protocol offers the following advantages over other procedures:

- Any absorption test can be performed without modifying the experimental protocol or equipment.
- The present procedure contains all the improvements of our earlier capillary absorption protocol based on continuous data recording and is applicable to the different types of absorption tests.
- Automatic data recording offers key advantages. A large number of data readings are collected. This frequency enables the study of absorption processes that display a rapid evolution.
- The development of completely automatic control for the water–specimen contact allows better standardization under the present protocol; this is critically important for capillary absorption tests, to avoid surface tension and flotation effects.
- In capillary absorption tests, the elimination of the water recirculation system used in our earlier procedure avoids surface vibrations in water in contact with the sample. A constant water level is attained simply by designing a specimen chamber with specific dimensions that minimize any alteration of water level.
- Designing a new specimen support system allows complete sample–water contact. Moreover, it allows good control of the horizontally of the specimen for the capillary absorption test.
- The continuous inspection of environmental conditions during the performance of hygroscopy and evaporation test is extremely important.
- The use of the present procedure permits absorption profiles to be fitted satisfactorily to a kinetic model dependent on the appropriate square root. ■

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