New Method for Performing **Capillary Absorption Test**

The measurement of capillary water absorption by porous building materials has been improved by a continuous data-recording method.

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Most deterioration of porous building materials used in construction is caused by interactions between the materials themselves and water—internal ice formation, intrapore crystal growth and pollutant attack. Among the different interaction mechanisms between the material and water, the capillary absorption process through the pore system is particularly important to material decay.

In recent years, capillary absorption has been the subject of several studies that provide some understanding of the processes that cause porous building material degradation. Most of the tests that have been used are based on a common procedure related to international specifications—RILEM (1980) and CNR-ICR (1986).

According to these specifications, a sample is dried until it has a constant mass; it then is brought in contact with a water surface. The mass increase caused by water absorption at specified time intervals is measured as a square-root function of time.

The amount of absorbed water is measured by interrupting the absorption process by extracting the sample, wiping it with a wet cloth to eliminate adhered water and determining its mass. This procedure can cause multiple errors by material loss in samples that have weak consistency, ineffective elimination of adhering water and excessive drying in unstable environmental conditions. Furthermore, if the test site location is a distance from the measuring scale, errors can be introduced by the different relative humidities and temperatures in the two environments.

Moreover, for the test performance, the sample must have certain controlled dimensions according to current standards, including a surface:volume ratio (S:V) of 1.2–2 cm^{-1}. This limitation does not affect common building materials. However, it might affect some archaeological and high-value-added materials.

The model suggested by the standard regulations presents two consecutive kinetic phase studies of the measuring process:

- An initial absorption stage, which is linear with the square root of the time, corresponding to the rise of the water until it reaches the upper part of the sample;
- An almost stationary stage related to the hydric saturation of the sample.

The capillary absorption coefficient, represented by the slope of the line obtained during the first stage, is the measurement most commonly used to characterize the process. The saturation time—the time required by the sample to reach its saturation phase—and the saturation water—the greatest amount of absorbed water—are measured.

The strict performance of the standard protocols eliminates a significant amount of data. This causes unreliable measurement of these parameters and does not permit characterization of small samples. Moreover, it prevents the development of more laborious kinetic models.

Comparison of Results Obtained from Capillary Absorption Methods

Sample	Method	Length (cm)	Porosity (%)	Water absorption coefficient ($g \cdot cm^{-2} \cdot s^{-1}$)	Saturation time (s)	Water absorbed ($g \cdot cm^{-2}$)
Cylinder, $S{:}V = 1.92\ cm^{-1}$	Standard	3.0	32.36	0.0074	11736	0.81
Cylinder, $S{:}V = 1.92\ cm^{-1}$	Proposed	3.0	32.06	0.0086	8496	0.80
Cylinder, $S{:}V = 2.5\ cm^{-1}$	Standard	1.6	32.10	0.0071	3852	0.44
Cylinder, $S{:}V = 2.5\ cm^{-1}$	Proposed	1.6	32.74	0.0089	2628	0.47
Tall cylinder, rapid saturation	Proposed	0.4	~32	0.0086	186	0.12
Porous cube, face a*	Proposed	3.8	19.00	0.0058	11425	0.41
Porous cube, face b*	Proposed	3.8	19.00	7.73×10^{-4}	39204	0.19

*Faces a and b are perpendicular to each other.

The new protocol proposed in this paper can be used for every sample. It provides an easy solution to all the inconveniences previously mentioned. It also provides a greater quantity of and more-accurate data sets.

The new protocol continues to be based on the amount of absorbed water as a function of the elapsed time. However, the test sample mass is measured without disturbing the system geometry, because contact between the water and the sample is never interrupted.

The New System

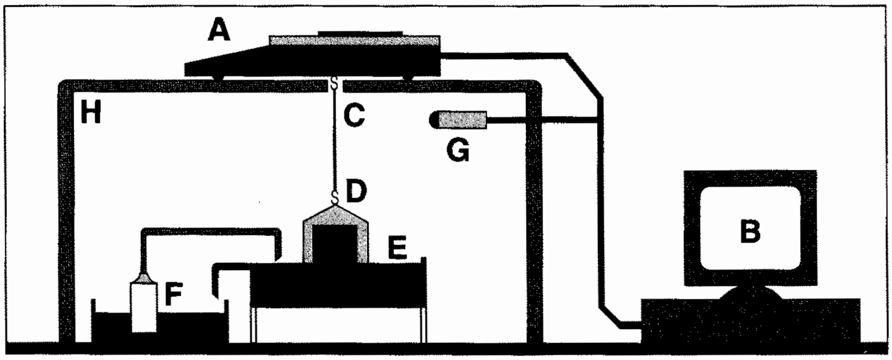
The new system is composed of four parts:

- Computerized Weighing System: A scale with the capability for weighing a sample by hanging was interfaced with a computer. The system accuracy must be better than 1/256 of the maximum sample weight. A scale with a serial output data interface (Model 300C, Precisa, PAG Oerlikon AG, Zurich, Switzerland) was used in this study. The sample was hung from the scale and put in contact with a water surface. The sample surface in contact with the water was kept, at all times, in a horizontal position by a specially designed inelastic support.
- Water System and Level Control: It is important to ensure rigorous stability of the water surface level to minimize deviations derived from floating effects and surface tension. Stability was implemented with a stationary recirculating water system that included a contact container where the sample–water interaction was accomplished and a water supply container equipped with a 50 cm^3/s high-stability electrical pump to provide an adjustable water volume to the contact container. The contact container has a V-shaped drain that maintains a stable water level, even when there are small changes in the water volume supplied by the pump. Under these conditions and in the absence of external alteration factors, equal water volumes produce equal water levels. Moreover, when a closed circuit system is used, the water can be used to control chemical and physical variables, such as pH, conductivity and turbidity. The quantification of these parameters makes it possible to relate them with sample alterations, such as dissolution, reprecipitation and pollution.
- Computer System: A computer system was used as data register. The software for this protocol—written on QB45—allowed data to be collected on predetermined time sequences, in accordance with the type of sample under study. Moreover, the program could record data sets of temperature, relative humidity, pH and conductivity. The input data were automatically and continuously stored in a computer file, and it is available for later graphical and mathematical processing.
- Environmental Parameters Stabilization System: The stabilization of some environmental parameters, e.g., temperature and relative humidity, is important to absorption measurement. Environmental stabilization was attained easily by locating the measuring system in a small, thermally isolated receptacle.

The experimental procedure is established in accordance with the following steps:

- The sample is dried until it exhibits a constant mass;
- The sample is hung from the scale in preparation of contact with the water surface;
- Contact between the sample and the water surface is accomplished by a slow increase in the water level, caused by electrical regulation of the pump-supplied water flow;
- Absorption and data recording begin when contact between sample and water surface is attained.

Process control can be improved by on-line plotting of absorbed water vs elapsed time. After the acquired data have been collected, they can be used to obtain the characteristic parameters of the test samples.



General diagram of the new system for the study of absorption capillary process on porous materials ((A) scale, (B) computer system, (C) hanging scale system, (D) sample and support system, (E) water contact container, (F) water supply container, (G) environmental sensor and (H) thermal isolation receptacle).

Obtained Results

Several tests have been performed with ceramic and stone materials. A few cases are presented here to provide an example of the possibilities of the new technique.

All test samples initially were dried until constant mass was attained. In all cases, the drying time was longer than the saturation time. The tests were performed at 20°C and 70% relative humidity.

Three types of tests were made:

- Comparison between the new method and the standard performance test for samples that meet the dimension specifications of standard regulations;
- Comparison between the new method and the standard performance test for samples that do not meet the dimension specifications of standard regulations;
- Application of the new method to samples that cannot be measured by standard regulations.

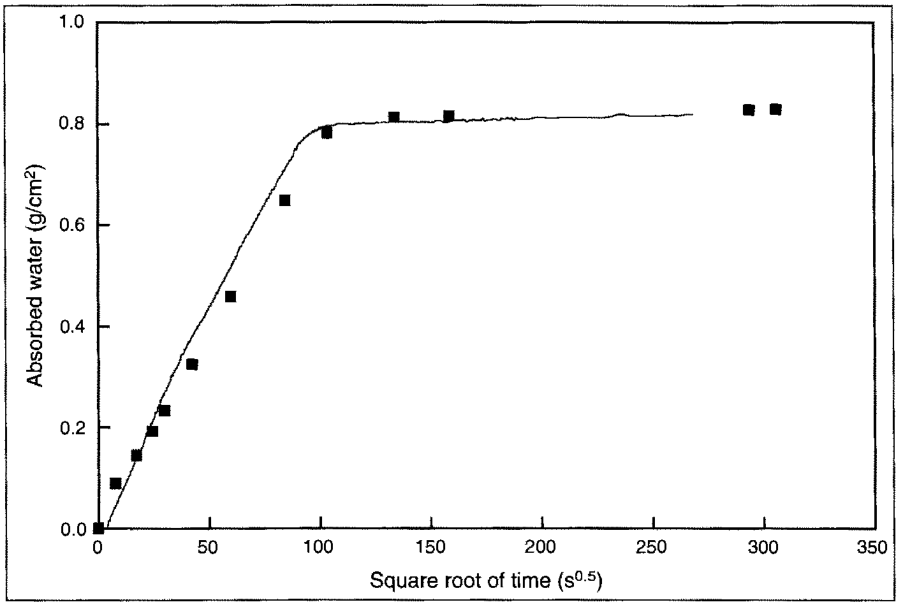
Ceramic test samples were made in the researchers' laboratory, using a commercial clay. This ceramic body usually delivers a high degree of homogeneity, which implies that the porosity of the sample was uniform and isotropic.

The particle size of this ceramic was measured (Model 2600C Laser Sizer, Malvern Instruments, U.K.). The results show a stable tetramodal distribution of particle size with bands centered at 6, 12, 22 and 40 μm. The porosity accessible to water test was based on RILEM (1981) procedures that confirmed sample homogeneity of pore-size distribution.

The samples were cylinder-shaped with a cross-sectional area of 8.04 cm^2. The cylinder base, which was in contact with the water, was shaped slightly convex to prevent the accumulation of bubbles at the sample–water interface. After it was molded, the sample was dried and baked in an electric oven at 850°C.

Methodology Comparison

The ceramic cylinder samples used here had a length of 3 cm and a $S{:}V$ ratio of 1.92 cm^{-1}. These ceramic samples were tested in accordance with the two methods described in this article. The total test time was ~24 h with a 3-h saturation time.



Mass evolution during absorption process as a function of the square root of the time, for a ceramic sample with a S:V ratio of 1.9 cm^{-1}, in the shape of a cylinder with a base area of 8.04 cm^2 and a height of 3 cm ((—) proposed method and (■) standard method).

Mass was measured 12 times during the absorption phase using standard methodology. These data allow the absorption and saturation phases to be easily defined.

The data collected during the first minutes can be more critical because of the complex protocol of extracting, drying, transporting to the scale, weighting and returning the sample to water contact.

On the other hand, the new method collects data every 2 s; therefore, it is able to take >5400 measurements during the absorption phase. The better definition of the new method permits a better fit for the absorption and saturation phases, as well as for the associated parameters. Furthermore, the transition from the absorption phase to the saturation phase can be accurately defined.

The study of microevolutions in the behavior of the system, emphasizing those present at the beginning of the experiment and caused by superficial tension and floating effects, also was possible.

The new method provided an absorption coefficient of 0.0086 g·cm^{-2}·s^{-1}, 16% greater than that obtained using the standard method. The water saturation value was 0.80 g, a 1.2% variation with the standard method. The lower value obtained for the absorption coefficient using the standard method is explained by erroneous drying of the sample before it was weighed.

Comparison was made between the new and standard methods for samples that do not meet dimensional requirements. This test is directed to those samples for which $S{:}V$ > 2 cm^{-1}; therefore, the saturation time is short. This case is often observed in archaeological pieces, in which dimensions tend to be limited by material availability.

The tested samples were clay cylinders baked in an electric oven, with a height of 1.6 cm ($S{:}V$ = 2.5 cm^{-1}). The total test time and the number of recorded data were similar to the previous case, but the saturation phase was attained in ~1 h.

The measured capillary absorption coefficient (0.0089 g·cm^{-2}·s^{-1}) and saturation water (0.47 g) by the new method were, respectively, 25% and 7% greater than standard method values. Both differences probably were caused by a greater proportional loss of water by defective drying caused by the smaller sample sizes. The new method provides better plots and greater accuracy in the computation.

Special Samples

An absorption capillary test was accomplished on two different materials in which the use of the standard method was not possible.

A 4-mm-high cylindrical ceramic sample with S:V = 6.3 cm^{-1} was tested. The saturation phase was achieved at 3.1 min, and, in this short time, the new system measured 93 data sets, totalling 900 data points. The data were satisfactory fitted to the suggested kinetic model with an absorption coefficient of 0.0086 g·cm^{-2}·s^{-1}, similar to the other cases, and a water saturation value of 0.12 g.

Stone material samples from a quarry in the vicinity of Cádiz, Spain, were measured. This stone is the most commonly used for local construction. It is a sedimentary limestone made of a weak conglomerate of broken shell, with 19% porosity and a high anisotropy in the pore distribution. The fragile nature of this stone is such that the application of the standard method was impossible, because material losses during manipulation were greater than the increase of water absorbed.

However, the new test was performed easily on two perpendicular faces of a 3.8-cm cube. Two different absorption profiles were observed, according to the preferential direction of the porous system face, with 57 × 10^{-4} and 7.73 × 10^{-4} g·cm^{-2}·s^{-1} values for capillary absorption coefficient.

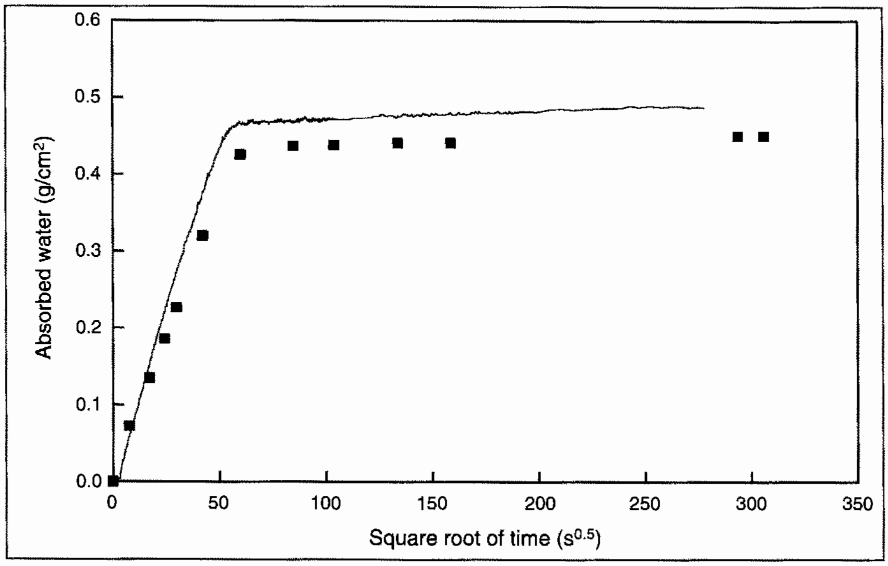
The measured correlation coefficients (0.9822 and 0.9854, respectively) for these tests were slightly lower than in previous cases. This, as well as the heterogeneity of pore size, permits assuming that the existence of various profiles is a function of the prevailing pore size. In this way, two or more slopes can be deduced for the absorption phase corresponding to different absorption speed, and a more-elaborate kinetic model may be considered for these heterogeneous materials. This research is the next project.

Conclusions

The study of the capillary water absorption process in porous materials presents greater reliability if a continuous data recording method is used.

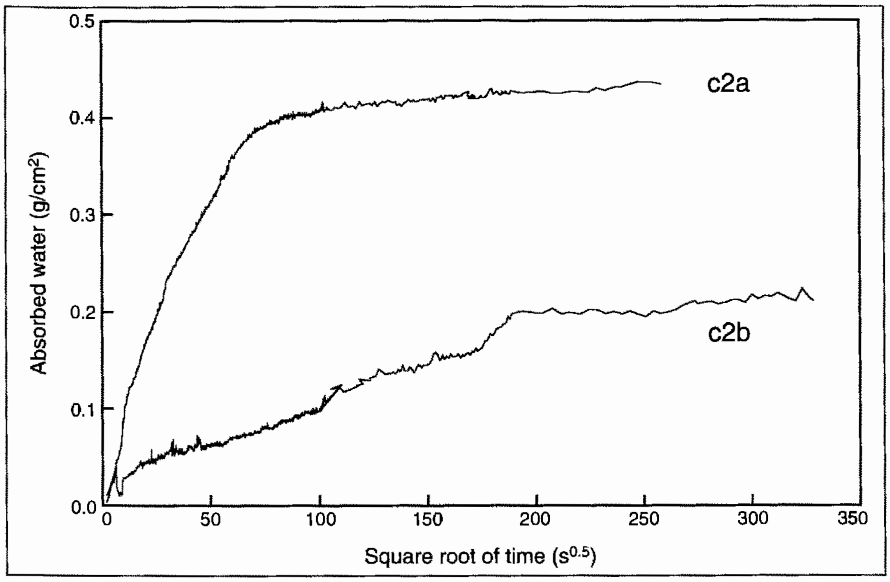
The newly designed system has the following advantages over the standard method:

- It is possible to record data online during measurement. Therefore, errors generated by the interruption of the sample–water contact are minimized. Elimination of sample transportation to the scale site results in a more coherent sample.
- Continuous control of the absorption process permits study of small samples. Continuous mass



Mass evolution during absorption process as a function of the square root of the time, for a ceramic sample with a S:V ratio of 2.5 cm^{-1}, in the shape of a cylinder with a base area of 8.04 cm^2 and a height of 1.6 cm ((—) proposed method and (■) standard method).

Mass evolution during absorption process as a function of the square root of the time, for a ceramic sample with a S:V ratio of 6.3 cm^{-1}, in the shape of a cylinder with a base area of 8.04 cm^2 and a height of 0.4 cm.



Mass evolution during absorption process as a function of the square root of the time for a sedimentary stone sample. Profiles correspond to two perpendicular faces of a 3.8-cm cube.

recording allows measurement of absorption capillary in materials with high absorption speeds not possible when using the standard method.

- Continuous recording of sample mass evolution eases the detection of temporary microevolutions that, in the standard method, commonly are considered as errors.
- The existence of a stabilized water circuit supply/drain in the contact container permits control of the water level, minimizing weight variations associated with floating effects or surface tension. In the standard method, the water absorbed by the samples is not returned continuously to the contact container, and, when several samples are being measured simultaneously, it is not possible to assure a constant water level.
- The new method allows the collection of much data, making it possible to fit them to different theoretical models. The lack of data when using the standard method permits only a linear fit for the amount of water absorbed vs the square root of the elapsed time. ■

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