



The Old Italian Technical Normative NORMAL 07-81 Can Be Improved? A Possible Accurate Measurement of Water Absorption by Stone

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Abstract

The measurement of water absorption by full immersion (WA) can be considered an inexpensive alternative to N₂ physisorption (BET), helium pycnometry, mercury intrusion porosity (MIP), because allows to evaluated the open porosity that can be entered by water. More, for stones of interest in the field of Cultural Heritage such measure results, may be, more suitable because water accessibility is the real direct and/or indirect source of their degradation. The WA is ruled by a very old Normative [1] that, in our opinion, foresees a handling of sample letting to a scarcely accurate measure; so, we here propose a procedure that don't need sample handling.

Introduction

The measure of WA or capillary absorption in stones are imperative in all the test aiming to evaluate the efficiency of hydrophobic coatings and consolidants as well as in artificial weathering tests. Both the measures are ruled by Normative on Cultural Heritage (CH) and both the documents are enough old so needing an improvement. In particular the NORMAL07/81 [1] foresees to weight a dried sample of regular shape (with a ratio surface/volume ranging from 2 to 1.2 cm²), to dip it in water and weight at different times after a packing on a humid cloth; then the sample must be dried again and all obtained data used to obtain the absorption capability (A%) and the imbibitions coefficient (IC%). Such an handling of the sample surely lets to errors that we propose to eliminate using a hydrostatic balance, i.e. we propose a typical “Colombo's egg”. The present research is a part of a wider research aiming to propose procedures for evaluating the state of conservation of CH artefacts and risk related to their porosity. Taking into account that stones are very heterogeneous materials, we also aim to propose smaller dimension of the sample to be analysed in order to decrease the invasiveness of the technique. We are testing different stones and, for each one, different dimensions; the use of other characterising analytical techniques and other tests are foreseen in order to obtain a database that, we hope, will allow to obtain, through a chemometric data treatment, a risk threshold for different stones bound to the presence of water inside them. In a preliminary research, already submitted for publication [2], we presented encouraging data obtained on three different marble samples by the proposed procedure while here we present data obtained on different size travertine specimens and a comparison with the NORMAL procedure.

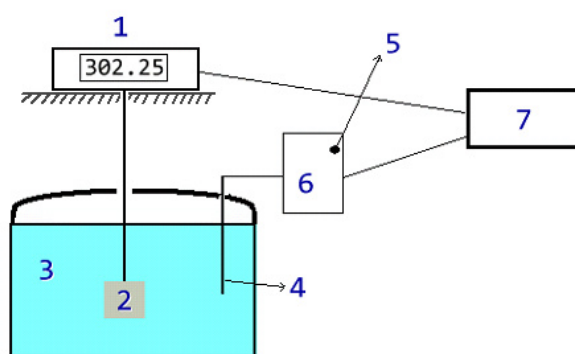


Fig. 1; Experimental apparatus. 1: balance, 2: specimen, 3: distilled water, 4 and 5: temperature sensors; 6: datalogger, 7: PC

Materials & Methods

12 cubic samples of travertine, 3 for each of 4 different edge (2, 3, 4, 5 cm) were furnished by Davide Sambuco Marble Laboratory (Roma, Italy). A Mettler mod. 502b (resolution 0.01 g, load 510 g) balance was used with a handmade hydrostatic apparatus (see scheme in fig. 1).

The weight increase of the dipped specimen was continuously recorded for 3000 min, at 1 min intervals, through a PC, equipped with Datalyse software, The temperature of both air and water was also monitored in order to obtain correct values of their density.

Results

A typical curve is shown in fig 2 together with the one obtained by the NORMAL procedure on the same specimen (a 5x5x5 cm cube). Even if fig. 2 reports the mass variation (in order to compare our data with NORMAL ones) our procedure allows to directly determinate the volume variation because we measure the Buoyancy force. Bulk and apparent density as well as pore volume that can be

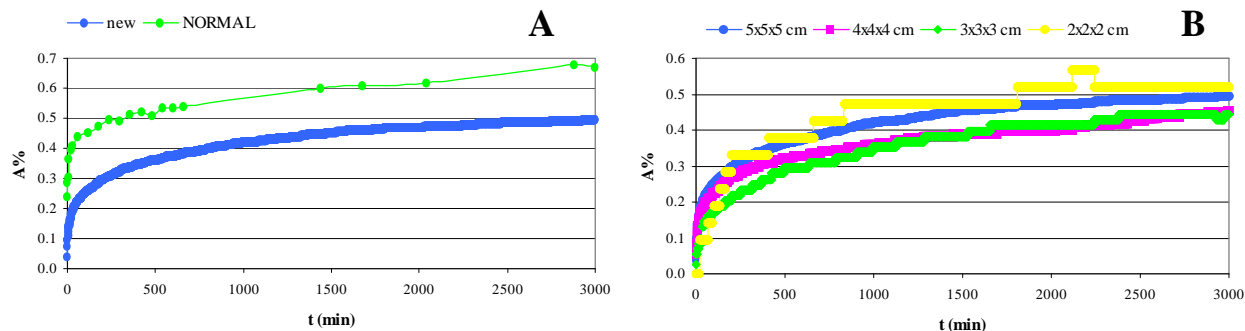


Fig. 2; Comparison of WA curves: **A**) by our proposed method and the one from NORMAL 07/81 on a 5x5x5 cm travertine cube; **B**) for travertine cubes of different size

entered by water can be also estimated; an extrapolation of the curves surely let to more accurate data because our experimental initial value is affected by an error due to the moving of the specimen while to obtain the final one a too long time is required. Figure 2A shows a significantly lower curve for the proposed method. We cannot say what of the curves is more accurate because, as above said we have a sure “in defect error” on our first experimental value but it is highly probable that both the initial and final experimental values, obtained by the NORMAL method, are affected by an “in excess” error due to the water not adsorbed but only remaining inside the external cavities (typical of travertine). Surely no one of the two values is accurate as we chose to stop the measures at 3000 minutes and for both the saturation was not reached. Figure 2B shows a no significant trend as a function of the cube’s size. Values obtained to draw the four curves, really, have a standard deviation similar to those obtained using three different cubes of the same size; this was expected taking into account that the number and size of the surface’s cavities greatly differ in both the same and different cubes; as a fact the differences between the geometric volume (measured with a 0.05 mm resolution) and the initial estimated volume significantly varies. From figure 2B it can be seen the effect of the balance resolution, obviously the curve is less detailed when the size decreases. When all data of the 4 dimensions and of the 3 repetitions are obtained probably a simple Anova can be useful to study the trends. In preliminary tests [2] performed on three different marble’s cubes, 2x2x2 cm, using an analytical balance (0.01 mg), we obtained well detailed curves; in the present research we are only evaluating the effect of the error that, in our opinion, affects the NORMAL method as a consequence of the sample’s handling and this requires an accurate statistical approach.

Conclusions

Both NORMAL and our method require long lasting measures and we must collect lot of data in order to have significant information by chemometry; anyway results obtained from our previous tests [2] as well from those here presented are encouraging and surely stimulating for chemometricians.

References

- 1) Normal 07-81, Assorbimento d’acqua per immersione totale capacità di imbibizione, 1981
- 2) S.H. Plattner, R. Reale, G. Visco, M.G. Papa and M.P. Sammartino, Proposal of a new analytical procedure for the measurement of water absorption by stone. Preliminary study for an alternative to the Italian technical normative NORMAL 07-81, *Chem. Cent. J.*, submitted.