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A round robin campaign on the hygric properties of porous building materials

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Abstract. The reliable determination of the hygric properties of porous building materials is important. In earlier round robin campaigns large discrepancies of measured hygric properties were found among different labs. Later studies indicated that differences in lab conditions and more importantly, personnel’s operation procedures and data processing methods, might have the greatest impact. To gain further insight, a new round robin campaign has been launched by KU Leuven (Belgium), to which another eight institutes contributed. A relatively stable and homogeneous ceramic brick is tested, and 3 standard tests are performed: the vacuum saturation test, the capillary absorption test and the cup test. During the campaign, two rounds of measurements are performed. In the 1\textsuperscript{st} round, tests are performed according to participants’ respective experimental protocols. Next, a strict and detailed common protocol is prescribed. This paper reports on the results obtained in the 1\textsuperscript{st} round of measurements. Results show that not much progress has been made since the EC HAMSTAD project: the vacuum saturation test leads to the most consistent results, while the cup test produces the largest discrepancies, most probably originating from sample sealing and humidity control.

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1 Background

It is widely accepted that the reliability of the hygrothermal analysis of buildings and built structures depends largely on the accuracy of the input material data. It is therefore important to determine the hygric properties of porous building materials accurately. In the past decades, many round robin campaigns for the measurement of the hygric properties of porous building materials have been performed [1-7]. One of the more famous is the EC HAMSTAD project [1], wherein KU Leuven from Belgium coordinated the overall campaign and another five participants – CTU Prague from Czech Republic, TNO Building and Construction Research from the Netherlands, Technical University of Dresden from Germany, The University of Edinburgh from United Kingdom and National Research Council from Canada – measured a variety of hygric properties of 3 representative porous building materials: autoclave aerated concrete, calcium silicate and ceramic brick. Results showed that the different labs did not always obtain similar results, especially for the vapor resistance factor (μ) from the cup test. Other round robin campaigns also showed obvious discrepancies between different labs, whatever the material or the test was.

To have a better understanding and control of potential error sources, in-depth studies have been conducted. The repeatability and reproducibility analysis proves that neither the material inhomogeneity nor the experimental errors rooted in the methodologies themselves play a significant role in causing the conspicuous inter-laboratory discrepancies [8]. Further studies on the impact of time and personnel in the same lab also show that they do not cause large errors if the protocol remains unchanged [9]. Consequently, it is the experimental procedure, condition control and data processing that may have the greatest impact.

Now that 15 years have passed since the completion of the EC HAMSTAD project, it is interesting to identify potential improvements of these inter-laboratory discrepancies. For this purpose, a new round robin campaign has been initiated by KU Leuven, with another 8 institutes contributing to it. In the following sections, we will first introduce this round robin campaign briefly. Next, experimental results from different labs will be presented and discussed. Finally, some general conclusions will be drawn.

2 Campaign in brief

This campaign proposes a small-scale round robin test on selected hygric properties, trying to quantify the consistency of the experimental results from different labs and to identify the sources of discrepancies. It was planned in the end of 2017 and formally started early 2018. In total 9 institutes are involved: KU Leuven (Belgium, the coordinator), University of Porto (Portugal), China Academy of Building Research (P.R. China), Łódź University of Technology (Poland), Technical University of Dresden (Germany), Technical University of Denmark (Denmark), The University of Edinburgh (United Kingdom), Lund University (Sweden) and Czech Technical University in Prague (Czech Republic). Since there is no funding for this campaign, all participants are voluntarily involved and financially unsupported.

Because of this limited budget, only one target material was chosen in this campaign: the Robusta ceramic brick [10]. It is relatively homogeneous and stable, expected to remain unchanged during the whole test period. Raw bricks from the same batch were chosen randomly and distributed by KU Leuven to all other participants, to minimize potential material inhomogeneity.

During the round robin campaign, the vacuum saturation test is performed for obtaining the bulk density and the open porosity, the capillary absorption test is carried out for
measuring the capillary absorption coefficient and the capillary moisture content, while the cup test is executed for determining the vapor resistance factor. The overall campaign has two rounds of measurements: in the 1st round, all participants perform these tests based on their respective experimental protocols used in their own labs; in the 2nd round, a strict and detailed experimental protocol is prescribed and tests are performed again. Since the 2nd round tests are still ongoing, we only introduce the 1st round results in this paper. In a follow-up publication, more detailed and complete results will be reported and analyzed.

3 Results and discussion

In this section, we will present and analyze the results obtained from different tests according to their respective routine protocols. Note that not all participants have finished all 3 tests for the 5 properties, and the lab numbers in the following analysis are not the same as the affiliation numbers for co-authors. The results are represented both graphically and numerically, the latter with the relative standard deviation (RSD) calculated by Eq.(1).

$$RSD = \frac{1}{\bar{x}} \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x}_i)^2}{n-1}}$$

where $x_i$ is the average value of a given property obtained by Lab $i$, $\bar{x}_i$ the average of $x_i$, and $n$ the total number of labs.

3.1 Vacuum saturation test

The bulk density ($\rho_{\text{bulk}}$, kg·m$^{-3}$) and the open porosity ($\phi$) obtained from the vacuum saturation test are illustrated in Fig.1. For $\rho_{\text{bulk}}$ the results obtained from different labs are very close, with a relative standard deviation of 1.3% in our case and 1.0% in the EC HAMSTAD project, both negligible. For $\phi$, the relative standard deviation is 5.0%, slightly larger than that in the EC HAMSTAD project (2.0%). However, Lab 2 reports a noticeably low value, while other labs remain close. After a thorough check, the root has been located in the experimental procedure: while all other labs first evacuated the air in the vacuum container and then filled in water, Lab 2 operated in the opposite order. Consequently, some air was retained in the sample, underestimating the open porosity. By excluding the results from Lab 2, we can reduce the relative standard deviation to 2.2% for $\phi$, roughly the same as in the EC HAMSTAD project. In short, there is not much to worry about the vacuum saturation test if the procedure is correct.

![Fig. 1. Results of the vacuum saturation test.](https://doi.org/10.1051/matecconf/201928202011)
3.2 Capillary absorption test

The capillary absorption coefficient \( A_{\text{cap}} \) and the capillary moisture content \( w_{\text{cap}} \) obtained from the capillary absorption test are illustrated in Fig. 2. For \( w_{\text{cap}} \) the results obtained from different labs are similar, with a relative standard deviation of 7.7\%, almost the same as in the EC HAMSTAD project (8.3\%). For \( A_{\text{cap}} \) the relative standard deviation for all labs is 14.0\%, slightly smaller than the 19.6\% in the EC HAMSTAD project. It is obvious that the \( A_{\text{cap}} \) value of Lab 7 is much lower.

A potential issue is that an automatic capillary absorption setup was used by Lab 7, while all other labs performed this test manually. However, previous study proves that automatic and manual methods should give similar results [11]. It is therefore highly possible that there were operational mistakes or instrumental malfunctions for this automatic setup, calling for correction in the 2\textsuperscript{nd} round measurements. If the abnormal result from Lab 7 is excluded, the relative standard deviation for remaining labs can be further reduced to 9.4\%.

Based on the relative standard deviation, it seems that much progress has been made with respect to measuring \( A_{\text{cap}} \). However, it should be kept in mind that this deviation also includes the material inhomogeneity error, and we are using a much more homogeneous ceramic brick in this round robin campaign than in the EC HAMSTAD project. If this material error is excluded, the EC HAMSTAD project gives an inter-lab error of 14.4\% for \( A_{\text{cap}} \) [8], which is not much worse than our current result. Consequently, we cannot observe significant improvement in performing the capillary absorption test for deriving \( A_{\text{cap}} \).

![Fig. 2. Results of the capillary absorption test.](image)

3.3 Cup test

The vapor resistance factors (\( \mu \)) obtained from the cup test are illustrated in Fig. 3. As is clearly revealed, the smallest value goes down to 6.0 (Lab 6) while the largest value reaches 25.8 (Lab 7), producing a factor 4.3 difference. However, the ceramic brick used in this campaign is rather weakly hygroscopic, thus the resistance factor at different relative humidities (within the hygroscopic range) should not vary that much. If we neglect the impact of relative humidity and bring all results together, a relative standard deviation of 44.2\% can be obtained, slightly smaller than that in the EC HAMSTAD project (~60\%).

There are a number of factors that can possibly impact the cup test, but not all of them are essential. All measurements were carried out under normal atmospheric pressure in different labs, so it is difficult to imagine the air pressure to cause such a large discrepancy.
The resistance factor is weakly dependent on temperature [12], and all the labs performed the test in-between 20-23 °C. Consequently, the impact of temperature can also be excluded. The sample area has already been proved to have a limited impact, even not corrected for the masked edge [4, 5]. The sample thickness in different labs varied between 1.7 and 5 cm, and this impact should be evaluated together with the correction for surface resistance. The ceramic brick is, however, relatively vapor impermeable. So the sample thickness and surface resistance should have limited influence [2, 4, 5, 13].

Besides the above-mentioned factors, there are another two factors that have drawn many researchers’ attention: sample sealing [2, 9, 13] and humidity control [2, 3]. For sample sealing, the coating of the sample’s lateral side may lead to a penetration of sealant into the sample, reducing its real cross-sectional area and finally leads to an over-estimation of the resistance factor. On the contrary, improper sealing between the sample and the diffusion cup may lead to vapor leakage, and resultantly cause an under-estimation. This is especially serious when the material is relatively impermeable. For humidity control, desiccant and saturated salt solutions are often used. If the desiccant is not completely dry or if the salt solution is not saturated, its real relative humidity will be underestimated, and both under-estimation and over-estimation of the vapor pressure gradient across the sample can occur, leading to deviating results.

The vapor resistance factor is one of the most important hygric properties, especially in the hygroscopic range. It is therefore of utmost significance to determine it accurately. In the 2nd round measurements of this round robin campaign, special attention will be paid to it for improving the agreement among different labs.

![Fig. 3. Results of the cup test.](image-url)

**4 Conclusions**

A new round robin campaign aimed at comparing the measurements of the hygric properties of porous building materials in different labs is launched by KU Leuven and participated by another 8 institutes. A relatively stable and homogeneous ceramic brick is tested, and the vacuum saturation test, the capillary absorption test and the cup test are performed. In this campaign, two rounds of measurements are executed: in the 1st round, all participants perform these tests based on their respective experimental protocols used in their own labs; in the 2nd round, a strict and detailed experimental protocol is prescribed and
tests are performed again. This paper reports on the results obtained from the 1\textsuperscript{st} round measurements. Results show that the vacuum saturation test leads to consistent results among different labs, and the differences of the capillary absorption test results are also limited, so long as the experimental procedures are without problems. The cup test produces significant discrepancies, most likely originated from sample sealing and humidity control. When compared with the EC HAMSTAD project, however, not much progress has been made for all these 3 tests. In the following 2\textsuperscript{nd} round measurements, we will try to identify potential improvements with a common protocol.

References