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Interlaboratory Comparison of Hygric Properties of Porous Building Materials

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ABSTRACT: The precision of methods used for the determination of hygric properties of porous building materials was investigated. The study was performed in the framework of the EU-initiated HAMSTAD-project. Six laboratories measured the selected hygric properties of three porous building materials. While the most measured properties show acceptable agreement, yet, it was found that some of the existing standards or commonly accepted measurement methods need improvement. Most striking were large variations in the results of the vapour transmission tests performed in accordance to the existing European Standard.

KEY WORDS: hygric properties, interlaboratory comparison, round robin, vapour diffusion test, free water uptake experiment

INTRODUCTION

SINCE THE INTRODUCTION of the interstitial condensation calculations [4], the prediction of combined heat and moisture transport has been continually improving and many transient, numerical models for the hygrothermal analysis of building envelopes appeared. Significant progress has been made in the framework of the IEA Annex 24-project [7]. The Annex project focused on model development and comparison, material properties, boundary conditions and the impact of combined heat, air and moisture (HAM) transport on energy consumption and durability. Yet, attaining uniformity in the techniques used for measuring material properties, which serve as input for the HAM-models, has always remained difficult. Although a lot of databases exist, the reliability of the measurements has seldom been investigated. In 1988, BCR initiated a project on water permeation in building materials [1]. The vapour permeability of two materials, extruded polystyrene and particle board, was measured by 13 European laboratories. Despite exclusions of outliners, the range of variation in the measured water vapour permeability remained large. SBI [11] reports a round robin testing of retention curves measured with pressure plates. Other examples are rare.

In the framework of the EU-initiated HAMSTAD-project, an interlaboratory comparison of the measurement of the basic hygric properties of porous building materials was performed. In total six laboratories were involved. They all measured both the moisture capacitive and moisture transport properties of three building materials: calcium silicate plate, fired clay brick and cellular concrete.

MATERIALS INVESTIGATED

The interlaboratory comparison was performed on three building materials: calcium silicate plate, ceramic brick and cellular concrete.

Those materials were chosen because of the differences in pore structure, homogeneity and complexity of the pore structure.

Calcium silicate plate is a low-density board, mainly used as capillary active interior insulation used in thermal retrofitting of buildings. It has a high capillary absorption coefficient and capillary moisture content. It is largely composed of synthetic mineral xonotlite, a complex calcium silicate hydrate [6] that in addition to the randomly orientated cellulose fibres is clearly visible on SEM-images (see Figure 1). Calcium silicate has a fine pore structure, comprising the voids between matted acicular crystals (Figure 1). The material can be identified as homogeneous enough to simplify comparing the results.

The SEM-image in Figure 2 shows the typical pore structure of ceramic brick. Compared to the calcium silicate plate, the brick has a tubular pore structure. The total open porosity of the material is much lower. Due to the nature of the material and its production process, one can expect a higher variability in the pore structure than that of calcium silicate and thereby also higher variability in the material properties.

Most typical for the last material, aerated autoclaved concrete (AAC) also known as cellular concrete, is the absence of large artificial air pores created during the production process has large air voids. The obtained pore structure of AAC is illustrated in Figure 3. The spherical air pores are clearly visible. The walls of the air pores consist of grains of sand embedded in a cement–lime matrix. This matrix provides the cohesion of the material and consists of fine plate-shaped crystals in a jumble (Figure 3).

The last material is chosen because it shows a rather complex pore structure. To avoid differences due to the production process, for all three materials specimens of the same batch were sent to all participating laboratories.

BASIC HYGRIC CHARACTERISATION

The minimum characterisation of the materials consisted of the determination of the bulk and matrix density, total open porosity, capillary moisture content and water absorption coefficient. The first three properties are obtained from a vacuum saturation test, the last two demand to perform a time-controlled capillary water uptake experiment.

Vacuum Saturation Test

The total open porosity Ψ_o (–) of a porous material is defined as the ratio of the volume of the open pores to the total volume of the sample. The bulk density ρ (kg/m^3) is defined as the ratio of the dry mass of the sample to its volume, while the matrix density ρ_{mat} (kg/m^3) is defined as the ratio of the

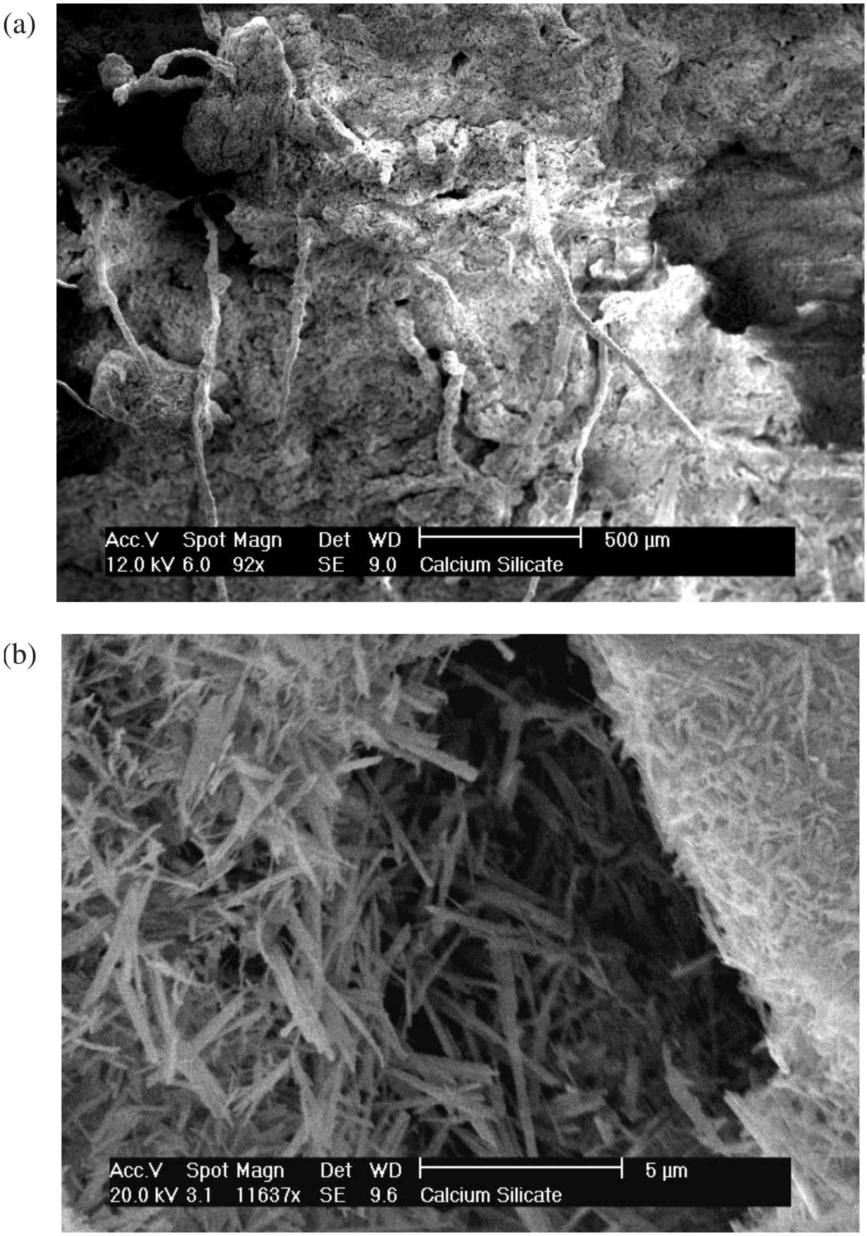


Figure 1. *Microstructure of calcium silicate plate: (a) low magnification, illustrating the randomly orientated cellulose fibres; (b) higher magnification, showing the acicular form of the mats of the xonotlite particles. (SEM-images by [6]).*

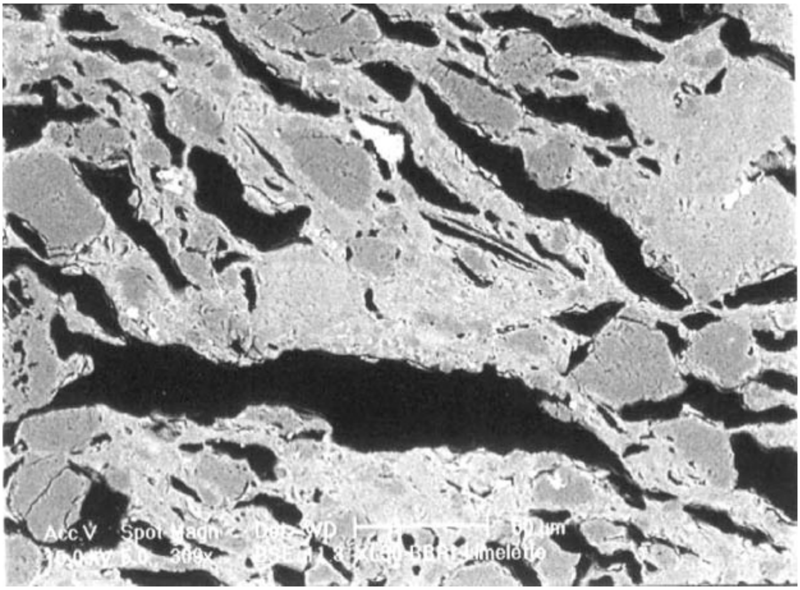


Figure 2. SEM-image of a polished epoxy impregnated ceramic brick sample. The pores appear in black. The baseline measures 360 μm. (Image from [2]).

dry mass to the volume of the solid matrix, including closed pores. All data are obtained from a vacuum saturation test. A precise prescription of the test as well as of the followed sample preparation can be found in [10].

Figure 4 compares the cumulative distribution function for measurements of the total open porosity on the three materials, while Table 1 summarises all results of the vacuum saturation tests. The porosity values measured by Laboratory 5 are slightly lower than the values reported by the other laboratories. This suggests that no complete filling of the open porosity by water is achieved at Laboratory 5. Overall, for all three materials, the vacuum saturation experiment appears to be highly reproducible. The largest deviations occur for cellular concrete. This can be attributed to the typical pore structure of AAC in which removal of air is more difficult. Furthermore, for a number of AAC samples, after vacuum saturation a slight increase in the dimensions was found. The swelling of the material might also account for some of the discrepancies between the results of the different laboratories.

Free Water Uptake Experiment

The capillary moisture content $w_{\mathrm{cap}}\,(\mathrm{kg/m^3})$ and the water absorption coefficient A $(\mathrm{kg/m^2\,s^{1/2}})$ are obtained from a one-dimensional free

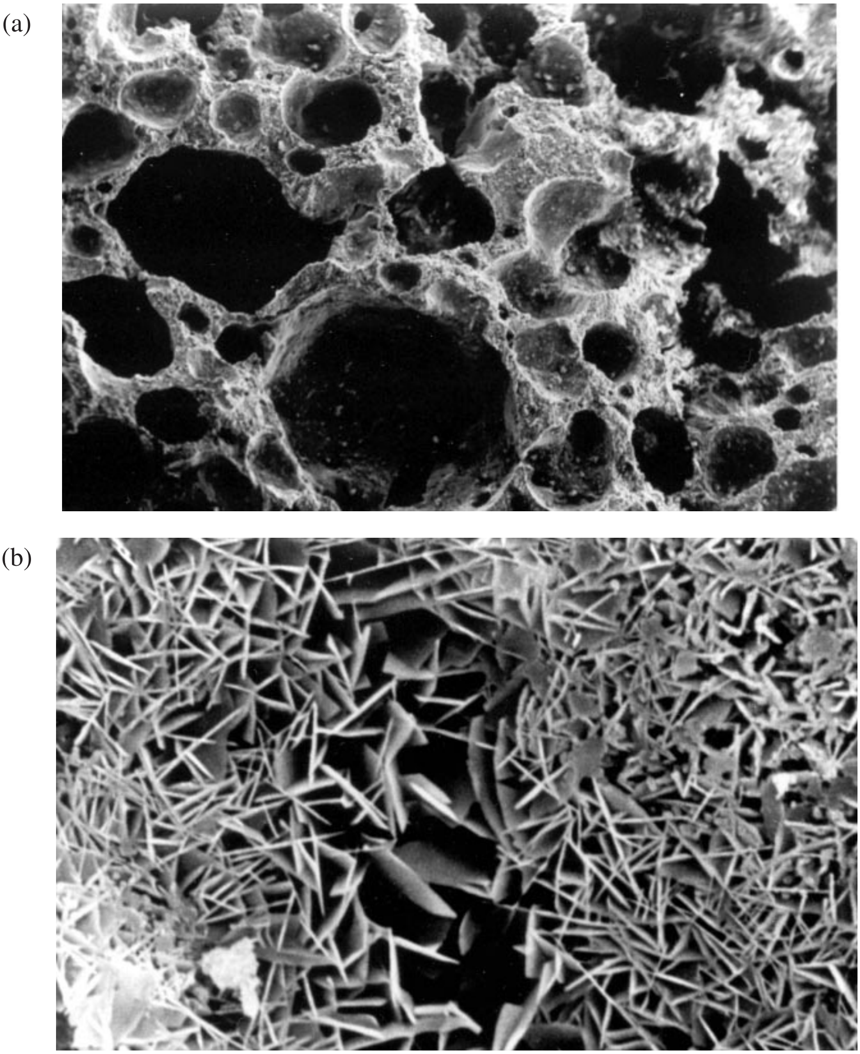


Figure 3. *Pore structure of cellular concrete: (a) low magnification, with the spherical artificial air pores clearly visible; (b) higher magnification, showing the micro-capillaries between the plate-shaped crystals in the macro-pore walls. (Images from [9]).*

imbibition experiment, in which an oven-dry sample is allowed to absorb liquid water from a free water plane.

In general, an imbibition experiment can be divided into two stages. During the first stage, the position of the waterfront gradually approaches the

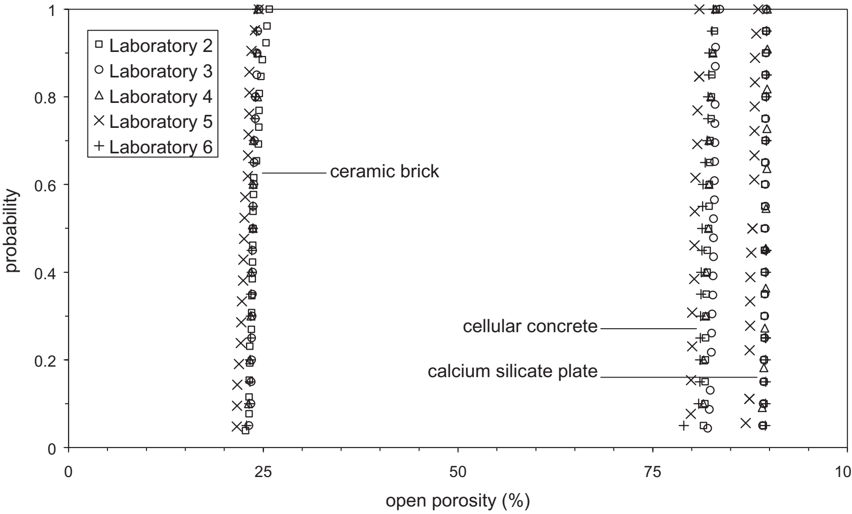


Figure 4. *Cumulative distribution functions for measurements of the total open porosity.*

opposite side of the sample. Water uptake is governed by capillary and viscous forces. During the second stage, the waterfront has reached the upper surface of the specimen and any further increase of the moisture content can be contributed to a dissolution and removal of the air that is entrapped in water, and to a variation at the infiltrating waterfront. The water absorption coefficient A is defined as the slope of the first stage of the cumulative inflow curve as a function of square root of time. The capillary moisture content w_{cap} is, by definition, equal to the moisture content of the specimen at the end of the transition from the first to the second stage. Sample preparation, including sample size, direction of flow, oven drying etc., as well as the boundary conditions during the experiment were carefully prescribed. The lateral sides had to be sealed to reduce evaporation, but the way of sealing remained free. Details of the prescriptions can be found in [10].

Table 2 shows the results. To determine the capillary moisture content and capillary absorption coefficient for the AAC appeared to be very difficult. As can be seen in Figure 5 the cumulative inflow versus square root of time is not linear. While there are many possible explanations for this non-linearity, for example, the presence of micro-cracks and the filling of the macro-pores directly in contact with the water plane at the initial stage or evaporation losses at the top side, a distinction between first and second stage of the process becomes arbitrary.

Ceramic brick and calcium silicate plate show a distinct first and second stage; and a linear relationship between cumulative water inflow and square

Table 1. Mean and standard deviation of the total open porosity Ψ_o(%), bulk density ρ (kg/m^3) and matrix density ρ_{mat} (kg/m^3). In brackets the number of samples is given. The results of Laboratory 5 (plotted in italic) are not incorporated in the mean values.

	Calcium Silicate Plate		**Fired Clay Brick**		**Cellular Concrete**	
	Mean	**St Dev**	**Mean**	**St Dev**	**Mean**	**St Dev**
Open Porosity						
Laboratory 2	89.40	0.08 (20)	23.98	0.80 (26)	82.21	0.43 (20)
Laboratory 3	89.44	0.13 (20)	23.78	0.30 (20)	82.81	0.33 (23)
Laboratory 4	89.52	0.21 (11)	23.73	0.44 (10)	82.15	0.50 (10)
Laboratory 5	*87.81*	*0.40 (18)*	*22.70*	*0.76 (21)*	*80.45*	*0.40 (13)*
Laboratory 6	89.56	0.06 (20)	23.64	0.35 (20)	81.50	0.81 (20)
Mean	89.47	0.13 (71)	23.81	0.56 (76)	82.20	0.74 (73)
Bulk Density						
Laboratory 2	270.32	1.22 (20)	2001.19	18.25 (26)	454.52	9.37 (20)
Laboratory 3	269.24	0.97 (20)	2005.60	6.76 (20)	442.03	7.87 (23)
Laboratory 4	266.06	2.16 (11)	1992.81	10.73 (10)	432.24	13.71 (10)
Laboratory 5	*265.75*	*1.50 (18)*	*2035.19*	*35.94 (21)*	*447.22*	*7.09 (13)*
Laboratory 6	263.97	0.76 (20)	2007.33	8.46 (20)	461.72	13.63 (20)
Mean	267.57	2.92 (71)	2002.86	13.25 (76)	449.51	14.93 (73)
Matrix Density						
Laboratory 2	2549.19	14.55 (20)	2632.66	5.34 (26)	2555.42	28.59 (20)
Laboratory 3	2548.77	27.30 (20)	2631.39	3.05 (20)	2572.24	14.30 (23)
Laboratory 4	2539.66	50.09 (11)	2612.81	3.06 (10)	2435.37	65.89 (10)
Laboratory 5	*2181.43*	*70.18 (18)*	*2633.14*	*35.94 (21)*	*2288.77*	*58.97 (13)*
Laboratory 6	2527.8	10.68 (20)	2628.75	1.81 (20)	2497.92	63.13 (20)
Mean	2541.57	26.61 (71)	2628.68	7.34 (76)	2528.52	64.31 (73)

root of time. For these kind of materials the determination of the capillary absorption coefficient and capillary moisture content appears to be reliable, though as can be seen in Figure 6, it is less reproducible than a vacuum saturation test. Analysing these results, it was concluded that the specimen handling and data acquisition caused most of these deviations. Especially the results of laboratories which are not experienced in the experimental procedure diverged. This is for instance the case for Laboratory 1. Therefore additional series of free water uptake experiments was carried out. This time, however, the specimen handling procedure was more precisely prescribed. This included a weighing schedule designed in such a way that an equidistant square root of time interval was obtained.

Three laboratories performed each at least three additional free uptake experiments. The results showed a very good agreement for calcium silicate plate (mean values respectively 1.18, 1.19 and 1.18 kg/m^{2}s$^{1/2}$). In case of

Table 2. Mean and standard deviation of the capillary moisture content w_{cap} (kg/m^3) and water absorption coefficient A (kg/m^{2}s$^{0.5}$). In brackets the number of samples is given. The results of Laboratory 1 (plotted in italic) are not incorporated in the mean values.

	Calcium Silicate Plate		**Ceramic Brick**		**Cellular Concrete**	
	Mean	**St Dev**	**Mean**	**St Dev**	**Mean**	**St Dev**
w_{cap} (kg/m^3)						
Laboratory 1	*324.33*	*159.91 (19)*	*129.60*	*9.37 (16)*	–	–
Laboratory 2	811.14	6.19 (21)	149.59	7.65 (17)	305.81	17.47 (12)
Laboratory 3	801.48	4.02 (10)	146.27	3.02 (10)	240.64	12.13 (10)
Laboratory 4	817.83	10.05 (10)	161.29	12.05 (10)	329.75	47.16 (10)
Laboratory 5	812.34	2.02 (10)	149.31	4.34 (15)	275.34	24.38 (10)
Laboratory 6	765.91	4.72 (11)	131.59	3.95 (12)	–	–
Mean	802.83	22.37 (62)	147.46	10.97 (64)	288.74	42.85 (42)
A (kg/m^{2}s$^{1/2}$)						
Laboratory 1	*0.395*	*0.184 (20)*	*0.107*	*0.038 (17)*	*0.034*	*0.020 (16)*
Laboratory 2	1.302	0.034 (21)	0.188	0.019 (21)	0.031	0.003 (16)
Laboratory 3	1.236	0.025 (10)	0.175	0.012 (10)	0.023	0.002 (10)
Laboratory 4	1.184	0.047 (10)	0.148	0.016 (10)	0.040	0.007 (10)
Laboratory 5	1.149	0.038 (10)	0.145	0.008 (21)	0.033	0.003 (10)
Laboratory 6	1.162	0.070 (11)	0.133	0.014 (12)	0.028	0.002 (7)
Mean	1.223	0.085 (62)	0.160	0.028 (74)	0.031	0.007 (53)

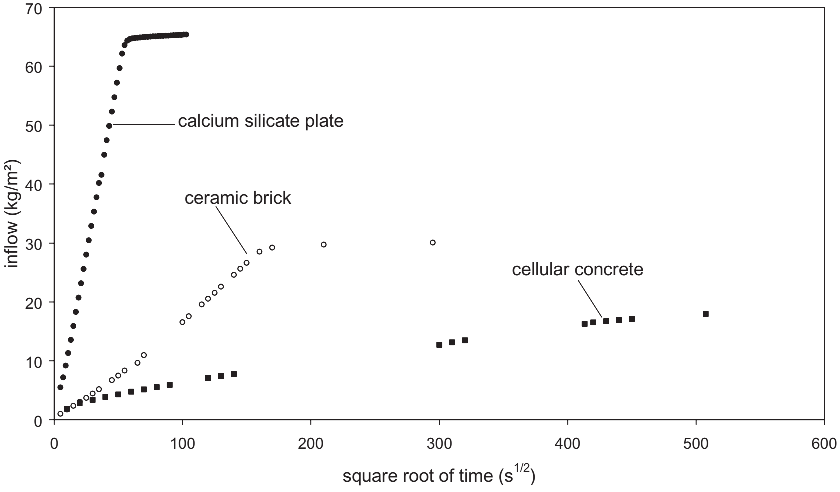


Figure 5. *Example of a cumulative inflow curve for free water uptake in calcium silicate plate, ceramic brick and cellular concrete. For the latter, no clear first and second stage can be determined, making the determination of the capillary absorption coefficient and capillary moisture content arbitrary.*

ceramic brick it was found that different ways of sealing gave different results. The agreement between samples with the same kind of sealing was acceptable. So, it can be concluded that a free water uptake experiment is an easy-to-perform and informative experiment, but there is a need for a precisely prescribed measuring method based on a profound study of all possible influences (e.g. the way of sealing) on the obtained results.

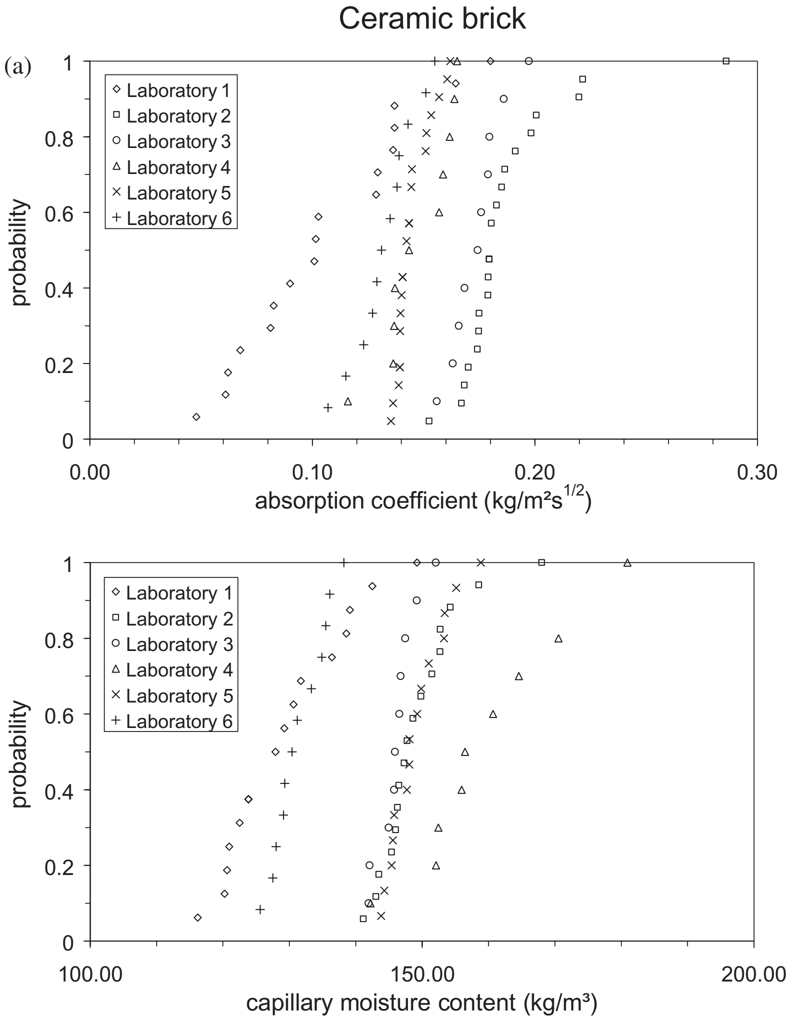


Figure 6. *Cumulative distribution functions of capillary absorption coefficient (top) and capillary moisture content (bottom) for ceramic brick and calcium silicate plate; (b) as measured by the participating laboratories.*

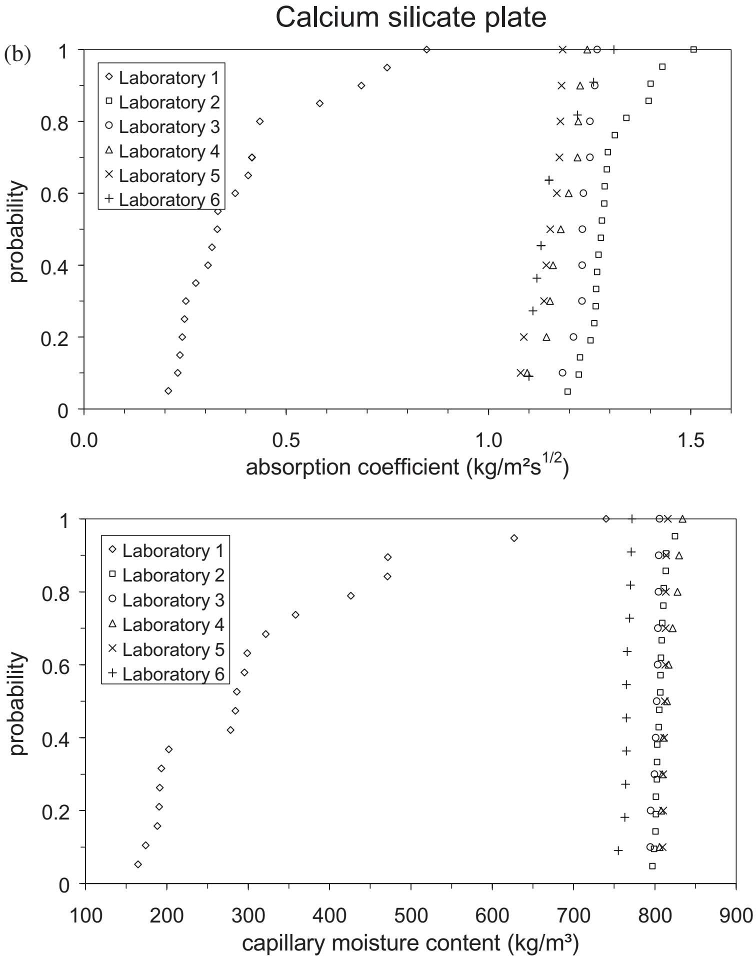


Figure 6. *Continued.*

CAPACITIVE MATERIAL PROPERTIES

The measurements of the capacitive material properties included isothermal ad- and desorption measurements, pressure plate measurements and mercury intrusion experiments.

Isothermal ad- and Desorption Measurements

For the determination of the isothermal adsorption curve, initially oven-dried samples are conditioned in dessicators with saturated salt solutions and at constant temperature until a static sorption equilibrium is attained between the relative humidity in the dessicator and the moisture content of the specimen. For the determination of the desorption isotherm initially capillary-saturated samples are used. For the AAC and calcium silicate plate the measurements are performed at six relative humidities (12, 33, 53, 79.5, 94 and 97%), both for the ad- and desorption isotherm. Since ceramic brick can be considered as hardly hygroscopic, the sorption isotherms were determined based on three relative humidities only (33, 79.5 and 94%). A precise prescription of the test, the followed sample preparation and an overview of all measurement data can be found in [10]. The data of ceramic brick is not relevant because the hygroscopic loading of the material is too low.

Figure 7 shows the adsorption and desorption isotherm of calcium silicate plate. The dashed line plotted on the adsorption data corresponds to the boundary of the mean values, with the outlier of Laboratory 4 not included. It can be seen that a rather good agreement is found as long as the relative humidity remains below 80%. At higher values the differences between the laboratories increase, but at the same time the standard deviation as determined by each laboratory increases likewise. The agreement of the desorption data is much less.

One can conclude that, although sorption data are commonly used, desorption measurements and adsorption at high relative humidity are probably not as reliable as believed. Possible causes are the purity of the salt, no constant relative humidity in the dessicators (opening the dessicator will disturb the measurement), the criteria for static equilibrium (in this study, repeated weighing, at intervals of at least one week, show a difference in mass of less than 0.1%), and in case of desorption measurements the fact that the moist specimen will increase the humidity in the chamber and possible condensation dripping on the specimens.

Pressure Plate Measurements

The pressure plates were mainly performed in accordance with the guidelines of a previous round robin test [11], except that in this study the experiments started from capillary saturation instead of vacuum saturation. Figure 8 compares the mean values as measured by the participating laboratories on calcium silicate plate. Except for the results of Laboratory 5, the measured retention curves are in close agreement but show a small horizontal shift as the curves go down. The dashed line corresponds to the boundary of the mean values, excluding the outlier of Laboratory 5. All other results and the exact measurement data can be found in [10].

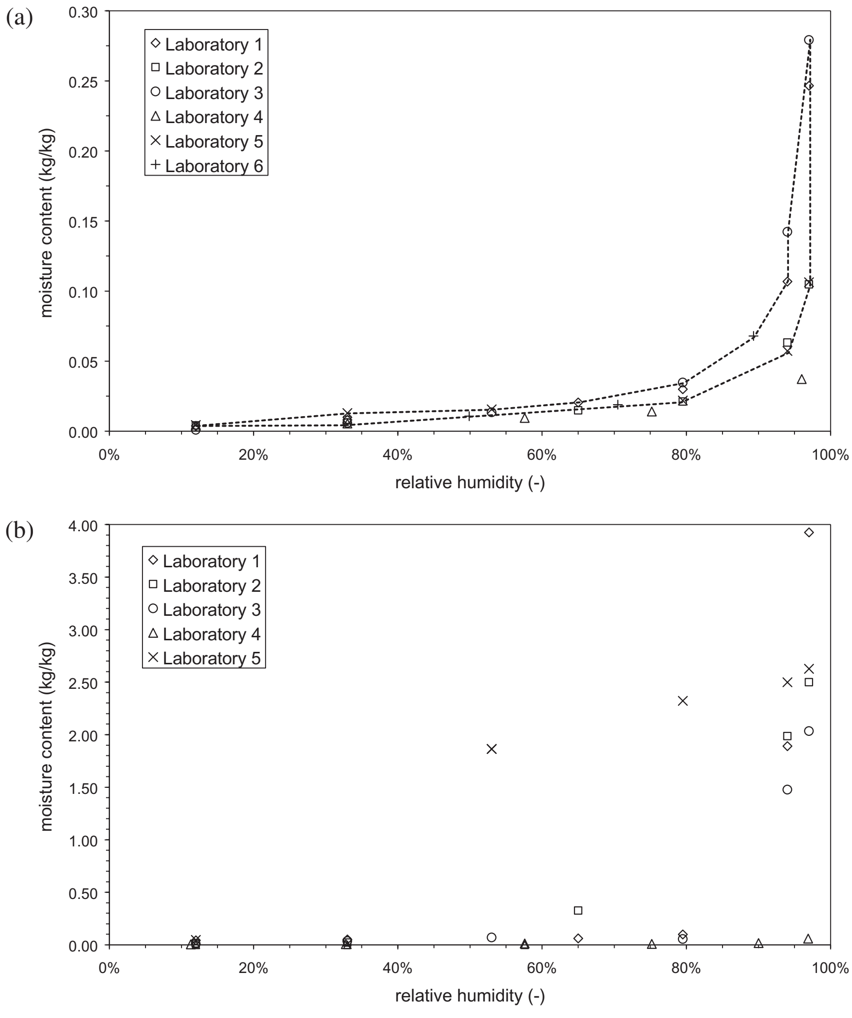


Figure 7. *Mean values of the isothermal adsorption data (a) and desorption data (b) as measured by the participating laboratories on calcium silicate plate. The dashed line plotted on the adsorption data corresponds to the boundary curve of the mean values with the outlier of Laboratory 4 not included.*

Mercury Intrusion Experiments

Mercury intrusion porosimetry was used to characterise the pore space of the three materials by measuring the cumulative pore size distribution. Details on sample preparation and fixed mercury data for the conversion

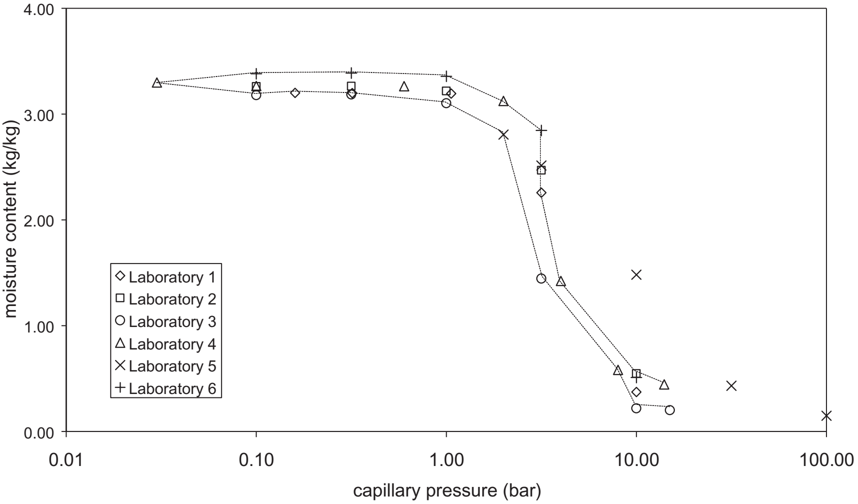


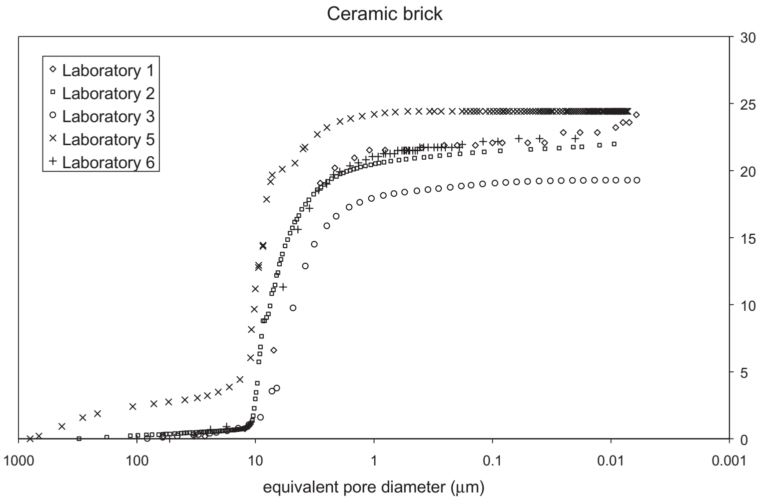
Figure 8. *Mean values of pressure plate data as measured by the participating laboratories on calcium silicate plate. The dashed line corresponds to the boundary curve of the mean values, excluding the outliers of Laboratory 5.*

of the raw data can be found in [10]. As can be seen in Figure 9, showing the obtained cumulative pore size distribution of ceramic brick and calcium silicate plate, a good general agreement of the curve shapes was found. As could be expected, the deviation between the obtained results is much higher for ceramic brick than for calcium silicate plate. This can be attributed to the lack of homogeneity of the material and the fact that mercury intrusion experiments are performed on small samples. In the case of calcium silicate plate, the observed differences are mainly a small horizontal shift between the curves, which may originate from the measuring procedure (manual or automated) and the corresponding criterion used for the determination of an equilibrium state at a certain pressure. It can be concluded that the mercury intrusion experiment is reproducible, but as generally known, the results of mercury intrusion porosimetry have to be interpreted with care.

VAPOUR DIFFUSION TESTS

The vapour transmission is determined in accordance to prEN ISO 12572, except for Laboratory 6, which is not using salt solutions. All three materials are measured at three relative humidities and 5 samples per relative humidity. In Figure 10, the mean values per laboratory of the measured vapour resistance factors are plotted as a function of relative

(a)



(b)

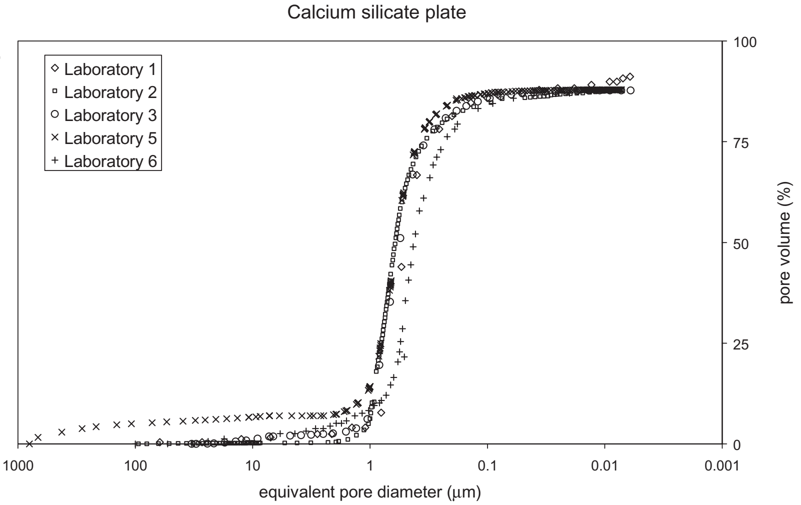


Figure 9. *Cumulative pore size distribution as measured by the participating laboratories with mercury intrusion porosimetry on ceramic brick (a) and calcium silicate plate (b).*

humidity. For all three materials the results of the different laboratories are in the same order of magnitude. However, for experiments that are carried out according to an existing ISO standard, the vapour resistance data show surprisingly high differences. The range between the highest and lowest values was two to four times higher than the low value. However, no systematic difference could be found between the participating laboratories.

DISCUSSION

Based on the interlaboratory comparison it can be concluded that two commonly used measurement techniques require further improvement: the free uptake experiment and the vapour diffusion experiment.

A non-linear behaviour of cumulative water inflow as a function of square root of time was found for AAC. All test data of cellular concrete

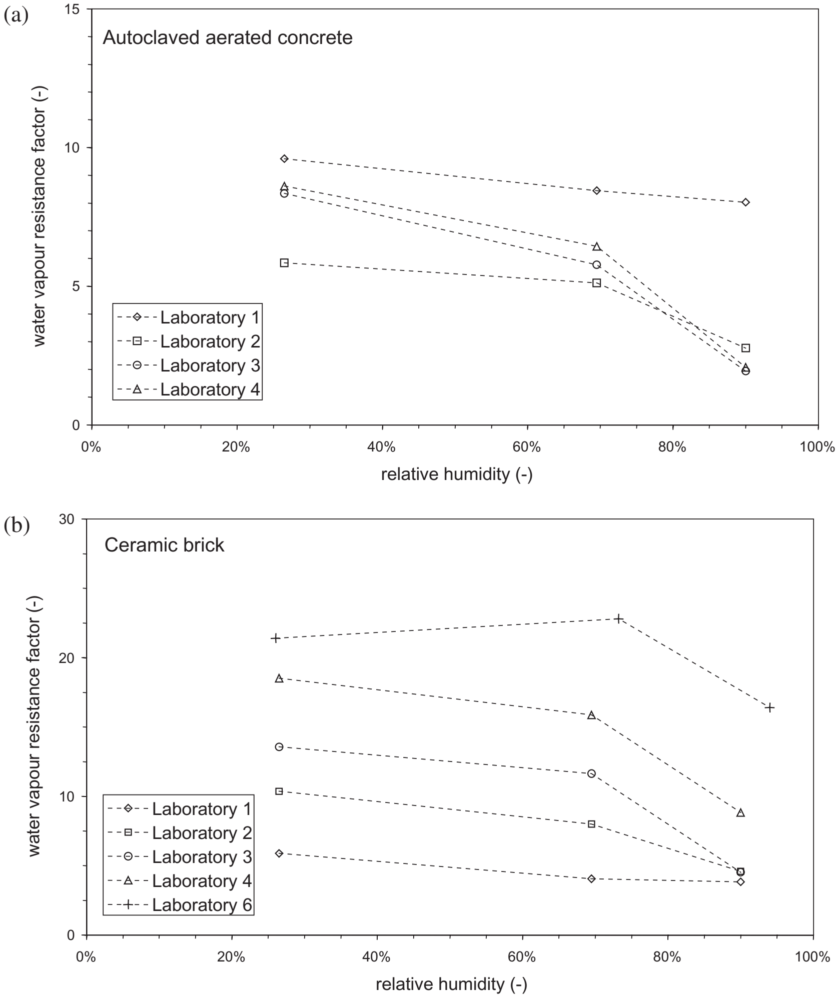


Figure 10. *Water vapour resistance factor (-) plotted as a function of relative humidity for cellular concrete (a), ceramic brick (b) and calcium silicate plate (c).*

(c)

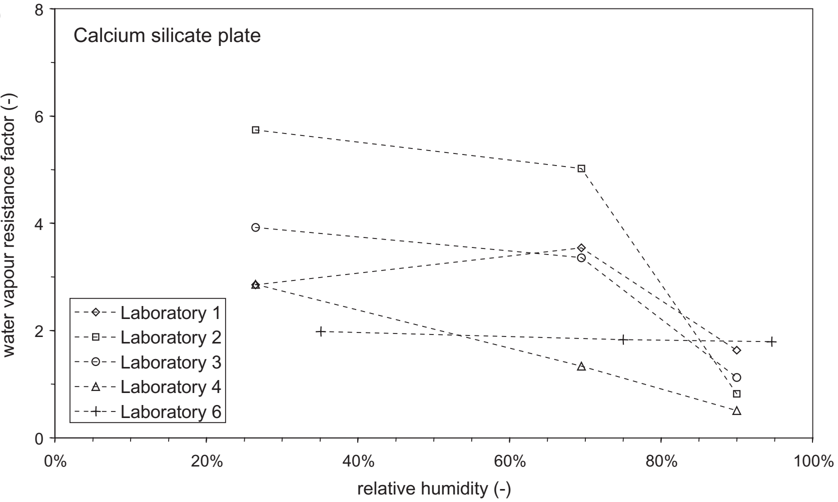


Figure 10. *Continued.*

showed noticeable curvature and force fitting to subsets of data, as performed by the different laboratories, resulted in a poor agreement of the absorption coefficient values. A possible explanation can be found in the microstructure of the material. Cellular concrete consists of large aeration pores imbedded in a fine-pored matrix of high suction. Furthermore, some micro-cracks are present as a result of the production process in the autoclave. One can expect that the filling of the aeration pores and the micro-cracks can not be described by a square root of time model. At least the effects of gravity have to be included. A further study should investigate whether the determination of absorption coefficient and capillary moisture content is impossible for these kind of materials or that the square root of time model can be elaborated (see e.g. [5]).

The additional free uptake experiments showed that the absorption coefficient of ceramic brick depended on the manner of the specimen sealing. This makes the assumption of the absorption coefficient being a material property debatable, since one can expect that for air tight materials the dimensions of the specimen will have the same influence. Therefore, there is an urgent need on a more detailed study of the influence of air and air pressure build-up on the uptake process, with e.g. the work of Descamps [3] as starting point.

Furthermore, it was found that at the end of the uptake process evaporation at the top side may become of the same order of magnitude as the uptake rate in materials with a low absorption coefficient. This makes a clear distinction between first and second stage arbitrary, complicating the determination of the capillary moisture content.

Concerning the water vapour transmission experiments, the range of variation in the measured vapour resistance factors was very large. Several possible causes for these differences have been put forward. It was found that the calcium chloride flocks used to obtain 0% relative humidity in the cup are often not dry enough resulting in a higher relative humidity in the cup and a lower gradient across the specimen. The manual handling of the cups and the small distance between solution level and specimen may bring the specimens in contact with the salt solution, disturbing the results. The standard gives examples of different suitable cups, allowing variation in shape and cups. Whether these variations influence the measuring accuracy is not known. One can conclude that all these items require a further study, what should result in an improvement of the existing standards.

CONCLUSIONS

In the framework of the EU-initiated HAMSTAD-project six laboratories measured the basic hygric properties of three porous building materials – calcium silicate plate, ceramic brick and autoclaved aerated concrete.

Measured values of the total open porosity, bulk density and matrix density, all based on vacuum saturation, were found highly reproducible for all three materials. For two materials determination of the capillary absorption coefficient and capillary moisture content with a one-dimensional free uptake experiment appeared reliable, although its precision depends on the nature of the material. Observed deviations originated mainly from differences in specimen preparation, specimen handling and data acquisition. The results of this study highlight the need to initiate a research project that would study various effects that may influence the results and produce an improvement to this test method. For cellular concrete, the lack of clear first and second stage of water absorption makes use of this technique arbitrary.

Sorption data, although commonly used, were found less reliable than expected. Differences originate from differences in precision of the used balance, frequency of opening the desiccators (and with that disturbing the relative humidity inside) and the way equilibrium is determined. Both the results of pressure plate measurements and mercury intrusion experiments showed to be in close agreement. Deviations between the different results increased for inhomogeneous materials (e.g. ceramic brick) or those with a more complex structure (e.g. AAC).

Finally, the vapour resistance data showed remarkably high differences, though the experiments were carried out according to the existing standard. Several possible causes for the deviations have been put forward, however, a new, in depth study should be started to improve the existing standard.

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