

# On the importance of desiccant during the determination of water vapor permeability of permeable insulation material

Thibaut Colinart<sup>1,\*</sup>, and Patrick Glouannec<sup>1</sup>

<sup>1</sup>Univ Bretagne Sud, UMR CNRS 6027, IRDL, 56100 Lorient, France

**Abstract.** Water vapor permeability of building materials is usually measured using dry cup test according to the ISO 12572 standard. For this test, suitable adsorbing desiccant should be used to provide stable low vapor pressure conditions within the cup and, thus, to ensure the good accuracy of the measurement. In this work, different adsorbing desiccants mentioned in the ISO 12572 standard are tested for measurements performed on wood fiber insulation. For each experiment, relative humidity is monitored inside the dry cup. The results indicate that 0 %RH is not reached inside the dry cup and boundary condition is not always stable for highly permeable construction materials, depending on the adsorbing desiccants. The impact of these observation is evaluated on the determination of water vapor diffusion resistance factor and compared to other sources of uncertainties.

## 1 Introduction

Water vapor permeability is one of the required properties when performing hygrothermal simulation of building envelopes. This property is mainly determined by means of cup test since the 1950's. Broadly speaking, this test consists in measuring under steady state and isothermal condition the moisture flow due to relative humidity gradient across a sample sealed on a cup containing desiccant or saturated salt solutions. The detailed protocol of the measurement is given in the latest version of the standards ISO 12572 [1] or ASTM E96 [2]. Cup test is found to be repeatable and reproducible [3] and impact of time and personnel is limited [4]. Despite the simplicity of the methods, numerous round robin tests [5-8] showed large inter-laboratory discrepancies. For instance, results obtained in the frame of HAMSTAD project [6] or recently by Janssen et al. [5] showed that the highest water diffusion resistance factor for ceramic brick is 4.5 times higher than the lowest one. Possible factors explaining these discrepancies are discussed hereafter.

The first ones concern the design of the experiment. For instance, specimens can be larger than the cup creating 2D vapor flow within the material. Even if the standard recommends correcting this masked edge effect, its impact is found to be rather limited on the results [7-9]. On the contrary, sealing is of high importance [10-12], especially for vapor tight materials [7]. Indeed, a faulty seal results in vapor leakage and, thus, higher flow rate. For porous building materials, molten sealants may penetrate far enough to introduce errors into the effective area under test [1].

The second ones concern the importance of surface air resistances compared material resistance, defined as

the ratio between the sample thickness and its water vapor permeability. From this definition, the lower the thickness, the lower the material resistance, the higher the influence of surface air resistance. Surface air resistance within the cup is the one due to vapor diffusion across still air layer between the sample and the desiccant. To limit its influence, standard [1] recommends air layer thickness of  $15 \pm 5$  mm. Nevertheless, applying correction is clearly necessary for permeable materials [9]. Above the cup, surface air resistance is function of local air velocity. Its influence is significant for permeable sample [13-15]. For instance, a mean deviation factor of 10 has been observed in [14] for wood fiber insulation. Although the standard [1] states that air velocity should be of at least  $2 \text{ m}\cdot\text{s}^{-1}$  above each specimen, this recommendation is in fact solely respected and evaluating the surface air resistance is still challenging [16].

Last, the inadequate control of ambient conditions, i.e. barometric pressure, temperature and relative humidity, can be a source of error. Even if the water vapor permeability may change with barometric pressure or temperature [17-19], their variations are rather limited during the experiments and their impact on the measurement can be assumed as negligible. The relative humidity outside the cup is usually controlled by climatic chamber or saturated salt solutions and do not vary to large extend. On the contrary, the accurate control of relative humidity inside the cup is challenging. To produce low relative humidity conditions inside the cup in case of dry cup experiment, it is recommended to use desiccants like calcium chloride  $\text{CaCl}_2$  [6-8, 20], Silica Gel [7, 15, 17] or Magnesium perchlorate  $\text{Mg}(\text{ClO}_4)_2$  [7]. Prior to experiment, they must be pre-dried to ensure high moisture adsorption capacity:

\* Corresponding author: [thibaut.colinart@univ-ubs.fr](mailto:thibaut.colinart@univ-ubs.fr)

200 °C for CaCl<sub>2</sub> [21] or 120°C for silica gel [22]. Nevertheless, some studies show an increase in measured relative humidity from 3 to 11 %RH [15, 21, 23] at the onset of the test. Furthermore, as moisture flows to the cup, the absorptive power of desiccants decreases leading to an increase of relative humidity within the cup during the experiment [6, 7, 15, 21, 23, 24]. For instance, Pazera and Salonvaara [23] report an increase of 7 %RH over 24h for permeable materials. Two consequences result from these observations. First, using 0 %RH in the cup in the measurement analysis seems not to be appropriate. Already in the 1950's, Chang and Hutcheon recommended to use of 5%RH as initial condition at the surface of calcium chloride. Second, systematic rise of the relative humidity inside the cup prevents a steady state from being achieved. Other authors use rather saturated salt solutions like LiCl [3, 18] or NaOH [13-14], even if manipulating cups with liquid can be tricky during weighing.

This review underlines the difficulty in measuring water vapor permeability of permeable materials, due to the large influence of surface air resistance and to the inadequate control of ambient conditions. This work aims to further investigate the second point by monitoring relative humidity control inside the cup during various water vapor permeability measurements of wood fiber insulation.

## 2 Materials and methods

### 2.1 Tested material

The tested material is wood fiber insulation with a thickness of 40 mm (Pavaflex Confort, supplied by Pavatex). It has a density of  $\rho = 50 \text{ kg.m}^{-3}$  and a low thermal conductivity ( $\lambda = 0.038 \text{ W.m}^{-1}.\text{K}^{-1}$ ). Therefore, this bio-based material is increasingly used in building envelope like wooden frame buildings. Nevertheless, it is strongly hygroscopic as shown in Fig. 1 [25]. Furthermore, product datasheet indicates a water vapor diffusion resistance factor of 2.

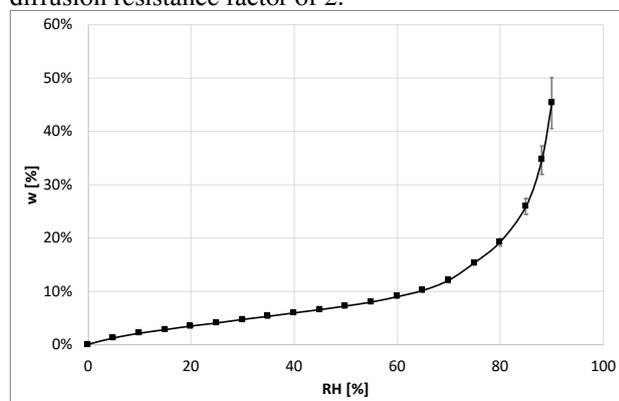


Fig. 1. Sorption isotherm of wood fiber insulation.

### 2.2 Dry cup method

Water vapor permeability is measured using dry cup method according to the ISO 12572 standard [1].

Samples with dimensions 100 x 100 x 40 mm<sup>3</sup> are tested. Prior to testing, they are conditioned at 23 °C and 50 %RH according to EN 13171 standard. They are then sealed to cups containing desiccant. Here, the cups with dimensions 100 x 100 x 60 mm<sup>3</sup> are made of PE and a vapor-tight aluminum tape was used to seal the sides of the sample with the side of the cup. The vapor tightness of this assembly was verified through a test on impermeable PVC.

Five desiccants are tested in this work: 5-years old silica gel (regenerated several times at temperatures ranging between 80 °C and 140 °C), new silica gel (regenerated one time at 120 °C), calcium chloride CaCl<sub>2</sub> powder (not regenerated prior to the experiment), phosphorus pentoxide P<sub>2</sub>O<sub>5</sub> powder (used as it) and potassium hydroxide KOH (partially saturated). Whatever the desiccant, the thickness of air layer between specimen and desiccant is always in the range  $17 \pm 3 \text{ mm}$ . Temperature and relative humidity in this air layer are monitored with an accuracy of 2 %RH and 0.5 °C with HygroPuce (Waranet, Auch, France). This sensor is 0.6 cm thick and 1.6 cm in diameter and wireless: it is therefore well adapted for such measurement, preventing vapor leakage through wire connection. For solid desiccants, sensor is placed either on desiccant surface or on a holder glued to the cup. For KOH solution, sensor is rather attached to specimen to prevent any damage due to liquid. Furthermore, special attention should be paid when handling the cup. For experiments performed with 5-years old silica gel, T and RH is also monitored at the bottom of the cup and above the sample.

Assemblies are placed in a climatic chamber (Mettmert HPP 108, Schwabach, Germany) at 23 °C and 50 %RH. Air velocity measured above the cup with a hot-wire anemometer is around  $0.15 \pm 0.05 \text{ m.s}^{-1}$ . Cups are removed punctually from the chamber to be weighed using two balances: Adventurer Pro AV4102C with a precision of 0.01 g and Explorer Pro EP613C with a precision of 0.001 g from Ohaus (Ohaus Corporation, Pine Brook, NJ, USA). According to the annex I of the standard [1], the minimal weighing interval should be respectively 20h and 2h to get results with an accuracy of 5% (assuming water vapor diffusion resistance factor  $\mu$  of 5 and theoretical vapor pressure difference  $\Delta p_v$  of 1400 Pa). The measurements are performed until water vapor flow rate  $g$  reaches steady state (as specified in section 8.1 of the standard [1]) or until the assembly has gained more than 1.5 g per 25 ml of desiccant in the cup. For this set-up, it corresponds to a mass gain of about  $2.5 \text{ kg.m}^{-2}$ .

This value may be compared to the weight loss of the sample: assuming that sample mean relative humidity drops from 50 to 25 %RH, mean moisture content decreases from 7.2 to 4.02 %, which corresponds to a weight loss of  $0.064 \text{ kg.m}^{-2}$ . Therefore, mass variations due to change of sample hygroscopicity are negligible compared to the ones expected during experiment.

## 3 Results and discussion

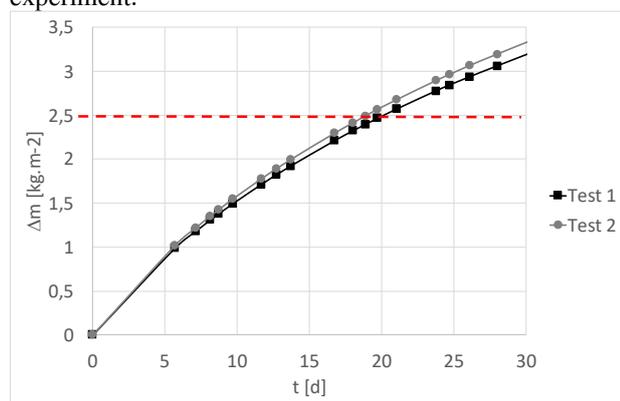
### 3.1 Preliminary results obtained with 5-years old silica gel

A first experiment, repeated twice, is done with 5-years old silica gel used as desiccant. Weight is measured once a day with the 0.01 g precision balance. Its evolution is plotted in Fig. 2, while water vapor flow rate variations are plotted in Fig. 3. T and RH are monitored every 20 min. Their variations are plotted in Fig. 4.

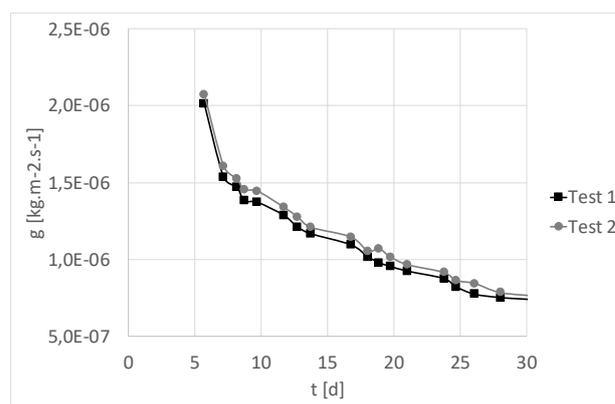
First, let note the good repeatability of mass, T or RH for the two measurements. As the experiment proceeds, moisture flows through the sample and mass increases. Here, mass gain of about  $2.5 \text{ kg.m}^{-2}$  is reached after  $19 \pm 1$  days.

Prior to the experiment, sensor provides relative humidity of 0 %RH and temperature of  $23 \text{ }^\circ\text{C}$  everywhere in the cup sealed with impermeable material. Right after sealing the wood fiber insulation sample to the cup, relative humidity reached already a value of  $9 \pm 1$  %RH in the air layer, while 0 %RH is still observed at the bottom of the cup. Moisture provided by exterior air and sample is not instantaneously and totally adsorbed by the desiccant. Simultaneously, we observe a significant temperature peak up to  $32 \text{ }^\circ\text{C}$  due to moisture adsorption by desiccant. After 24 h, relative humidity in the air layer has increased by 10 %RH, which is slightly higher than the observation of Pazera and Salonvaara [23]. After 4 to 5 days, relative humidity starts to increase at the bottom of the cup: moisture is transferred through the porous bed of desiccant. After 5 days, relative humidity increases slowly in the cup and temperature is equal to  $23 \text{ }^\circ\text{C}$ . After 28 days, relative humidity in the air layer reached a value of 42 %RH.

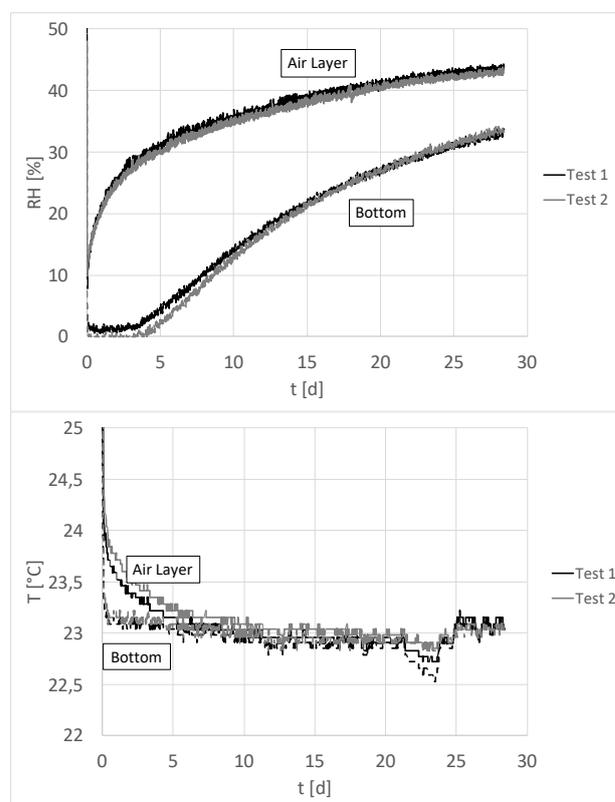
Finally, because of relative humidity increase in the air layer, vapor pressure gradient across the sample decreases. Consequently, water vapor flow rate is reduced by a factor 2 (see Fig. 3). Particularly, steady state could not be reached for this experiment: the smallest deviation from the mean of 5 consecutive measurements is 9 %, observed at the beginning of the experiment.



**Fig. 2.** Mass variations of dry cup containing 5-years old silica gel.



**Fig. 3.** Water vapor flow rate variations through the dry cup containing 5-years old silica gel.



**Fig. 4.** Relative humidity and temperature variations within the air layer and at the bottom of the cup containing 5-years old silica gel.

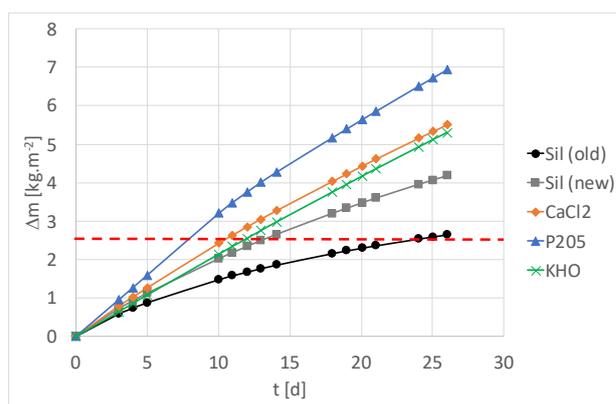
### 3.2 Comparison of desiccant

Further experiments are performed with the five desiccants. Results are shown in Fig. 5 to 7. For sake of clarity, only one curve per desiccant is plotted. First, note that the 5-years old silica gel behaves as in the preliminary experiment, showing the reproducibility of the method. New silica gel presents globally a similar behavior than old one. Nevertheless, because of its higher adsorption capacity, the mass increase is faster (mass gain of about  $2.5 \text{ kg.m}^{-2}$  is reached after 13 days) and relative humidity in the air layer is lower (39 %RH after 26 days). However, steady state is still not reached for new silica gel.

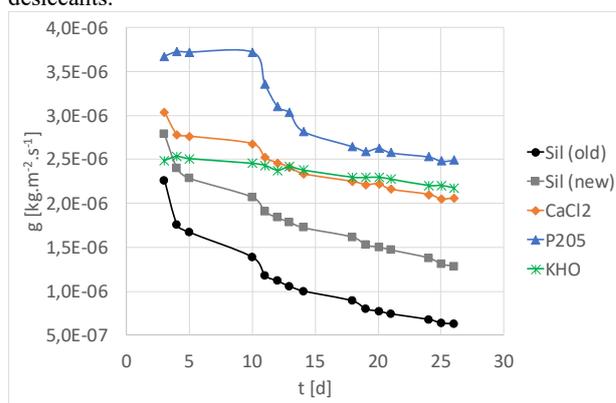
Besides, calcium chloride  $\text{CaCl}_2$  powder is the next desiccant in terms of adsorption capacity: mass gain of about  $2.5 \text{ kg}\cdot\text{m}^{-2}$  is reached after 10 days and relative humidity in the air layer reached 34 %RH after 26 days. Temperature behaves as for silica gel. The decrease in water vapor flow rate is smaller: steady state could be reached quite from the beginning, the highest deviation from the mean 5 consecutive measurements is 5 %. Note that a 2 mm thick rigid layer formed at the upper surface of desiccant was observed at the end of experiment.

Phosphorus pentoxide  $\text{P}_2\text{O}_5$  powder present a two-stage behavior that is different than previous desiccants. During the 10 first days, mass increase is rapid, resulting in much higher temperature in the air layer (up to  $25^\circ\text{C}$  after one week). Nevertheless, relative humidity remains almost constant and equal to 10 %RH. Consequently, water vapor flow rate is also constant. Then, relative humidity increases sharply for 5 days to 23 %RH. Due to the decrease in vapor pressure gradient across the sample, mass increase slows down and water vapor flow rate decreases. Temperature reaches rapidly the ambient value of  $23^\circ\text{C}$ . At the end of experiment, powder has changed to liquid and air layer increased from 20 to 37 mm: phase change occurred probably around the 10<sup>th</sup> day of experiment. Here, steady state is reached before the 10<sup>th</sup> day or after the 15<sup>th</sup> day of experiment.

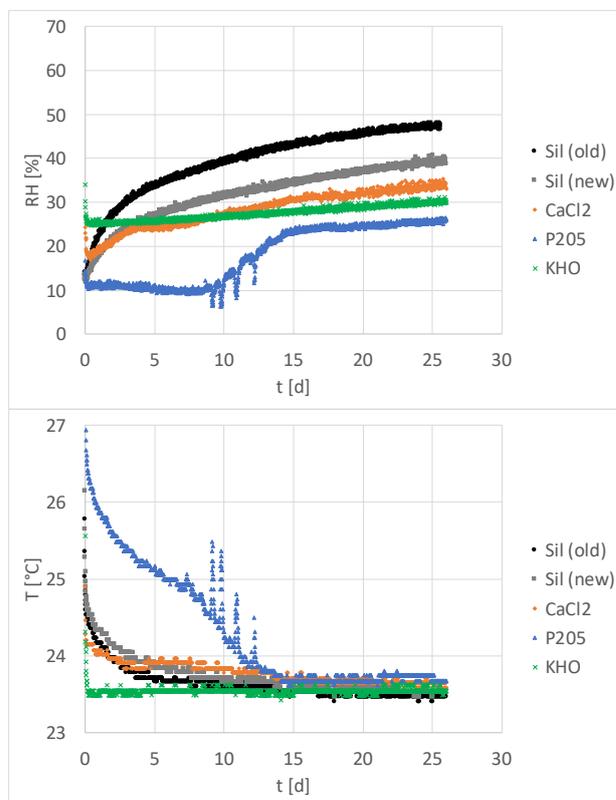
Last, mass increase is almost linear for potassium hydroxide KOH, as highlight by the slight decrease in water vapor flow rate. Obviously, steady state is reached from the beginning. In the present case, temperature is quite stable and relative humidity increases from 25 to 30 %RH. These values are much higher than the theoretical value of 8 %RH, due to the facts that the solution was not fully saturated, and that relative humidity is measured at the upper side of air layer. Nevertheless, (saturated) salt solution seems to provide the most stable conditions within the cup. At the end of experiment, we observed however that moisture has dissolved salt crystals and that air layer was reduced by 2 to 3 mm.



**Fig. 5.** Mass variations of dry cup containing different desiccants.



**Fig. 6.** Water vapor flow rate variations through the dry cup containing different desiccants.



**Fig. 7.** Relative humidity and temperature variations within the air layer of the cup containing different desiccants.

Water vapor diffusion resistance factor  $\mu$  is then calculated following four different approaches. First, relative humidity in the cup is set to a theoretical value of 5 %RH (leading to vapor pressure difference of 1257 Pa across the sample). If considered, air surface resistance in the cup is evaluated as specified in the Annex G of the standard (considering the entire air thickness whatever the case), while a resistance of  $3.5 \cdot 10^7 \text{ Pa}\cdot\text{kg}^{-1}\cdot\text{m}^2\cdot\text{s}^{-1}$  is assumed above the sample [16]. Then, relative humidity in the cup is set to the mean measured value over a period for which water vapor flow rate is the most stable. If considered, air surface resistance in the cup is evaluated considering the air thickness between sample and sensor. Results are sum up in Table 1.

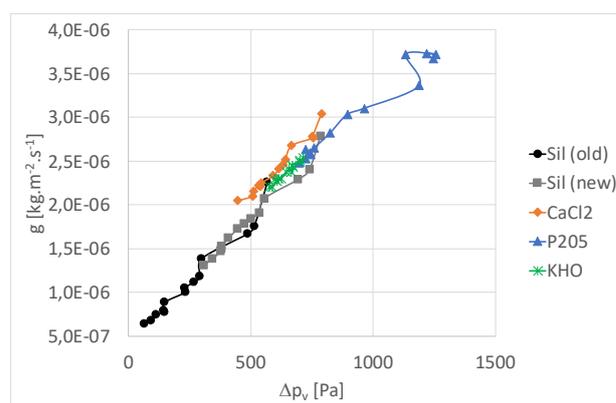
**Table 1.** Water vapor diffusion resistance factor  $\mu$  calculated following four different approaches.

	Sil. Gel (old)	Sil. Gel (new)	CaCl <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	KOH
Period [d]	18-24	11-18	18-24	18-24	10-14
Steady state	No	No	Yes	Yes	Yes
Mean g [mg.m <sup>-2</sup> .s <sup>-1</sup> ]	0.72	1.68	2.15	2.57	2.40
$\mu$ (Th. RH No Surf. Res.)	8.50	3.66	2.86	2.39	2.56
$\mu$ (Th. RH Surf. Res.)	7.91	3.14	2.19	1.72	1.94
Meas. RH [%]	45.5	33.7	31.9	24.7	27.1
Meas. $\Delta p_v$ [Pa]	128	472	525	738	661
$\mu$ (Meas. RH No Surf. Res.)	0.87	1.37	1.2	1.41	1.35
$\mu$ (Meas. RH Surf. Res.)	0.4	0.98	0.65	1.06	1

First, the measurements confirm that wood fiber insulation is very permeable. 5-years old silica gel gives results much different than other desiccant due to the low water vapor flow rate and the small vapor pressure gradient across the sample. These results are not further analyzed. When theoretical relative humidity is considered, deviations up to 1.41 are observed between the highest and the lowest value. These deviations are reduced to 0.33 when considering measured relative humidity. In comparison, applying a correction for surface air resistance leads to a reduction of  $0.6 \pm 0.07$  of water vapor diffusion resistance factor. These results indicate that assessing to the accurate conditions in the cup is of higher importance than correcting the surface air resistance. When both are considered, we note however that calculated factors may drop below unity. It should mean that water vapor diffusion is faster in the

material than in the air. Even if supposed “enhanced diffusion” phenomenon can be found in the literature [26-27], it does not apply to fibrous materials like wood fiber insulation at such low moisture content. Here, we rather think that the usual methodology proposed in the standard do not apply well for permeable materials.

To overcome this limitation, we propose a new analysis of the measurement. Fig. 8 presents the water vapor flow rate variations as function of measured vapor pressure difference across the sample. Whatever the desiccant, a rather linear behavior is observed, the slope of the curve providing directly the permeance. Water vapor diffusion resistance factors evaluated through this method are sum-up in Table 2. We note first that all calculated factors are larger than unity, even by accounting for surface air resistance. For solid desiccant, the permeance can be evaluated for a range of vapor pressure difference across the sample of at least 300 Pa. Calculated  $\mu$  tend to increase with mean relative humidity of the sample in accordance with literature [14]. Regarding potassium hydroxide KOH, the calculated value is much higher than others. Here, the slope is evaluated over a range of vapor pressure difference across the sample of only 139 Pa, which may be too small to reach a sufficient accuracy.



**Fig. 8.** Water vapor flow rate variations as function of measured vapor pressure difference across the sample.

**Table 2.** Water vapor diffusion resistance factor  $\mu$  calculated following the proposed new approach.

	Sil. Gel (old)	Sil. Gel (new)	CaCl <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	KOH
$\Delta p_v$ range [Pa]	65-513	306-739	443-752	696-1187	574-713
Mean sample RH [%]	46.0	41.9	39.8	36	38.9
$\mu$ (No Surf. Res.)	1.77	1.9	2.65	2.62	6.24
$\mu$ (Surf. Res.)	1.30	1.50	2.10	2.28	5.9

### 3.3 Influence of balance precision

A further experiment is done with Silica gel (old and new) and  $\text{CaCl}_2$  by using the 0.001 g precision balance. Weight measurements are made twice a day. As expected, mass, relative humidity and temperature variations are very similar to the ones presented in Fig. 5 and 7. However, increasing weight measurement frequency is not sufficient for reaching steady state when silica gels are used. On the other hand, more data are available which is interesting for evaluating more accurately the permeance as in Fig. 8.

### 4 Discussion and conclusions

A set of dry cup experiments has been performed on wood fiber insulation with a focus on the influence of desiccant. For all experiments, temperature and relative humidity has been monitored in the air layer between the sample and the desiccant, in the view of evaluating the dryness within the cup. In hermetically closed environment, all solid desiccants are able to maintain dry conditions, the sensor indicating 0 %RH. At the beginning of dry cup experiment, dry conditions are never reached in the cup: initial measured relative humidity varies between 10 and 17 %RH. Therefore, using 0 %RH (or even 5 %RH) in the analysis is not correct. Besides, because of the exothermal adsorption process, measured temperature in the air layer can be up to 10 °C higher than ambient temperature. Isothermal conditions are not respected at the beginning and may take between 3 and 10 days to be reached. Nevertheless, influence of temperature on water vapor permeability remains slight for this range of variation. As experiment proceeds and moisture is adsorbed by the desiccant, relative humidity in the air layer and, thus, water vapor flow rate is not constant. Consequently, steady state is not necessary reached, avoiding a proper estimation of water vapor permeability as specified in the standard ISO 12572 [1]. Particularly, water vapor diffusion resistance factor may drop below unity, which has no physical meaning in the range of investigated relative humidity. Note furthermore that making measurement with a higher frequency on higher precision balance do not improve the possibility of reaching steady state. As alternative, a pseudo steady state analysis is proposed: since a linear behavior is observed between water vapor flow rate and measured vapor pressure difference across the sample, the permeance can be directly evaluated from the slope of the curve. Estimated water vapor diffusion resistance factors seem to be reliable and present a usual evolution with materials relative humidity.

In conclusion, using solid desiccant only is not recommended when measuring water vapor permeability of permeable building materials with dry cup experiment since it leads to conditions that are much different than the ones expected in the standard. Nevertheless, two alternatives can be considered. First, wireless sensor can be used to monitor conditions in the air layer and then to evaluate directly the permeance. Second, the use of

saturated salt solutions seems to provide more stable relative humidity and temperature. These conclusions and recommendation have to be generalized for other building materials (more or less permeable, more or less hygroscopic) in light of other influencing parameters like air velocity.

### References

1. ISO 12572, Hygrothermal performance of building materials and products - Determination of water vapour transmission properties-Cup Method (2016)
2. ASTM E96, Standard test methods for water vapor transmission of materials (2016)
3. C. Feng, H. Janssen, Y. Feng, Meng, Q. Building and Environment, **85**, 160 (2015)
4. C. Feng, J. Todorović, H. Janssen, Energy Procedia, **132**, 291 (2017)
5. C. Feng, A.S. Guimarães, N. Ramos, L. Sun, D. Gawin, P. Konca, J. Zhao, J. Grunewald, K.K. Hansen, C. Hall, M. Fredriksson, Z. Pavlík, H. Janssen, MATEC Web of Conferences, **282**, 02011 (2019)
6. S. Roels, J. Carmeliet, H. Hens, O. Adan, H. Brocken, R. Cerny, Z. Pavlík, C. Hall, K. Kumaran, L. Pel, R. Plagge, Journal of thermal envelope and building science, **27**, 307 (2004)
7. S. Roels, P. Talukdar, C. James, C.J. Simonson, International Journal of Heat and Mass Transfer, **53**, 5355 (2010)
8. G.H. Galbraith, R.C. McLean, Z. Tao, N. Kang, Building Research and Information, **20**, 364 (1992)
9. P. Mukhopadhyaya, K. Kumaran, J. Lackey, D. van Reenen, In Heat-Air-Moisture Transport: Measurements on Building Materials. ASTM International (2007)
10. M. Bomberg, In Water Vapor Transmission Through Building Materials and Systems: Mechanisms and Measurement. ASTM International (1989)
11. S.L. Zelinka, S.V. Glass, C.R. Boardman, Journal of Testing and Evaluation, **44**, 2396 (2016)
12. J. Richter, K. Staněk, Procedia Engineering, **151**, 277 (2016)
13. O. Vololonirina, B. Perrin, Construction and Building Materials, **102**, 338 (2016)
14. O. Vololonirina, M. Coutand, B. Perrin, Construction and Building Materials, **63**, 223 (2014)
15. F. McGregor, A. Fabbri, J. Ferreira, T. Simões, P. Faria, J.C. Morel, Materials and Structures, **50**, 193 (2017)
16. G. Talev, B.P. Jelle, E. Næss, A. Gustavsen, J.V. Thue, Journal of Building Physics, **37**, 103 (2013)
17. J. Fořt, Z. Pavlík, J. Žumár, M. Pavlíková, R. Černý, Journal of Building Physics, **38**, 156 (2014)
18. C. Feng, H. Janssen, Building and Environment, **99**, 107 (2016)

19. G.H. Galbraith, R.C. McLean, D. Kelly, *Building Research and Information*, **25**, 348 (1997)
20. R.C. McLean, *Building Research and Practice*, **18**, 82 (1990)
21. S.C. Chang, N.B. Hutcheon, *Canadian Journal of Technology*, **31**, 175 (1953)
22. K.C. Ng, H.T. Chua, C.Y. Chung, C.H. Loke, T. Kashiwagi, A. Akisawa, B.B. Saha, *Applied Thermal Engineering*, **21**, 1631 (2001)
23. M. Pazera, M. Salonvaara, *Journal of Building Physics*, **33**, 45 (2009)
24. R.D. Prangnell, *Matériaux et Construction*, **4**, 399 (1971)
25. J. Berger, T. Colinart, B.R. Loiola, H.R.B. Orlande, submitted to *Wood Science and Technology* (2020)
26. C.K. Ho, S.W. Webb, *Journal of porous media*, **1**, 71 (1998)
27. N. Shokri, P. Lehmann, D. Or, *Water resources research*, **45**, W10433 (2009)