

# Hygric properties of porous building materials (VI): a round robin campaign

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**Abstract:** Hygric properties of porous building materials are important for hygrothermal analysis. Their experimental determination is however not always reliable, shown by the discrepant results from different laboratories on the same materials. In this study, a recent round robin campaign initiated by KU Leuven (Belgium) and participated in by eight institutes from different countries is reported. Ceramic brick was selected as the target material. The bulk density and open porosity from vacuum saturation tests, the capillary absorption coefficient and capillary moisture content from capillary absorption tests, and the vapor permeability from cup tests were measured. Results were analyzed statistically and compared with a previous round robin project, EC HAMSTAD. The reproducibility errors for determining the capillary absorption coefficient were noticeably reduced when compared with the EC HAMSTAD project, and the different laboratories in the present study obtained similar results from vacuum saturation tests and capillary absorption tests without a common protocol. For cup tests, large inter-laboratory discrepancies still exist. However, with a stringent common protocol different laboratories achieved consistent results. For all properties a common protocol did not change the average results of all laboratories.

**Keywords:** porous building material, hygric property, vacuum saturation, capillary absorption, vapor diffusion, round robin

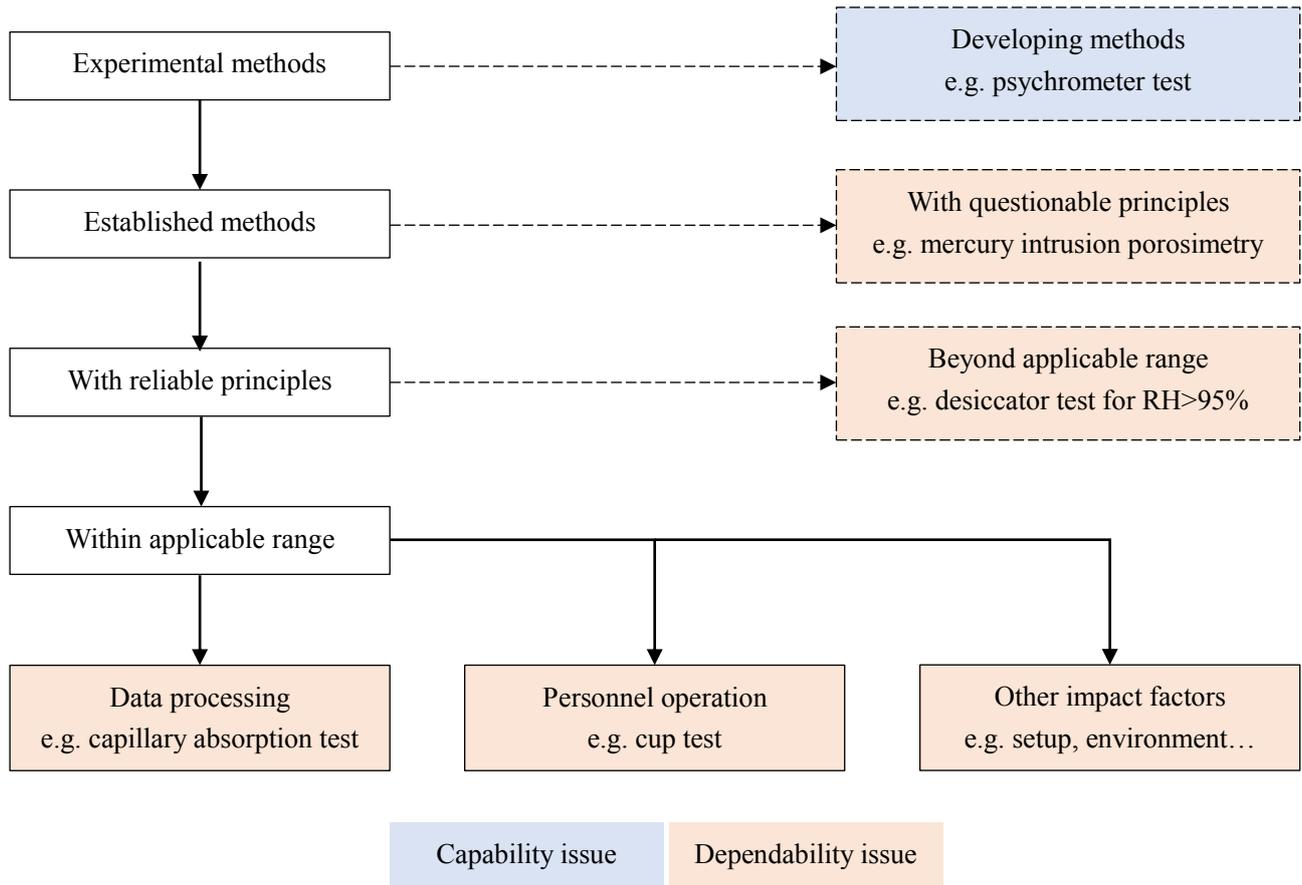
## 1. Introduction

### 1.1 Background

Hygrothermal simulations are valuable for properly designing new buildings and renovating existing ones [1-3]. The built environment can also be optimally understood, controlled and improved with the assistance of hygrothermal simulations [4-6]. To reach these goals, hygrothermal simulations of buildings or the built environment must be performed reliably. Studies show that different

42 hygrothermal models are mathematically similar or even equivalent, and that they can provide similar  
 43 results, as long as two central factors – material properties and boundary conditions – are kept the same  
 44 [7, 8]. It is therefore important to determine material properties as accurately as possible.

45 The material properties involved in hygrothermal simulations can be generally classified as thermal  
 46 properties (e.g. thermal conductivity) or hygric properties (e.g. vapor permeability). One method for  
 47 their determination is via modeling. For instance, pore-structure-based models can be used to predict  
 48 thermal conductivity [9] or moisture storage and transport properties (e.g. moisture retention curve and  
 49 moisture permeability [10]). These pore-structure-based methods are still under development however,  
 50 and measurements hence play the dominant role at present.



51  
 52 **Fig.1 Challenges of measuring hygric properties of porous building materials**

53  
 54 In general, measurements on the hygrothermal properties of porous building materials are physically  
 55 straightforward, contributing to their worldwide application. For decades, experimental protocols for  
 56 measuring thermal properties have been developed progressively. Numerous international, regional  
 57 and national standards are available (e.g. the ISO 8301 and ASTM C518 standards [11, 12]) to  
 58 prescribe the detailed operational procedures. The results are also relatively reliable. However,  
 59 measurements on hygric properties still face capability and dependability issues, as illustrated in Fig.1.  
 60 The capability issues refer to the fact that for some hygrothermal properties in the full humidity range  
 61 their test methods are not yet fully established. For example, there are methods to measure adsorption  
 62 moisture retention curves in the over-hygroscopic range [13-15], but these have not been standardized  
 63 yet. The dependability issues, on the other hand, indicate that the established methods can still fail to  
 64 produce trustworthy results. One possible cause is that the fundamental principles of some established

65 methods might be questionable. For instance, mercury intrusion porosimetry [16] is the most common  
 66 approach to determine the pore volume distribution of porous materials. Its results can be transformed  
 67 into the moisture retention curve for desorption. However, doubts have been raised on its fundamental  
 68 principles, such as the contact angle between mercury and the solid matrix [17], as well as the impact  
 69 of the ink bottle effect [18]. Consequently, the results from mercury intrusion porosimetry should be  
 70 treated carefully. Even if the basic principle of a test method is sound, it can still suffer from great  
 71 uncertainties beyond its applicable range. For example, the desiccator test [19] is one of the most  
 72 widely adopted techniques for measuring sorption isotherms in the hygroscopic range. However,  
 73 saturated salt solutions are not completely reliable for RH levels above 95%. Therefore, in this high  
 74 humidity range the results from desiccator tests show significant scatter [20]. In addition, many factors  
 75 – such as the data processing method [21], the personnel operation [22], etc. – can lead to uncertainties.  
 76 A powerful method to investigate these uncertainties is to launch round robin campaigns, where  
 77 different laboratories perform the same test on the same material. Unfortunately, almost all reported  
 78 round robin campaigns show large deviations between different participants, requiring further studies.

### 79 1.2 Round robin campaigns in brief

80 In the past decades, there have been many round robin campaigns on measured hygric properties of  
 81 porous building materials. Table 1 summarizes some of the most representative projects.

82

83 **Table 1 Representative round robin campaigns for measuring the hygric properties of porous building materials**

Project	Year of completion	Number of participants	Target materials	Measured properties
CEC EUR 14394 EN [23]	1993	13	XPS, particle board	Vapor permeability
Nordtest 1267-96 [24]	1998	4	Sandstone	Bulk density, open porosity, moisture retention
Nordtest 1529-01 [25]	2003	6	Hard wood fibre board, underlay for roofing, damp-proof course	Vapor permeability
EC HAMSTAD [26]	2003	6	Calcium silicate, aerated concrete, ceramic brick	Bulk density, open porosity, capillary absorption coefficient, capillary moisture content, vapor permeability, sorption isotherm, moisture retention, etc.
IEA Annex 41 [27]	2008	14	Gypsum board (coated and uncoated)	Vapor permeability, sorption isotherm

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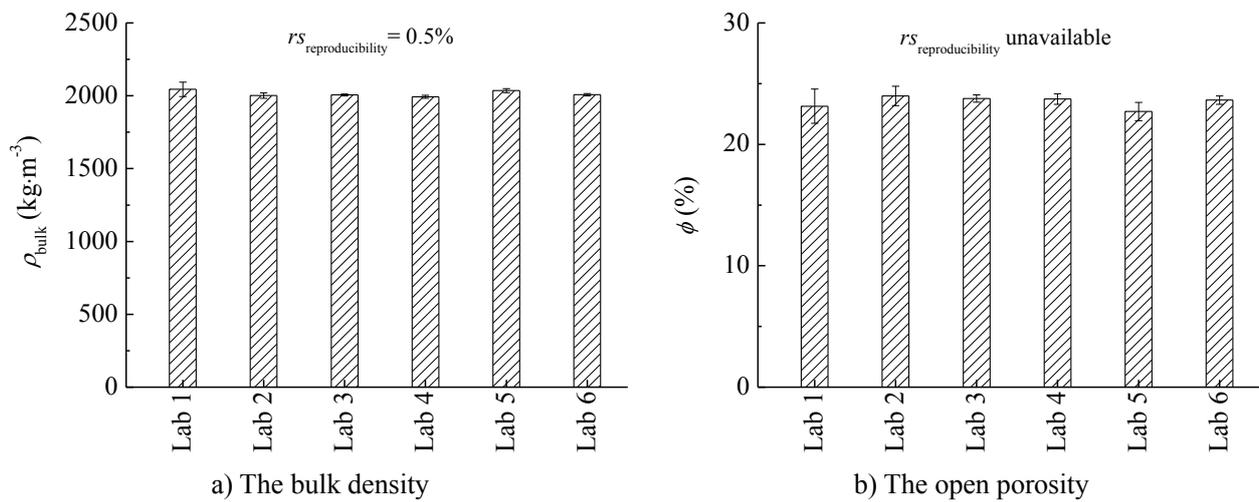
85 Of all these round robin campaigns, the EC HAMSTAD project is one of the most representative  
 86 examples. It involved three different porous building materials with “strong capillarity + strong  
 87 hygroscopicity” (calcium silicate), “weak capillarity + strong hygroscopicity” (autoclaved aerated  
 88 concrete) and “strong capillarity + weak hygroscopicity” (ceramic brick), representing a wide range  
 89 of porous building materials. The set of measured hygric properties was also extensive, covering  
 90 storage and transport properties in both the hygroscopic and the over-hygroscopic ranges, following  
 91 general guidelines. Consequently, in the following analysis we use the results from the EC HAMSTAD  
 92 project for comparison.

93 Some results from the EC HAMSTAD project for ceramic brick are illustrated in Fig. 2-4, with the  
 94 error bars showing standard deviations for duplicate samples. These results include the bulk density  
 95 ( $\rho_{\text{bulk}}$ ,  $\text{kg}\cdot\text{m}^{-3}$ ) and open porosity ( $\phi$ ) from the vacuum saturation test, the capillary absorption  
 96 coefficient ( $A_{\text{cap}}$ ,  $\text{kg}\cdot\text{m}^{-2}\text{s}^{-0.5}$ ) and capillary moisture content ( $w_{\text{cap}}$ ,  $\text{kg}\cdot\text{m}^{-3}$ ) from the capillary absorption

97 test, as well as the vapor permeability (expressed as the vapor diffusion resistance factor  $\mu$ ) from the  
 98 cup test. All these test methods are well established and used within their application ranges. Two  
 99 tendencies can be clearly identified. Firstly, the discrepancies between different laboratories are often  
 100 much greater than the uncertainties within respective laboratories. Consequently, this restricts us to the  
 101 reproducibility errors ( $r_{S\text{reproducibility}}$ , explained in Section 2.3, also indicated in Fig. 2-4), which account  
 102 for the inter-laboratory differences. Secondly, the reproducibility errors for the moisture transport  
 103 properties (such as  $A_{\text{cap}}$ ) are in most cases greater than those for the moisture storage properties (such  
 104 as  $w_{\text{cap}}$ ).

105 In fact, these two tendencies observed in the EC HAMSTAD project are prevalent in all round robin  
 106 campaigns. Motivated by these phenomena, some in-depth studies have been conducted for a better  
 107 insight into and control of potential error sources. For example, repeatability and reproducibility  
 108 analysis proves that neither the material inhomogeneity nor the experimental errors rooted in the  
 109 methodologies themselves play a dominant role in the conspicuous inter-laboratory discrepancies [28].  
 110 Furthermore, time and personnel in the same lab do not lead to large errors, as long as protocols remain  
 111 unchanged [22]. As a result, the experimental procedure, environmental control and the data  
 112 processing appear to be the most responsible.

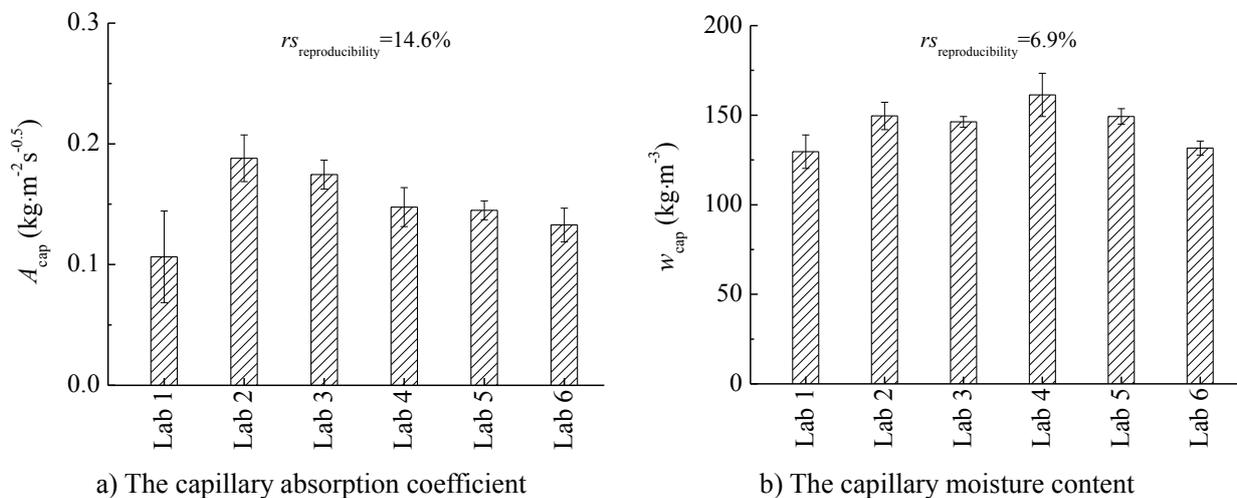
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**Fig. 2 Results of vacuum saturation tests for ceramic brick (from the EC HAMSTAD project [26])**

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**Fig. 3 Results of capillary absorption tests for ceramic brick (from the EC HAMSTAD project [26])**

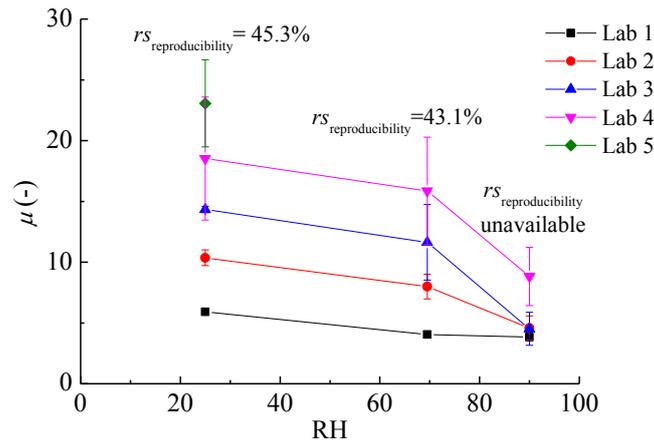


Fig. 4 Results of cup tests for ceramic brick (from EC HAMSTAD project [26])

### 1.3 Objectives

As many years have passed since the completion of the above-mentioned round robin campaigns, a new small-scale round robin campaign was initiated by KU Leuven (Belgium), to gain fresh input to quantify the consistency of experimental results from different laboratories, to locate sources of discrepancies, and to identify potential improvements in different laboratories. This campaign was planned at the end of 2017 and formally started in early 2018. Besides KU Leuven as the project coordinator, eight other institutes contributed to this campaign by measuring hygric properties: University of Porto (Portugal), China Academy of Building Research (P.R. China), Łódź University of Technology (Poland), University of Edinburgh (United Kingdom), Technical University of Dresden (Germany), Lund University (Sweden), Technical University of Denmark (Denmark) and Czech Technical University in Prague (Czech Republic). In addition, the X-ray diffraction (XRD) data were analyzed by University of Strathclyde (United Kingdom) for extra information. It should be noted that there was no direct funding for this campaign, so all participants were voluntarily involved and financially self-supported.

In the following sections, we first introduce the material and the methods used in this round robin campaign. Next, the experimental results from the participating laboratories are presented along with statistical analysis and comparison with the EC HAMSTAD project. After that, the conclusions from this study are drawn.

## 2. Material and methods

In this section, we first present the target material used for this round robin campaign. After that, the experimental arrangement and test methods are explained. Finally, statistical analysis methods are introduced.

### 2.1 Material

Given that this round robin campaign was unfunded, only a single target material was used: the Robusta Vandersanden ceramic brick [29]. It has a dark brown color and a raw dimension of 21cm×10cm×5cm. This brick does not go through carbonation, hydration or any other noticeable chemical change during the test period. Its mineral composition obtained by quantitative XRD is illustrated in Fig. 5. The crystalline minerals present are quartz (41.3%), Ca-rich plagioclase feldspar (anorthite) (19.7%), K-feldspar (sanidine) (2.7%), hematite (4.1%) and diopside (7.8%). The estimated silica glass content is 24.4% using the PONKCS method [30]. The mineral assemblage is typical of a

150 modern production brick with considerable calcium content, and with hematite and diopside as high-  
151 density components. From the crystallographic mineral densities and the estimated density of the  
152 glassy phase [31], we estimate a solid density for this brick ceramic of 2740-2775 kg·m<sup>-3</sup>. Fig. 6  
153 illustrates the pore volume distribution of this brick obtained by the mercury intrusion porosimetry.  
154 Based on its pore size, we can expect the brick to be strong in capillarity and weak in hygroscopicity.

155 For this round robin campaign, raw bricks from the same batch were randomly selected and  
156 distributed by KU Leuven to all other participants, to minimize the impact of material inhomogeneity.  
157

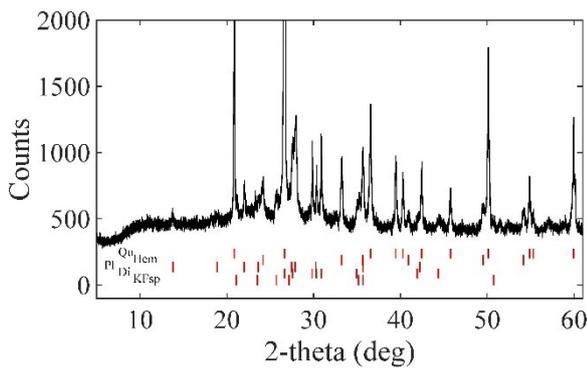


Fig. 5 The mineral phases of the target brick

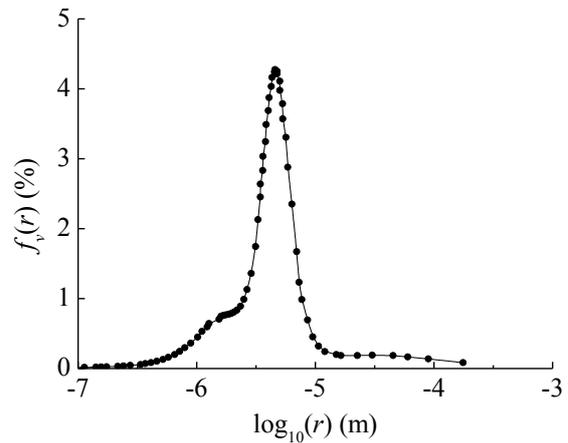


Fig. 6 The pore volume distribution of the target brick

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## 159 2.2 Test methods

160 This round robin campaign focuses on established methods for measuring the hygric properties of  
161 porous building materials. The vacuum saturation test was executed for bulk density and open porosity,  
162 the capillary absorption test was performed for capillary absorption coefficient and capillary moisture  
163 content, and the cup test was carried out for vapor permeability. As the target brick is weak in  
164 hygroscopicity, the determination of hygroscopic sorption isotherms was not included. Moreover,  
165 because the pressure plate setup was not available in all participating laboratories, the moisture  
166 retention curve was not measured.

167 Two separate rounds of experiments were performed in the campaign. In the 1<sup>st</sup> round, all  
168 participants carried out the tests according to their respective experimental protocols used in their own  
169 laboratories. Here, no instructions regarding sample size, relative humidity levels to include in the cup  
170 test, etc. were given. For the 2<sup>nd</sup> round, a strict and detailed common protocol was prescribed for each  
171 test. The latter was based on standards ASTM C1585, ASTM C1794, ASTM E96, ISO 12572 and ISO  
172 15148 [32-36], as well as on previous studies [28, 37-39]. Below, some key information on the  
173 common protocols for the 2<sup>nd</sup> round is given, while full descriptions are provided in Appendix A.

### 174 2.2.1 General requirements

175 The ambient temperature for all measurements should be maintained within 20-25°C, with a  
176 fluctuation smaller than ±1°C. The surface 0.5-1 cm of raw bricks should be removed, and at least 4  
177 duplicates are required for each test. Sample dimensions should be measured with calipers reading to  
178 0.01 mm. Samples should be dried in a ventilated oven at 70°C for at least 7 days.

### 179 2.2.2 Vacuum saturation test

180 The air pressure in the vacuum container should stay below 3000 Pa for at least 4 h to completely  
181 evacuate the air inside samples. While filling water, the water level should rise at a speed of around 5

182 cm·h<sup>-1</sup> (or slower) until all samples are completely underwater. After returning the air pressure in the  
183 container to atmospheric pressure, samples should be kept underwater for at least 24 h before weighing.

#### 184 2.2.3 Capillary absorption test

185 The top and lateral sides of the sample should be wrapped with either plastic film or aluminum foil,  
186 with 1-2 small holes at the top to allow air evacuation. The bottom 1 cm of the lateral sides should be  
187 left unwrapped to avoid capillary uptake between the sample and the wrap. The water in the basin  
188 should stay 3-5 mm above the sample bottom. The capillary absorption coefficient should be derived  
189 according to the one-tangent method [21], and the capillary moisture content should be calculated  
190 according to the Appendix.

#### 191 2.2.4 Cup test

192 The sample should be sealed on the opening of the diffusion cup with epoxy, paraffin or other vapor-  
193 tight methods. While sealing, the penetration of the sealant into the sample should be minimized. The  
194 air layer resistance inside the cup should be corrected, and the surface transfer resistance above the  
195 sample should be minimized by increasing the air velocity to at least 1 m·s<sup>-1</sup>. While processing the  
196 data, the sample's masked edge (if any) should be corrected according to the ISO 12572 standard [35].

### 197 2.3 Statistical analysis methods

198 To quantitatively evaluate the experimental results, two statistical methods were employed. First,  
199 the t-test was used to compare the overall results from the 1<sup>st</sup> and the 2<sup>nd</sup> rounds of experiments. Due  
200 to the availability of time, personnel and experimental facilities, not all laboratories finished all three  
201 tests for the five properties in both rounds. As a result, the paired t-test is not applicable and hence the  
202 independent t-test was utilized instead [40]. Specifically, for any given property, the average value of  
203 each lab was calculated first. Next, all the lab-averaged values were classified into two groups for the  
204 1<sup>st</sup> and the 2<sup>nd</sup> rounds, respectively. After that, the independent t-test on these two groups was  
205 performed, verifying whether the lab-averaged results of the two rounds of experiments are statistically  
206 different.

207 The main aim of the independent t-test is to compare the averages of the 1<sup>st</sup> round and the 2<sup>nd</sup> results  
208 of all laboratories. However, it cannot clarify the major concern of this study – the discrepancies  
209 between different laboratories – in a thorough way. For this reason, we also adopted the statistical  
210 indicators proposed by the ISO 5725 standard [41], which have also been applied in other studies [22,  
211 28]:

- 212 a) The material error ( $rS_{\text{material}}$ ): representing the errors (relative standard deviations) caused by a  
213 material's inhomogeneity;
- 214 b) The repeatability error ( $rS_{\text{repeatability}}$ ): representing the errors in repeating the measurements on the  
215 same samples when all relevant factors (operator, equipment, calibration, etc.) remain unchanged;
- 216 c) The reproducibility error ( $rS_{\text{reproducibility}}$ ): representing the errors of replicate measurements in  
217 different laboratories.

218 In general,  $rS_{\text{material}}$  can never be eliminated, since no material is perfectly homogeneous. Meanwhile,  
219  $rS_{\text{repeatability}}$  reflects the smallest random error inherent to a test method (when  $rS_{\text{material}}$  is not considered)  
220 and hence indicates the lower limit of experimental accuracy. If any error is smaller than or similar to  
221  $rS_{\text{material}}$  and  $rS_{\text{repeatability}}$ , it cannot be clearly identified and can be neglected accordingly. Finally,  
222  $rS_{\text{reproducibility}}$  stands for the largest random errors in different laboratories and directly show their  
223 discrepancies. Obviously, the smaller  $rS_{\text{reproducibility}}$  is, the more similar results different laboratories can  
224 achieve.

225 The calculation of  $rS_{\text{material}}$ ,  $rS_{\text{repeatability}}$  and  $rS_{\text{reproducibility}}$  is intricate: details have been elaborated in

226 ref. [28, 41], and we do not repeat them in this paper. It should be noted that due to the calculation  
 227 complexity,  $r_{S\text{reproducibility}}$  is not always available. This mainly happens to extreme cases when the  
 228 between-lab errors are comparable to the within-lab errors (such as the open porosity in the EC  
 229 HAMSTAD project, Fig. 2 b), so that the differences between different laboratories cannot be  
 230 distinguished. It is also worth mentioning that  $r_{S\text{material}}$  and  $r_{S\text{repeatability}}$  obtained in different laboratories  
 231 can vary slightly. However, since they are normally much smaller than  $r_{S\text{reproducibility}}$ , their small  
 232 variances should only have a limited impact on the general analysis. In this round robin campaign, we  
 233 adopt the  $r_{S\text{material}}$  and  $r_{S\text{repeatability}}$  for the target ceramic brick obtained by KU Leuven. As is clearly  
 234 reflected in Table 2, these two errors are typically smaller than 10% (cup tests), 5% (capillary  
 235 absorption tests) and 1% (vacuum saturation tests).

237 **Table 2 The material and repeatability errors of the target ceramic brick**

Test	Vacuum saturation		Capillary absorption		Cup test*		
Property	$\rho_{\text{bulk}}$	$\phi$	$A_{\text{cap}}$	$w_{\text{cap}}$	$\mu_1$	$\mu_2$	$\mu_3$
$r_{S\text{material}}$ (%)	0.13	0.45	2.76	3.39	8.36	8.79	6.84
$r_{S\text{repeatability}}$ (%)	0.17	0.44	2.02	0.41	2.73	0.55	1.89

238 \* The RH settings for  $\mu_1$ ,  $\mu_2$  and  $\mu_3$  are 11.3%-53.5%, 53.5%-84.7% and 84.7%-97.4%, respectively.

### 240 3. Results and discussion

241 In this section, we report the results obtained from the vacuum saturation test, the capillary  
 242 absorption test and the cup test (detailed values are reported in Appendix B). Analysis of and  
 243 comparisons between the 1<sup>st</sup> and the 2<sup>nd</sup> rounds of experiments are made. Results from the EC  
 244 HAMSTAD project are also referred to. It must be emphasized that the lab numbers in this section are  
 245 denoted as A-I, without any link to the affiliation numbers for the co-authors. It should also be  
 246 remarked that the brick used in this campaign differs from the EC HAMSTAD brick in the  
 247 homogeneity and investigated properties. However, these differences mainly affect the material errors  
 248 to a limited degree, and a comparison of reproducibility errors is still valid. Lastly, it is worth  
 249 mentioning that the common protocols were derived from the routines of Lab A. In other words, Lab  
 250 A was following the common protocols all the time. For this reason, the same set of data from Lab A  
 251 suits both rounds.

252 All tests were carried out at 20-25°C. In this limited temperature range the capillary absorption  
 253 coefficient can vary by about 5% [37, 39, 42], while all other investigated properties can be considered  
 254 as temperature-independent [37]. Consequently, equation (1) derived from ref. [37] was used to correct  
 255 the capillary absorption coefficients from different laboratories to values at 20°C for better comparison.  
 256 Note that Lab I did not report the temperature in the 1<sup>st</sup> round for the capillary absorption test, so their  
 257 results remained uncorrected.

$$258 \quad A_{\text{cap}}(20^\circ\text{C}) = \frac{A_{\text{cap}}(T)}{0.0112(T-273.15)+0.7756} \quad (1)$$

259 where  $T$  is the absolute temperature, K.

#### 260 3.1 Vacuum saturation tests

261 Fig. 7 illustrates the bulk density and open porosity obtained from the vacuum saturation test. A first  
 262 glance shows that for both properties the discrepancies between the different laboratories are not  
 263 significant. However, the open porosity reported by Lab B is noticeably lower in both rounds of

264 experiments. After a thorough check, the experimental procedure has been identified as the main  
 265 reason: contrary to other laboratories who first evacuated the air in the vacuum container and then  
 266 filled in water, Lab B operated in the reverse order. As a result, some air was probably retained in the  
 267 sample, leading to the underestimated open porosity. Moreover, Lab F surprisingly provided an even  
 268 smaller open porosity than Lab B, which is problematic. There is no clear explanation for this  
 269 underestimation, and the insufficient air evacuation before water filling may also be the most possible  
 270 reason.  
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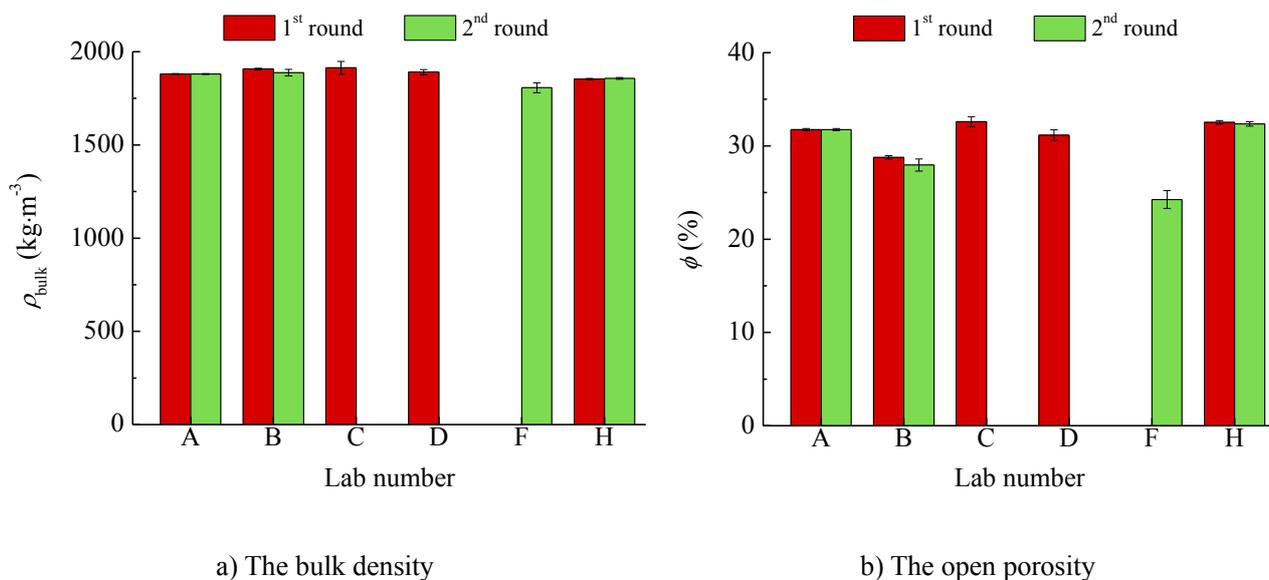


Fig. 7 Results of the vacuum saturation test

272  
 273  
 274 With bulk density and open porosity, the matrix density could be easily derived for a further check  
 275 [43, 44]. As summarized in Table 3, the matrix densities from most laboratories are similar.  
 276 Nevertheless, Lab F in the 2<sup>nd</sup> round gave an abnormally low value, confirming the existence of  
 277 mistakes during the test. However, the determination of bulk volume by Archimedes buoyancy does  
 278 not require complete air evacuation. Thus the bulk densities obtained by Lab B in both rounds and Lab  
 279 F in the 2<sup>nd</sup> round were still reasonable. The matrix density provides a quality check on the Archimedes  
 280 porosity. For this ceramic brick, the mean matrix density calculated from the measured bulk density  
 281 and measured porosity is 2753 and 2706  $\text{kg}\cdot\text{m}^{-3}$  for the 1<sup>st</sup> round and the 2<sup>nd</sup> round, respectively. These  
 282 values are in acceptable agreement with the estimation from the mineralogical composition by XRD  
 283 (Section 2.1) and place this brick at the high end of the known solid density range of ceramic bricks,  
 284 broadly 2600-2750  $\text{kg}\cdot\text{m}^{-3}$  [43]. However, several laboratories reported values either higher or lower,  
 285 which cannot be reconciled with the known composition. This confirms the value of using the matrix  
 286 density as a quality check [44].

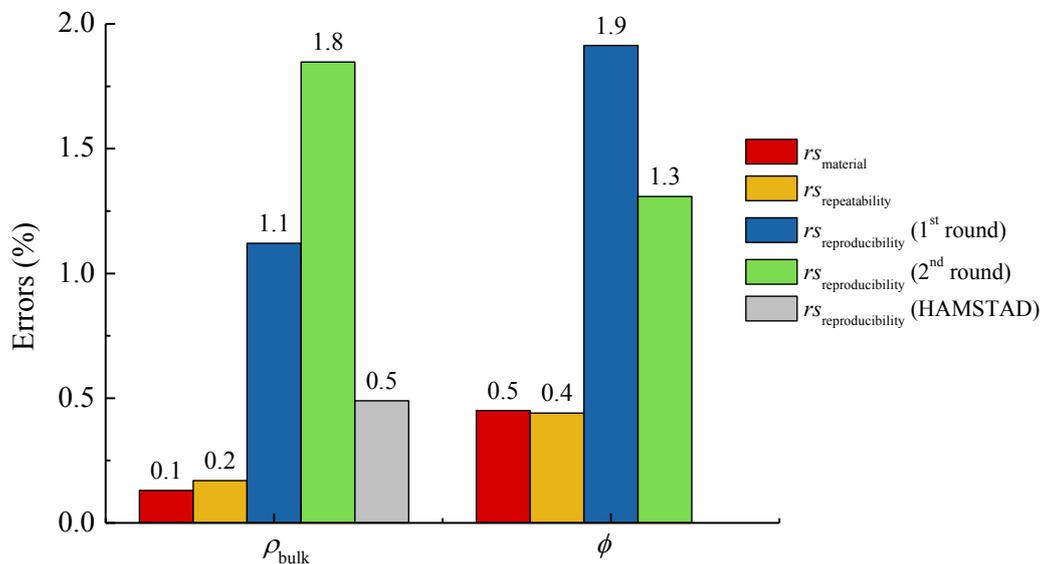
Table 3 The matrix density ( $\text{kg}\cdot\text{m}^{-3}$ ) from different laboratories

Lab No.	A	B	C	D	F	H
1 <sup>st</sup> round	2754	2677	2839	2746	-	2750
2 <sup>nd</sup> round	2754	2620	-	-	2385	2744

289  
 290 To have a quantitative view on the discrepancies between different laboratories in this round robin

291 campaign, we refer to the statistical parameters described in Section 2.3. It should be noted that the  
 292 open porosity from Lab B was not included for error calculations, as its problematic procedure  
 293 produces unrepresentative results. The deviating open porosity from Lab F in the 2<sup>nd</sup> round was also  
 294 excluded. To start with, the independent t-tests were performed to compare the lab-averaged results in  
 295 the 1<sup>st</sup> and the 2<sup>nd</sup> rounds of experiments for all laboratories. The calculated p-values are 0.164 and  
 296 0.947 for the bulk density and the open porosity respectively, indicating that the average values of bulk  
 297 density and open porosity were not statistically different in both rounds. In other words, the common  
 298 protocol for the vacuum saturation imposed in the 2<sup>nd</sup> round of experiments did not change the average  
 299 results of all laboratories significantly.

300 Furthermore, reproducibility errors were evaluated to check whether the common protocol reduced  
 301 the discrepancies between different laboratories. The calculated results are illustrated in Fig.8, in  
 302 comparison with the EC HAMSTAD project. Clearly, in terms of the variations between different  
 303 laboratories, the determination of the bulk density and the open porosity is very satisfactory in both  
 304 rounds. The inter-laboratory discrepancies stay within 2%, albeit slightly greater than those in the EC  
 305 HAMSTAD project. It should be noted that for the bulk density the reproducibility errors in the 2<sup>nd</sup>  
 306 round are slightly greater than those in the 1<sup>st</sup> round. This, however, does not demonstrate that a  
 307 common protocol exerted a negative impact in this case, because fewer laboratories were involved in  
 308 the 2<sup>nd</sup> round, leading to greater statistical uncertainties. Anyway, it can be concluded that the vacuum  
 309 saturation test is highly reliable and a common protocol is not indispensable, as long as the  
 310 experimental procedure is correct. This may be attributed to the fact that the vacuum saturation test is  
 311 very simple in both operational procedure and data processing, without strong impact factors. It is also  
 312 possible that the respective protocols adopted by different laboratories were the same as or modified  
 313 from a standard, with similar and adequate details.

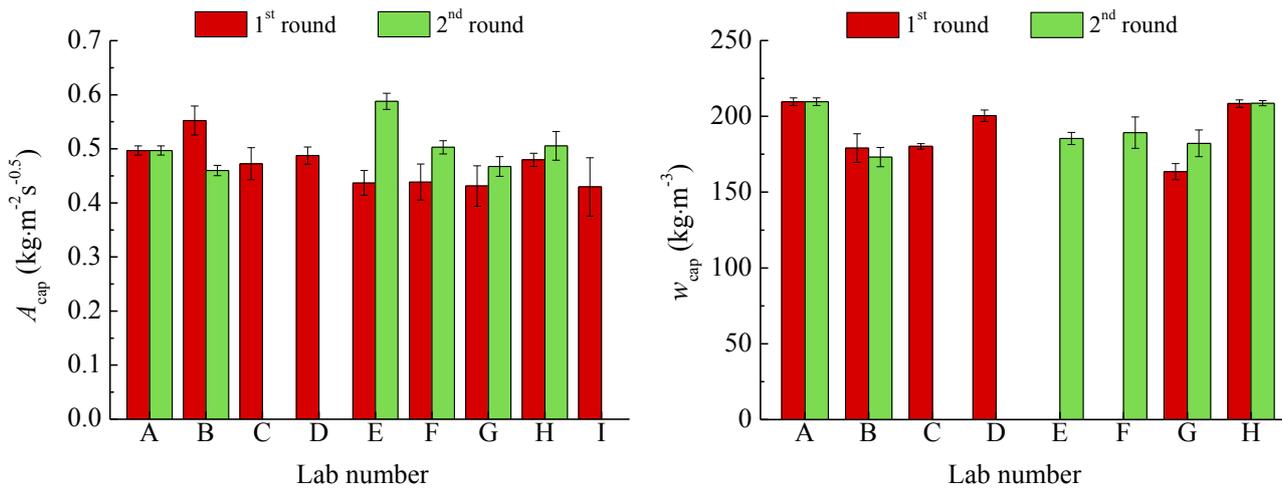


314  
 315 **Fig. 8 Experimental errors of the round robin vacuum saturation test**  
 316

### 317 3.2 Capillary absorption tests

318 Fig. 9 illustrates the capillary absorption coefficient and capillary moisture content obtained from  
 319 the capillary absorption test. Generally speaking, the results of all laboratories stay reasonably similar.  
 320 The calculated p-values from the independent t-tests comparing the overall results of two rounds are  
 321 0.154 and 0.906 for the capillary absorption coefficient and the capillary moisture content, respectively.

322 Similar to the vacuum saturation test, the capillary absorption test hence also showed no statistical  
 323 change in terms of the average results of different laboratories in the 1<sup>st</sup> and the 2<sup>nd</sup> rounds. It should  
 324 be mentioned that in the 1<sup>st</sup> round both Lab G and Lab I used automatic capillary absorption setups,  
 325 while for all other cases in both rounds the manual method was adopted. To analyze the impact of  
 326 different setups, we conducted Duncan's multiple range test [40], comparing the average capillary  
 327 absorption coefficients of Lab G in the 1<sup>st</sup> and the 2<sup>nd</sup> rounds, Lab I in the 1<sup>st</sup> round and all other  
 328 laboratories in the 1<sup>st</sup> round. Results show that all these average values are not statistically different  
 329 ( $p=0.074$ ). This means both automatic and manual measurements could produce similar results,  
 330 agreeing with an earlier study [45].  
 331



a) The capillary absorption coefficient

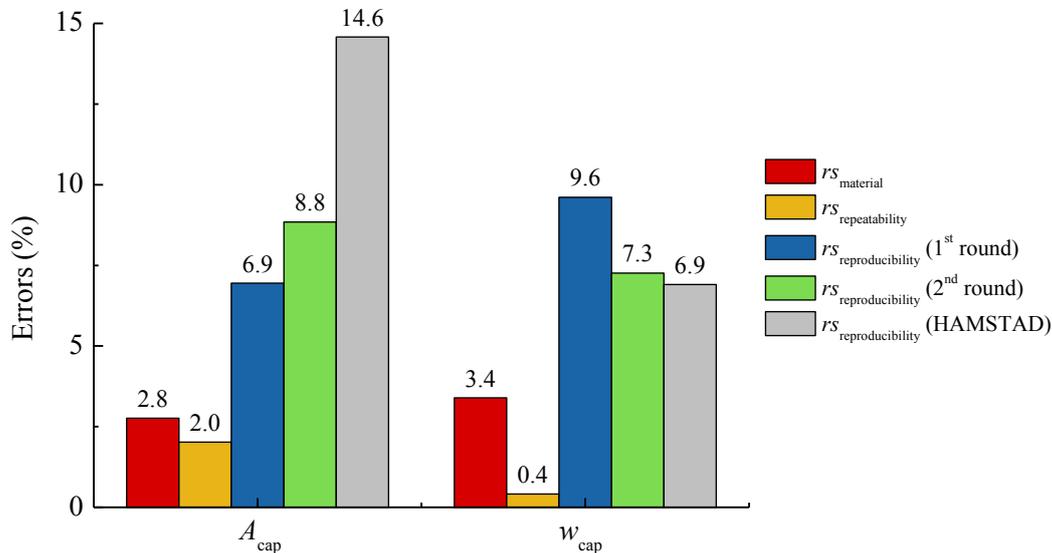
b) The capillary moisture content

**Fig. 9 Results of the capillary absorption test ( $A_{cap}$  corrected to 20°C)**

332  
 333  
 334 The reproducibility errors are illustrated in Fig. 10. It is clear that compared with the EC HAMSTAD  
 335 project, the inter-laboratory errors for measuring the capillary absorption coefficient in this round robin  
 336 has been reduced obviously, even without a common protocol. Resultantly, in this campaign the  
 337 reproducibility errors with and without a common protocol are not very different, always staying  
 338 within 7%-10%, acceptable in most circumstances. It can thus be concluded that the capillary  
 339 absorption test has become dependable now, and that further details through a more specific common  
 340 protocol are not necessary anymore.

341 To have further insight, Table 4 summarizes the key information – sample size and sealing method  
 342 – of the capillary absorption tests in the 1<sup>st</sup> round. In comparison, all laboratories turned to  
 343  $8\text{cm}\times 4\text{cm}\times 12\text{cm}$  as the sample size and used plastic film or aluminum foil for sealing in the 2<sup>nd</sup> round,  
 344 as required by the common protocol. From such information, several interesting phenomena can be  
 345 observed. Firstly, Labs C, E and F used the raw brick as the sample in the 1<sup>st</sup> round, and their results  
 346 (both the capillary absorption coefficient and the capillary moisture content) are among the smallest.  
 347 One possibility is that the brick surface has slightly different characteristics than the core due to the  
 348 manufacturing process. Another potential reason is that due to the coarseness and irregularity of the  
 349 raw brick, the sample size was overestimated, leading to underestimated results. As is reflected in Fig.  
 350 9 a), once switched to the core material in the 2<sup>nd</sup> round, the capillary absorption coefficients from  
 351 Labs E and F increased immediately. Secondly, varied sample sizes – especially the height – were

352 chosen by respective laboratories in the 1<sup>st</sup> round. The results are however not highly different, even  
 353 when compared with the 2<sup>nd</sup> round experiments with the same sample size. This agrees with an earlier  
 354 study, stating that the sample size has a limited impact on the capillary absorption test [21]. Last but  
 355 not least, the sealing methods used by different laboratories in the 1<sup>st</sup> round showed a great variety,  
 356 and no significant influence can be observed. However, the cases with penetrating sealants (e.g. Labs  
 357 E, F and I) generally produced lower capillary absorption coefficients, indicating that the potential  
 358 sealant penetration may reduce the cross-sectional area. It is therefore more advisable to choose films  
 359 for the sealing, as suggested in ref. [21]. Interestingly, Lab H did not seal samples in the 1<sup>st</sup> round but  
 360 still obtained consistent results with other laboratories. This is because the aim of sealing is to prevent  
 361 evaporation during the capillary absorption process. If a sample absorbs much moisture within a short  
 362 period of time (such as the brick investigated in this study), then the evaporation from its surfaces only  
 363 exerts a limited impact and the sealing is hence no longer indispensable. Since it is difficult to define  
 364 “much” and “short period” quantitatively, it may be more advisable to always seal samples with films.  
 365



366  
 367 **Fig. 10 Experimental errors of the round robin capillary absorption test**  
 368

369 **Table 4 Key information of the 1<sup>st</sup> round capillary absorption tests for respective laboratories**

Lab No.	Sample size (cm)		Sealing method
	Surface	Height	
A	8×4	12	Non-adhesive plastic film
B	8×4	12	Non-adhesive plastic film
C	10×5*	21	Epoxy
D	8×4	12	Plastic film
E	10×5*	21	Paint
F	10×5*	21	Silicone paste
G	4×3	5	aluminium foil
H	8×4	12	No sealing
I	4×4	Unreported	Epoxy

\* Raw bricks were used.

370

371

372 As a matter of fact, the capillary absorption process has been extensively studied in the past decades  
373 and most impact factors have been thoroughly investigated. However, there is one factor remaining  
374 controversial: time correction. During the manual measurement the sample must be taken out of the  
375 water basin for weighing periodically, and no consensus has been reached concerning whether to  
376 correct this time interval. Normally 5-10 points are determined for calculating the capillary absorption  
377 coefficient, and with practice each weighing could be limited to 15-20 s, producing a total time of 2-3  
378 min without water contact at the bottom. Since capillary absorption tests normally take a couple of  
379 hours, time correction resultantly produces a larger capillary absorption coefficient by 2%-5% in most  
380 cases. This correction partly explains that Lab G obtained a slightly larger capillary absorption  
381 coefficient in the 2<sup>nd</sup> round (the manual method with time correction was used) when compared with  
382 their 1<sup>st</sup> round result (the automatic method was used, involving no time correction), and that Lab I  
383 reported the smallest capillary absorption coefficient in the 1<sup>st</sup> round (also the automatic method was  
384 used). However, overall there has been no decisive evidence supporting or against time correction,  
385 calling for further study.

### 386 **3.3 Cup tests**

387 Fig. 11 illustrates the vapor diffusion resistance factors obtained from the cup tests. Compared with  
388 the vacuum saturation test and the capillary absorption test, the scatters of the cup test between different  
389 laboratories are conspicuously larger in the 1<sup>st</sup> round. This trend can also be observed in the EC  
390 HAMSTAD project and other round robin campaigns. For our case, the smallest value bottoms at 6.0  
391 (Lab F) while the largest value goes up to 25.8 (Lab G), producing a factor 4.3 difference. This  
392 difference is too large to be solely attributed to the limited RH dependence of the resistance factor for  
393 the studied brick, and the inter-laboratory discrepancy should play the key role. In the 2<sup>nd</sup> round,  
394 notable improvement can be observed, as the results generally display a closer distribution. However,  
395 Lab F strangely gave an even worse result, as its reported value decreased from 6.0 in the 1<sup>st</sup> round to  
396 4.2 in the 2<sup>nd</sup> round at the same RH condition. One explanation is that both the air layer inside the cup  
397 and the masked edge of the sample were corrected in the 2<sup>nd</sup> round but uncorrected in the 1<sup>st</sup> round by  
398 Lab F. These corrections resultantly lead to a smaller resistance factor. Moreover, the sample thickness  
399 was reduced from 5 cm in the 1<sup>st</sup> round to 3 cm in the 2<sup>nd</sup> round for Lab F, making the influence of  
400 corrections more significant. Nevertheless, the large deviation of Lab F cannot be completely attributed  
401 to these reasons and a more profound factor must exist. One possibility is the sealing leakage, which  
402 typically causes underestimated resistance factors [46], especially for relatively non-permeable  
403 materials (as the ceramic brick in this campaign). For statistical analysis, the results of Lab F in the 2<sup>nd</sup>  
404 round are hence excluded.

405 It should be noted that different laboratories performed the cup test in this campaign at slightly  
406 different RH settings. It is therefore impossible to conduct statistical analysis strictly at the same RH.  
407 However, the ceramic brick used in this campaign is very weak in hygroscopicity (its equilibrium  
408 moisture content at RH 85% in the desiccator test is less than 0.05%  $\text{kg}\cdot\text{kg}^{-1}$ ). Thus, the liquid island  
409 effect should be limited, and it is reasonable to summarize the results into the dry cup group and the  
410 wet cup group, depending on whether the average RH in the test is below or above 50%. The  
411 independent t-tests comparing the 1<sup>st</sup> and the 2<sup>nd</sup> rounds provide p-values of 0.573 and 0.776 for dry  
412 cup and wet cup respectively, showing that the lab-averaged vapor diffusion resistance factors had no  
413 statistical difference in both rounds.

414 The calculated reproducibility errors are illustrated in Fig. 12, in comparison with the EC  
415 HAMSTAD project. As is clearly reflected, the 1<sup>st</sup> round results display almost the same



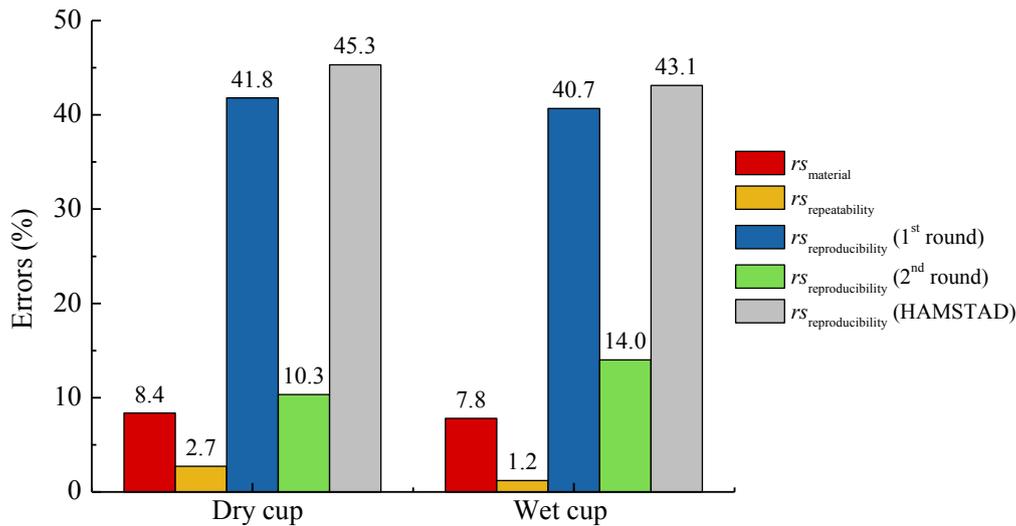


Fig. 12 Experimental errors of the round robin cup test

Table 5 Key information of the 1<sup>st</sup> round cup tests for respective laboratories

Lab No.	Sample size (cm)		Sealing method	Correction for		
	Surface	Thickness		Surface resistance	Air layer in the cup	Masked edge
A	Diameter = 8	3	Epoxy	No	Yes	No masked edge
B	10×10	1.7	Paraffin	No	No	No masked edge
C	Diameter = 7	3.5	Paraffin	No	No	No masked edge
D	Diameter = 6.4	2	Tape	No	Yes	No masked edge
E	20×20*	5	Paraffin and tape	Yes	Yes	Yes
F	Diameter = 8	5	Silicone paste	No	No	No
G	7×7	2.4	Paint and paraffin	No	Yes	No masked edge
I	Diameter = 9	3	Plasticine	No	No	Unreported

\* Combination of two raw bricks

The cup test is a classic and widely adopted method to determine the vapor permeability. As revealed by other round robin campaigns (such as those listed in Table 1), it is one of the most difficult tests to obtain similar results in different laboratories. Various factors can exert possible impacts. It is therefore important to specify these details to reduce the discrepancies between different laboratories. Table 5 summarizes the key information – sample size, sealing method and correction – of the cup tests in the 1<sup>st</sup> round. In comparison, all laboratories turned to a fixed thickness of 3 cm and similar surface areas in the 2<sup>nd</sup> round. The air layer and the masked edge (if any) were corrected, and the air velocity was also increased to minimize the surface resistance in the 2<sup>nd</sup> round.

Cup tests are normally performed around standard atmospheric pressure in different laboratories, so the limited air pressure fluctuation is unlikely to cause a large discrepancy [23, 47]. It has also been shown – both theoretically and experimentally – that temperature's influence is not strong [37], so the normal experimental temperature – usually around 20-25°C – is unlikely to be a main source of error. Moreover, the sample's surface area does not have a significant impact [23, 47], as also reflected in Table 5. As a result, these factors are not very strictly prescribed in the common protocol.

In addition to these weak impacts, three other factors are worth special attention. The first one is sample thickness, which should be evaluated together with the correction for the air layer inside the

456 cup and the surface resistance outside the samples. The  $s_d$  value for the air layer and surface resistance  
457 normally amounts to 3-5 cm in total. If the sample's  $s_d$  value is too small, then the correction of the air  
458 layer and surface resistance becomes very important. For this reason, the ISO 12572 standard [35]  
459 requires the sample's  $s_d$  value to be at least 10 cm. Moreover, the air velocity above the surface should  
460 be high enough and the air layer in the cup should be as thin as possible. Combining Fig. 11 a) and  
461 Table 5, it can be generalized that those laboratories with a small sample thickness (Labs B, D and G)  
462 tended to have larger resistance factors in the 1<sup>st</sup> round, in accordance with the aforementioned analysis.

463 The second important impact is sample sealing [22, 27, 46], which consists of the lateral coating of  
464 the sample and the fixing of the sample on the diffusion cup. If not handled properly, the lateral coating  
465 can cause sealant penetration deep into the sample, reducing the real cross-sectional area for vapor  
466 diffusion and finally leads to an overestimated resistance factor. On the contrary, imperfect sealing  
467 between the sample and the diffusion cup will lead to vapor leakage and resultantly an underestimation  
468 of the resistance factor. Obviously, the more impermeable the material is, the greater the impact of the  
469 sample sealing can be. Looking at Table 5, one can easily notice that Lab E combined two raw bricks  
470 for the test in the 1<sup>st</sup> round, and the results were almost the smallest. This may result from the imperfect  
471 sealing between the two raw bricks. Once switched to the sample cut from a single brick, the results  
472 from Lab E increased to the average value.

473 The last crucial factor is humidity control [25, 46]. In the cup test, desiccant and saturated salt  
474 solutions are most frequently used to create the desired RH inside and/or outside the cups. If the  
475 desiccant becomes wet or if the salt solution fails to remain saturated, the real RH will be higher than  
476 the assumed value. As a result, the vapor pressure gradient across the sample can be underestimated  
477 or overestimated, leading to deviating results. It is therefore important to handle the desiccant and  
478 saturated salt solutions very carefully.

479 In the 2<sup>nd</sup> round of experiments, we imposed stringent requirements concerning the aforementioned  
480 important impacts. As demonstrated by the results, the agreement between different laboratories  
481 achieved significant improvement with the common protocol. It is therefore necessary to pay special  
482 attention to these factors while carrying out cup tests.

#### 483 **4. Conclusions**

484 A round robin campaign aiming at the hygric properties of porous building materials has been  
485 launched. A ceramic brick was selected as the target material. The vacuum saturation test for the bulk  
486 density and open porosity, the capillary absorption test for the capillary absorption coefficient and  
487 capillary moisture content, and the cup test for the vapor permeability, were performed. Results from  
488 nine participating laboratories from different countries show that:

- 489 a. Different laboratories can obtain similar results from the vacuum saturation test and the capillary  
490 absorption test, even without a common protocol;
- 491 b. Compared with the EC HAMSTAD project, the inter-laboratory discrepancies for the capillary  
492 absorption coefficient in this round robin campaign are much smaller;
- 493 c. The state-of-the-art for the cup test remains frustrating. However, with a stringent common  
494 protocol prescribing important impact factors, it is possible to achieve relatively consistent cup test  
495 results between different laboratories;
- 496 d. A common protocol can possibly reduce the discrepancies between laboratories, but not the  
497 average result of all laboratories.

498 Finally, it should be kept in mind that although a common protocol may reduce inter-laboratory

499 discrepancies, it does not necessarily represent the preferred or recommended procedures of the  
500 individual participating laboratories.

501

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## Appendix A: The common protocols for the round robin campaign

This appendix gives the full descriptions of the common protocols imposed in the 2<sup>nd</sup> round of the round robin campaign. General requirements, as well as the detailed experimental procedures and data processing methods, are explained for the vacuum saturation test, the capillary absorption test and the cup test.

### 1. General requirements

- a) The ambient temperature for all measurements should be maintained within 20-25°C, with a fluctuation smaller than ±1°C;
- b) The surface 0.5-1 cm of the raw bricks should be removed for preparing samples, and at least 4 duplicates (without cracks observable by the naked eye) are required for each test;
- c) Measure sample dimensions with calipers reading 0.01 mm. For each dimension (length  $L$ , width  $W$ , thickness  $I$  and diameter  $D$ , m), measure at least twice at different locations and take the average;
- d) Samples should be dried in a ventilated oven at 70°C for at least 7 days. When 3 successive weighings (reading 0.01 g, but preferably 0.001 g) at intervals of at least 1 day show a relative fluctuation below 0.1%, stop the drying process and take the average as the dry mass  $m_{\text{dry}}$  (kg).

### 2. Vacuum saturation test

Samples should have a size of 8cm×4cm×1cm (can be cut from the capillary absorption samples after finishing that test).

#### 2.1 Experimental procedure

- a) Put dry samples in a vacuum container and evacuate the air inside. The air pressure in the container should stay below 3000 Pa for at least 4 h;
- b) Fill in distilled/deionized water into the container gradually. When the water level touches the bottom of samples, maintain a water level rise of around 5 cm/h (or slower) until all samples are completely submerged;
- c) Keep filling in water until the water level is 2 cm above the top of the sample. Then return the air pressure in the container to atmospheric pressure;
- d) After at least 24 h, weigh samples underwater and record the underwater mass  $m_{\text{under}}$  (kg);
- e) Take samples out of water and use a piece of moist paper/tissue/cloth to remove the liquid water on the surfaces. Then determine the wet mass  $m_{\text{wet}}$  (kg) in the air immediately;

#### 2.2 Data processing

- f) The open porosity  $\phi$  should be calculated by:

$$\phi = \frac{m_{\text{wet}} - m_{\text{dry}}}{m_{\text{wet}} - m_{\text{under}}} \quad (\text{A-1})$$

- g) The bulk density  $\rho_{\text{bulk}}$  (kg·m<sup>-3</sup>) should be calculated by:

$$\rho_{\text{bulk}} = \frac{m_{\text{dry}} \cdot \rho_{\text{water}}}{m_{\text{wet}} - m_{\text{under}}} \quad (\text{A-2})$$

where the water density ( $\rho_{\text{water}}$ , kg·m<sup>-3</sup>) should be taken according to the water temperature.

### 634 3. Capillary absorption test

635 Samples should have a size of 12cm×8cm×4cm, and one 8cm×4cm surface is used as the bottom to  
636 ensure the capillary absorption along the brick's longitudinal direction.

#### 637 3.1 Experimental procedure

- 638 a) Wrap dry samples with either plastic film or aluminum foil on all surfaces except for the bottom.  
639 Leave 1-2 small holes at the top to allow air evacuation. To avoid capillary uptake between the  
640 sample and the wrap, the bottom 1 cm of the lateral sides should be left unwrapped. Note that for  
641 this test the dry mass includes the wrap;
- 642 b) Pour distilled/deionized water (pre-conditioned to the ambient temperature) into a shallow basin,  
643 where a metal/plastic sample holder with a limited contact surface is placed. The water level in the  
644 basin should be 3-5 mm above the top of the sample holder;
- 645 c) Put the wrapped sample (cooled down to ambient temperature) on the sample holder softly. The  
646 moment the sample touches water, start the timer (reading 1 s);
- 647 d) At time 2, 4, 7, 10, 15, 20, 25, 30, 40, 50, 60, 80, 100, 120, 150, 180 and 210 min, take the sample  
648 out of water and use a piece of moist paper/tissue/cloth to remove the liquid water adhered to the  
649 bottom. Immediately weigh the sample for  $m(t)$  (kg) and put it back on the holder. The accumulated  
650 duration that the sample is not absorbing water from the basin (during the weighing process) should  
651 be corrected in the time  $t$  (s);

#### 652 3.2 Data processing

- 653 e) Plot  $[m(t)-m_{\text{dry}}]/(W \cdot I)$  against  $t^{0.5}$  and distinguish the 1<sup>st</sup> and 2<sup>nd</sup> stages of the capillary absorption  
654 process. There may be 1-2 points in the transition zone between the 1<sup>st</sup> and 2<sup>nd</sup> stages. Discard  
655 them;
- 656 f) Fit the data points in the 1<sup>st</sup> stage with the following linear equation:

$$657 \quad \frac{m(t)-m_{\text{dry}}}{W \cdot I} = A_{\text{cap}} \cdot t^{0.5} + c \quad (\text{A-3})$$

658 where the slope is defined as the capillary absorption coefficient ( $A_{\text{cap}}$ ,  $\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-0.5}$ );

- 659 g) Fit the data points in the 2<sup>nd</sup> stage linearly, and calculate its cross point with the fitted straight line  
660 for the 1<sup>st</sup> stage. The capillary moisture content ( $w_{\text{cap}}$ ,  $\text{kg} \cdot \text{m}^{-3}$ ) should be calculated according to  
661 the following equation:

$$662 \quad w_{\text{cap}} = \left. \frac{m(t)-m_{\text{dry}}}{L \cdot W \cdot I} \right|_{\text{cross point}} \quad (\text{A-4})$$

### 663 4. Cup test

664 Samples should have a thickness of 3 cm. If round samples are used, the diameter should be 8-10  
665 cm. For square samples 8-10 cm is required for the side length. The test should be carried out along  
666 the brick's thickness direction.

#### 667 4.1 Experimental procedure

- 668 a) Seal the sample on the opening of the diffusion cup. The sealant can be epoxy, paraffin or other  
669 vapor-tight methods. While sealing, try to minimize the penetration of the sealant into the sample;
- 670 b) Put diffusion cups with sealed samples in a chamber where the relative humidity is controlled.  
671 Ensure an air velocity of at least  $1 \text{ m} \cdot \text{s}^{-1}$  above the sample surface;
- 672 c) Inside the diffusion cup, humidity should be controlled by either saturated salt solutions or  
673 desiccant, while this can be achieved by saturated salt solution, HVAC system or other reliable

674 methods outside the diffusion cup. Choose between the following two options (the 1<sup>st</sup> is  
675 recommended):

676  
677

**Table A1 RH conditions for the cup test**

RH options	RH settings	Lower RH (%)	Higher RH (%)
1 <sup>st</sup>	Dry	0 or 11 (desiccant or LiCl)	54 (Mg(NO <sub>3</sub> ) <sub>2</sub> )
	Intermediate	54 (Mg(NO <sub>3</sub> ) <sub>2</sub> )	84 (KCl)
	Wet	84 (KCl)	94 (KNO <sub>3</sub> ) or 97 (K <sub>2</sub> SO <sub>4</sub> )
2 <sup>nd</sup>	Dry	0 or 11 (desiccant or LiCl)	54 (Mg(NO <sub>3</sub> ) <sub>2</sub> )
	Wet	54 (Mg(NO <sub>3</sub> ) <sub>2</sub> )	94 (KNO <sub>3</sub> ) or 97 (K <sub>2</sub> SO <sub>4</sub> )

678

- 679 d) After an initial period of 5-7 days for reaching steady-state, start weighing the diffusion cups for  
680  $m(t)$  (to 0.1 g, preferably to 0.01 g) every 2-4 days, until 7 successive weighings give excellent  
681 linear fitting results ( $R^2 > 0.99$ ). The time should be recorded to the single minute;  
682 e) Measure/estimate the thickness of the air layer ( $l_{\text{air}}$ , m) in the diffusion cup between the lower  
683 surface of the sample and the upper surface of the saturated solution (or desiccant) to 1 mm;

#### 684 4.2 Data processing

- 685 f) Fit the mass of the diffusion cup  $m(t)$  against the time  $t$  linearly. The slope should be denoted as  $\dot{G}$   
686 for the vapor flow rate ( $\text{kg}\cdot\text{s}^{-1}$ );  
687 g) Calculate the vapor flux ( $\dot{g}$ ,  $\text{kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ ) by:

$$688 \quad \dot{g} = \frac{\dot{G}}{A} \quad (\text{A-5})$$

689 where  $A$  is the sample's cross-sectional area ( $\text{m}^2$ ). In case a masked edge of the sample exists, the  
690 vapor flux should be corrected according to the ISO 12572 standard [35];

- 691 h) Calculate the total vapor diffusion resistance  $R_{\text{total}}$  ( $\text{m}^2\text{sPa}\cdot\text{kg}^{-1}$ ) by:

$$692 \quad R_{\text{total}} = \frac{\Delta p_v}{\dot{g}} \quad (\text{A-6})$$

693 where  $\Delta p_v$  (Pa) is the vapor pressure difference in and outside the diffusion cup, obtained based  
694 on the ambient temperature and the RH conditions;

- 695 i) Calculate the resistance of the air layer inside the diffusion cup  $R_{\text{air}}$  ( $\text{m}^2\text{sPa}\cdot\text{kg}^{-1}$ ) by:

$$696 \quad R_{\text{air}} = \frac{l_{\text{air}}}{\delta_{\text{air}}} \quad (\text{A-7})$$

697 where  $\delta_{\text{air}}$  is the vapor permeability of stagnant air. At 20-25°C, its value can be taken as  $2 \times 10^{-10}$   
698  $\text{kg}\cdot\text{m}^{-1}\cdot\text{s}^{-1}\cdot\text{Pa}^{-1}$ ;

- 699 j) Calculate the vapor diffusion resistance of the sample  $R_{\text{sample}}$  ( $\text{m}^2\text{sPa}\cdot\text{kg}^{-1}$ ) by:

$$700 \quad R_{\text{sample}} = R_{\text{total}} - R_{\text{air}} \quad (\text{A-8})$$

- 701 k) Calculate the vapor permeability  $\delta_{\text{sample}}$  ( $\text{kg}\cdot\text{m}^{-1}\cdot\text{s}^{-1}\cdot\text{Pa}^{-1}$ ) and resistance factor  $\mu$  of the sample by:

$$702 \quad \delta_{\text{sample}} = \frac{l}{R_{\text{sample}}} \quad (\text{A-9})$$

$$703 \quad \mu = \frac{\delta_{\text{air}}}{\delta_{\text{sample}}} \quad (\text{A-10})$$

- 704 l) Calculate  $\mu$  at different RH settings, and express the results against the average RH in and outside  
705 the diffusion cup.



707

## Appendix B: Experimental results from the round robin campaign

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709 This appendix provides detailed experimental results from the round robin campaign.

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**Table A-2 Results of the vacuum saturation tests**

Property	Lab	Round	Temp. (°C)	Results					Average*	
$\rho_{\text{bulk}}$ ( $\text{kg}\cdot\text{m}^{-3}$ )	A	1 <sup>st</sup>	19.9	1879	1880	1878	1880	1884	<b>1880 (2)</b>	
		2 <sup>nd</sup>	19.9	1879	1880	1878	1880	1884	<b>1880 (2)</b>	
	B	1 <sup>st</sup>	Unreported	1909	1903	1912	1909	1903	<b>1907 (4)</b>	
		2 <sup>nd</sup>	21.4	1880	1885	1856	1902	1902	1899	<b>1887 (18)</b>
	C	1 <sup>st</sup>	23	1874	1931	1935			<b>1913 (34)</b>	
		2 <sup>nd</sup>								
	D	1 <sup>st</sup>	Unreported	1907	1884	1898	1903	1879	1875	<b>1891 (13)</b>
		2 <sup>nd</sup>								
	F	1 <sup>st</sup>								
		2 <sup>nd</sup>	23	1778	1818	1837	1794		<b>1807 (26)</b>	
H	1 <sup>st</sup>	Unreported	1856	1854	1853	1848	1859	1850	<b>1853 (4)</b>	
	2 <sup>nd</sup>	25	1862	1853	1859	1853		<b>1857(5)</b>		
$\phi$ (%)	A	1 <sup>st</sup>	19.9	31.7	31.8	31.7	31.9	31.6	<b>31.7 (0.1)</b>	
		2 <sup>nd</sup>	19.9	31.7	31.8	31.7	31.9	31.6	<b>31.7 (0.1)</b>	
	B	1 <sup>st</sup>	Unreported	28.5	28.7	29.0	28.7	28.9	<b>28.8 (0.2)</b>	
		2 <sup>nd</sup>	21.4	27.5	27.4	29.2	28.1	27.6	28.0	<b>27.9 (0.7)</b>
	C	1 <sup>st</sup>	23	32.0	32.8	33.0			<b>32.6 (0.5)</b>	
		2 <sup>nd</sup>								
	D	1 <sup>st</sup>	Unreported	30.4	31.4	30.9	30.7	31.7	31.8	<b>31.2 (0.6)</b>
		2 <sup>nd</sup>								
	F	1 <sup>st</sup>								
		2 <sup>nd</sup>	23	25.0	24.0	23.0	25.0		<b>24.3 (1.0)</b>	
H	1 <sup>st</sup>	Unreported	32.7	32.5	32.2	32.6	32.7	32.5	<b>32.5 (0.2)</b>	
	2 <sup>nd</sup>	25	32.2	32.3	32.2	32.7		<b>32.4 (0.2)</b>		

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\* Data in parenthesis are standard deviations.

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**Table A-3 Results of the capillary absorption tests**

Property	Lab	Round	Temp. (°C)	Results*						Average**
$A_{cap}$ ( $\text{kg}\cdot\text{m}^{-2}\text{s}^{-0.5}$ )	A	1 <sup>st</sup>	20.1	0.487	0.500	0.508	0.491	0.502		<b>0.497 (0.009)</b>
		2 <sup>nd</sup>	20.1	0.487	0.500	0.508	0.491	0.502		<b>0.497 (0.009)</b>
	B	1 <sup>st</sup>	23	0.587	0.604	0.588	0.527	0.557	0.563	<b>0.571 (0.028)</b>
		2 <sup>nd</sup>	20.5	0.463	0.471	0.524***	0.455	0.451	0.473	<b>0.462 (0.010)</b>
	C	1 <sup>st</sup>	23	0.49	0.53	0.48	0.44	0.48	0.51	<b>0.488 (0.031)</b>
		2 <sup>nd</sup>								
	D	1 <sup>st</sup>	19.8	0.505	0.477	0.478				<b>0.486 (0.016)</b>
		2 <sup>nd</sup>								
	E	1 <sup>st</sup>	23	0.491	0.449	0.452	0.433	0.433		<b>0.452 (0.024)</b>
		2 <sup>nd</sup>	23	0.600	0.601	0.610	0.594	0.633		<b>0.608 (0.015)</b>
	F	1 <sup>st</sup>	20	0.456	0.460	0.400				<b>0.439 (0.033)</b>
		2 <sup>nd</sup>	23	0.536	0.506	0.521	0.516			<b>0.520 (0.013)</b>
	G	1 <sup>st</sup>	23	0.438	0.461	0.401	0.418	0.443	0.513	<b>0.446 (0.039)</b>
		2 <sup>nd</sup>	23	0.459	0.495	0.478	0.500			<b>0.483 (0.019)</b>
	H	1 <sup>st</sup>	25	0.500	0.508	0.524	0.494			<b>0.507 (0.013)</b>
		2 <sup>nd</sup>	25	0.513	0.569	0.510	0.543			<b>0.534 (0.028)</b>
I	1 <sup>st</sup>	Unreported	0.373	0.443	0.490	0.468	0.298***	0.374	<b>0.430 (0.054)</b>	
	2 <sup>nd</sup>									
$W_{cap}$ ( $\text{kg}\cdot\text{m}^{-3}$ )	A	1 <sup>st</sup>	20.1	209.5	208.1	206.4	212.1	212.1		<b>209.6 (2.5)</b>
		2 <sup>nd</sup>	20.1	209.5	208.1	206.4	212.1	212.1		<b>209.6 (2.5)</b>
	B	1 <sup>st</sup>	23	192.1	172.6	175.1	169.6	189.9	174.8	<b>179.0 (9.5)</b>
		2 <sup>nd</sup>	20.5	170.7	168.4	184.9	170.5	168.7	175.5	<b>173.1 (6.3)</b>
	C	1 <sup>st</sup>	23	182.4	178.6	180.5	178.6	178.6	182.4	<b>180.2 (1.8)</b>
		2 <sup>nd</sup>								
	D	1 <sup>st</sup>	19.8	204.4	197.0	199.8				<b>200.4 (3.7)</b>
		2 <sup>nd</sup>								
	E	1 <sup>st</sup>								
		2 <sup>nd</sup>	23	189.1	183.7	182.1	181.9	190.2		<b>185.4 (4.0)</b>
	F	1 <sup>st</sup>								
		2 <sup>nd</sup>	23	197	193	174	193			<b>189.3 (10.3)</b>
	G	1 <sup>st</sup>	23	164.7	172.8	157.3	160.7	164.2	161.4	<b>163.5 (5.3)</b>
		2 <sup>nd</sup>	23	169.8	184.3	184.1	190.5			<b>182.2 (8.8)</b>
	H	1 <sup>st</sup>	25	209.4	204.9	210.7	208.6			<b>208.4 (2.5)</b>
		2 <sup>nd</sup>	25	209.8	206.3	208.4	210.2			<b>208.7 (1.8)</b>

717 \* Original values, temperature dependence uncorrected;

718 \*\* Data in parenthesis are standard deviations;

719 \*\*\* Outlier, discarded.

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**Table A-4 Results of the cup tests**

Round	Lab	Temp. (°C)	RH (%)		$\mu$				Average*	
1 <sup>st</sup>	A	23.1	11.3-53.5	11.3	11.3	12.3	11.6		<b>11.6 (0.5)</b>	
			53.5-84.7	10.6	10.5	10.6	12.6		<b>11.1 (1.0)</b>	
			84.7-97.4	9.6	10.1	10.7	10.4		<b>10.2 (0.5)</b>	
	B	23	0-50	17.1	17.1	22.1**	15.8	15.2	<b>16.3 (1.0)</b>	
	C	23	50-94	15.5	15.0	13.3	16.5	16.5	15.6	<b>15.4 (1.2)</b>
	D	19.8	54-75	20.3	25.9**	19.0	19.8	19.7		<b>19.7 (0.5)</b>
			75-95	14.4	9.4**	16.5	15.2	15.1		<b>15.3 (0.8)</b>
	E	23	0-50	10.0	9.2					<b>9.6 (0.6)</b>
			50-93	5.0	9.8					<b>7.4 (3.4)</b>
	F	23	50-95.5	6.2	6.4	5.9	5.6	6.0		<b>6.0 (0.3)</b>
G	22.7	1-36	23.8	25.2	28.3	27.1	25.1	25.2	<b>25.8 (1.6)</b>	
I	23	0-50	13.2	13.2	12.5	12.3			<b>12.8 (0.5)</b>	
		50-94	6.3	7.6	8.3	5.2			<b>6.8 (1.4)</b>	
2 <sup>nd</sup>	A	23.1	11.3-53.5	11.3	11.3	12.3	11.6			<b>11.6 (0.5)</b>
			53.5-84.7	10.6	10.5	10.6	12.6			<b>11.1 (1.0)</b>
			84.7-97.4	9.6	10.1	10.7	10.4			<b>10.2 (0.5)</b>
	B	20	0-52	12.5	13.0	10.8	14.0	12.0	13.7	<b>12.7 (1.2)</b>
			52-85	12.1	11.0	10.0	8.7	8.3	9.1	<b>9.9 (1.5)</b>
			85-97	10.1	8.7	10.2	9.5	7.8	7.7	<b>9.0 (1.1)</b>
			0-50	14.7	14.4	14.3				<b>14.5 (0.2)</b>
	E	23	50-93	13.8	14.1	14.5				<b>14.1 (0.4)</b>
			54-94	3.5	5.1	4.7	3.5	4.3		<b>4.2 (0.7)</b>
	F	23	54-94	3.5	5.1	4.7	3.5	4.3		<b>4.2 (0.7)</b>
G	23	80.3-96	10.1	11.2	11.8	11.1			<b>11.0 (0.7)</b>	

\* Data in parenthesis are standard deviations;

\*\* Outlier, discarded.