HYGROTHERMAL PROPERTIES OF LIGHT EARTH INSULATION MATERIALS: EVALUATION OF UNCERTAINTIES AND CONSEQUENCES

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Abstract

Hygrothermal simulation of bio-based materials requires heat or moisture storage and transfer properties. Even if their determination is defined in numerous standards, characterization implies uncertainties. In this work, we aim to evaluate the uncertainties in the determination of heat and moisture storage properties of light earth insulation materials and of their constitutive raw materials (hemp shiv and earth). Heat thermal capacity is evaluated by Differential Scanning Calorimetry (DSC). Sorption isotherm is evaluated by gravimetric methods. Attention is paid on protocols and methods, on the measurement repeatability or on the influence of drying temperature and relative humidity. Results show that uncertainties up to 10 % are noted for heat capacity, while they may rise up to 100 % for sorption isotherm. These values are compared to uncertainties due to material variability. In addition, a discussion is proposed regarding the validity of mixing law. Last, the consequences of all uncertainties are evaluated on the prediction of hygrothermal behavior of an existing wall insulated with light earth insulation materials.

Keywords:

Hemp concrete; Uncertainty; sorption isotherm; heat capacity; hygrothermal simulation;

1 INTRODUCTION

One major feature of bio-based building materials is that there are hygroscopic, i.e. they can adsorb water vapor from its surrounding air with given relative humidity and temperature. This property is generally characterized by the moisture content which is defined as the ratio between the amount of adsorbed moisture within the material and its dry mass.

Equilibrium moisture contents are usually measured by two experimental gravimetric methods: Saturated Salt Solution (SSS) method (which is detailed in the standard ISO 12571 [ISO 12571 2013]) and Dynamic Vapor Sorption (DVS) method. For both methods, the measurement consists in exposing a sample to constant relative humidity and temperature and measuring its mass until equilibrium. Afterward, the moisture content is evaluated knowing the mass obtained in a dry state.

Because of its simplicity, SSS method is largely used in the field of building physics. Previous studies showed this method gives reliable results when the RH is below 80 % [Roels et al. 2004], [Roels et al. 2010], [Feng et al. 2015], while errors increase when the RH rises above 80%. Possible reasons are inaccurate RH control in dessicators, sensitivity to mold growth for high RH levels or deviations in dry mass determination in case of interlaboratory comparison [Roels et al. 2010]. In addition, this method presents two limits. First, the number of measurement points is limited to the number of available salts. Second, this method is time-consuming. Indeed, the standard mentions that “test specimen shall be representative of the product and have a mass of at least 10 g”. In the case of bio-based building materials, as representative element volume is generally larger than 5x5x5 cm^3, moisture transfer within the sample until equilibrium takes several weeks.

These drawbacks can be partly overcome by DVS method: automated devices set accurately RH levels around the sample by adjusting a mixture of dry and water vapor saturated gas streams and measure sample mass variation either for a fixed time or until a pre-specified mass variation criterion is reached. Even if the repeatability and reproducibility of DVS method is good [Bui et al. 2017], the measurement accuracy depends however to the above-mentioned criterion [Glass et al. 2018]. In addition, the maximum mass supported by DVS device do not exceed 5 g: this is less than the 10 g specified in the standard ISO 12571 and the representativeness of the sample is questioned.

In the literature, there are several attempts to compare SSS and DVS methods [Peuhkuri et al. 2005], [Fabbri

et al. 2017], [Bui et al. 2017]. Excluding the above-mentioned uncertainties related to each method, the differences between both methods are generally found to be slight (in particular when the RH is below 80 %) and are mainly attributed to the determination of the dry mass: oven-drying at elevated temperature for SSS method and drying with dry gas (air or nitrogen) for DVS method. Interestingly, Fabbri et al. [Fabbri et al. 2017] proposed to adopt the DVS drying protocol to their SSS experiments in order to make a reliable comparison. They observed graphically a good consistency between both methods without quantifying the errors.

As underlined above, an accurate knowledge of the dry mass is of high importance when measuring sorption isotherms. For instance, the standard ISO 12571 recommends to dry samples in oven in accordance to the standard ISO 12570 [ISO 12570 2000]. In the latter, it is mentioned that drying temperature should be set to the one specified in the product standard or by default to 40 °C, 70 °C (recently reduced to 65 °C) or 105 °C depending on the materials sensitivity to temperature. In addition, the relative humidity should be maintained below 10 %. However, product standards are rather scarce for bio-based building materials. Furthermore, a brief overview of the two previous ICBBM conference proceedings allow concluding that there is no consensus about the best drying technique, even for identical materials. Therefore, spread values of moisture content / sorption isotherms can be encountered in the literature. Consequently, as other properties, like specific heat capacity, thermal conductivity or moisture diffusion, may also depend on moisture content, reviews and comparisons of results from the literature is therefore difficult.

In this work, we aim to investigate the influence of various drying protocols on moisture content and to compare the results to other sources of uncertainties. This study is applied here to light earth building materials and their constitutive materials hemp shiv and earth slip.

2 MATERIALS AND METHODS

2.1 Light earth building material

The studied material is only made of earth and hemp. Here, earth is obtained from an excavation near Nocé (Normandy, France). It is a clayey sand composed of 15% clay, 8% silts, 63% sand and 13% gravels. The soil Methylene Blue Value is 1.05 g/100g and the soil Methylene Blue Activity is 7. To ensure a good workability of earth during material fabrication, earth slip is prepared by diluting and sieving raw earth at 2 mm.

Hemp is provided by producer from the same region. The variety of these hemp shives is Fedora 17. The seeding density is 50 kg.m^3 and the harvesting year is 2013. Further characteristics about hemp can be found in [Vinceslas et al. 2017].

Light earth building material is then prepared by mixing earth slip and hemp shiv. As presented in Table 1, three formulations and two setting processes are tested. Further details about fabrication process can be found in [Vinceslas et al. 2017].

Tab. 1: Main characteristics of tested light earth building materials.

Material	Hemp fraction	Setting process	Dry density
M1	42 %	Moulded	316
M2	53 %	Moulded	233
M3	34 %	Sprayed	304

2.2 Characterization methods

Sorption isotherm and specific heat capacity are measured for the three tested light earth building materials as for the constitutive materials hemp shiv and earth slip. The different characterization methods are detailed hereafter.

Sorption measurement with Saturated Salt Solution method (SSS)

For SSS method, the main specifications defined in the standard ISO 12571 are recalled here. For each material, three samples representative of the product with a mass of at least 10 g are placed on glass cups. As hemp shiv have dry density less than 300 kg.m^{-3}, the weighing cups have an area of at least 100 ×100 mm^2. For light earth building materials, cubic samples with dimensions of 5 x 5 x 5 cm^3 are prepared.

Then, samples are dried prior to sorption experiments. As highlighted in the introduction, setting drying temperature is an important stage of the protocol. For earth-based materials, standards recommend drying temperature ranging from 40 °C [ISO 11464 2006] to 110 °C [EN 14063-1 2016]. For cellulosic-based materials, values ranging between 70 °C [EN 13171 2013] and 103 °C [ISO 13061-1 2014] can be found. Based on these elements, the following drying protocols are tested in this work:

- Initial drying at 105 °C in a ventilated oven,
- Initial drying at 40 °C and final drying at 105 °C in a ventilated oven,
- Initial drying at 40 °C and 20 % in a climatic chamber and final drying at 105 °C in a ventilated oven.

Note that the air in the oven is frequently the same air as in the lab. Consequently, the relative humidity in the oven depends on the climate in the laboratory: for instance, assuming that air vapor contents are 7 and 14 g.m^{-3} respectively in wintertime and in summertime, relative humidity varies from 1 to 2 % when drying temperature is 105 °C, but from 16 % to 30 % when drying temperature is 40 °C.

Once the constant mass is reached after drying, *i.e.* when the weight change between two consecutive weighing made 24 hours apart is less than 0,1 % of the total mass, sample are then placed until equilibrium in desiccators equipped with fans in which relative humidity is controlled by saturated salt solutions. Four to eight relative humidity increasing in stages are tested until moisture equilibrium, the temperature being maintained at 23 ± 0.5 °C.

Sorption measurement with DVS device (DVS)

For DVS method, sorption measurements are made on one sample with IGASorp-HT device. The protocol is the following: 10 to 100 mg of material is placed in the IGAsorp microbalance, which has resolution of 0.1 µg.

Prior to the start of the adsorption analysis, the sample is dried in flowing air (250 mL/min) until a constant weight is reached. Here, two drying protocols similar to the previous ones are tested:

- Initial drying at 105 °C with dry gas,
- Initial drying at 40 °C with humid gas (RH = 20 %).

Then, it is exposed to increasing humidity from 10% to 95%, in 15% humidity steps, the testing temperature being 23 °C. The equilibrium mass at each step can be determined either by extrapolation of a single exponential curve fit to the time-dependent mass response following a step change in RH or when maximum hold time is reached. After each experiment, final drying at 105 °C with dry gas is performed.

At least 3 experiments were conducted on earth slip and hemp shiv, but none on light earth building material. Indeed, since the sample mass should be less than 5g, it is rather difficult to get a sample representative of the whole material.

Specific heat capacity

The specific heat capacity measurements are carried out using a micro-DSC III calorimeter (Setaram, Calluire, France) according to the standard ISO 11357-4. A minimum mass of 100 mg is placed in 1 cm^3 sealed vessels. Two methods are tested: a continuous method for which temperature is increased from 5 °C to 30 °C using a heating rate of 0.2 $°C.min^{-1}$ and a stepwise method for which temperature is increased from 15 °C to 25 °C using a heating rate of 0.2 $°C.min^{-1}$. Three measurements are made on all materials dried according the above-mentioned drying protocols.

3 RESULTS

3.1 Earth slip and hemp shiv

Moisture contents w50 and w80

Moisture contents measured at relative humidity of 50 ± 2 % and 80 ± 2 % are presented in Fig. 1 and 2. In addition, uncertainties are evaluated in Table 2.

Repeatability could be analyzed almost for all tests. Values obtained with the DVS method are the most repeatable: relative errors do not exceed 2.3 %. This is due to the high precision of the microbalance and to the accurate control of ambient conditions. On the other hand, larger variabilities are observed for moisture contents measured with the SSS method. For instance, relative errors up to 37 % are noted when relative humidity is fluctuating during drying stage. Drying the samples in climatic chamber under controlled conditions allow reducing the variability in dry mass determination: relative errors are reduced to 3.2 % and 11.7 % respectively for earth slip and hemp shiv. Since earth slip is macroscopically homogeneous, the errors may be due to two reasons: the weight-scale precision and the relative humidity control accuracy within desiccators. For hemp shiv, additional uncertainty may be due to the heterogeneity of the sample.

The sample conditioning before sorption experiment is of high importance. For instance, the absence of relative humidity control during drying at 40 °C may lead to high discrepancy, as underlined by the relative errors up to 46 % observed for earth slip. Nevertheless, the main influencing parameter remains the drying temperature whatever the measurement method, as highlighted by the relative error higher than 69 %. Note that the relative error is higher at 50 % than 80 %, since the absolute moisture uptake is lower for this relative humidity. Last, we observe an influence of thermal history on the results, since drying before or after the sorption experiment do not lead to the same moisture content. Differences of 10 % are evaluated for earth slip and up to 43 % for hemp shiv. The materials undergo probably a modification of their structure and/or the surface properties.

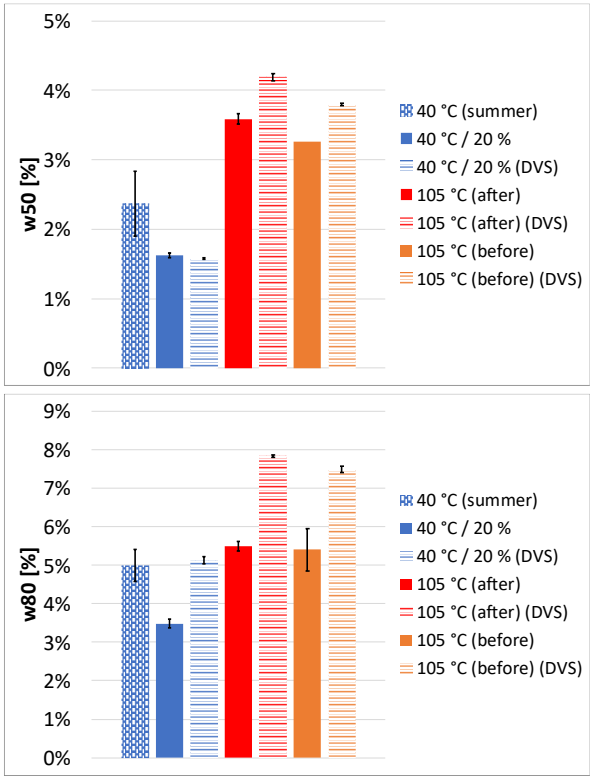

Fig. 1: Moisture content w50 and w80 of earth slip.

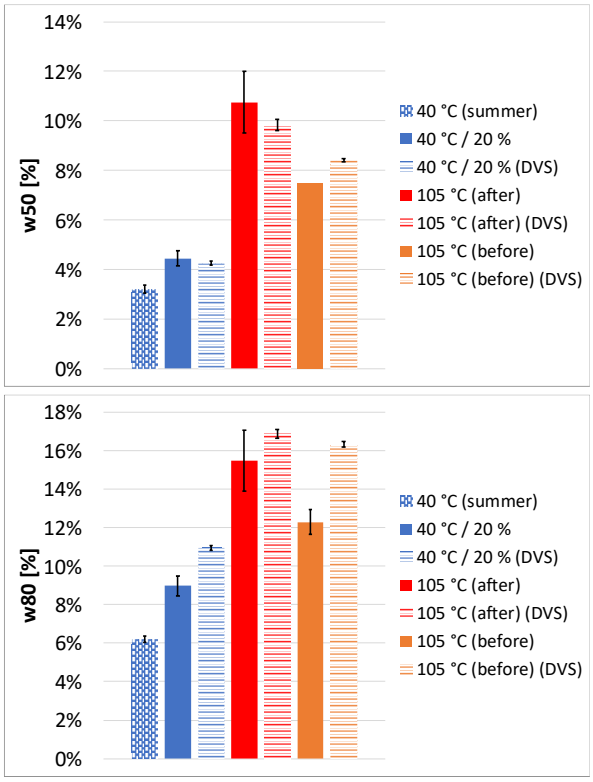

Fig. 2: Moisture content w50 and w80 of hemp shiv.

When comparing the measurement method, we observed that differences exist (up to 50 % for earth slip and 38 % for hemp shiv) and that DVS method lead always to higher moisture content. This finding is logical when samples are dried at 105 °C: DVS uses dry gases while 1 to 2 % of relative humidity is remaining in air for oven drying.

Tab. 2: Maximal uncertainties related to the measurement of moisture content w50 and w80 of earth slip and hemp shiv.

	Earth slip	Hemp shiv
Repeatability (SSS)	3,2 %	11,7 %
Repeatability (DVS)	1,8 %	2,3 %
Drying relative humidity (SSS)	46 %	31 %
Drying temperature (SSS)	102 %	69 %
Drying temperature (DVS)	141 %	99 %
Thermal history (SSS)	10 %	43 %
Thermal history (DVS)	10 %	16 %
Measurement Method	50 %	38 %

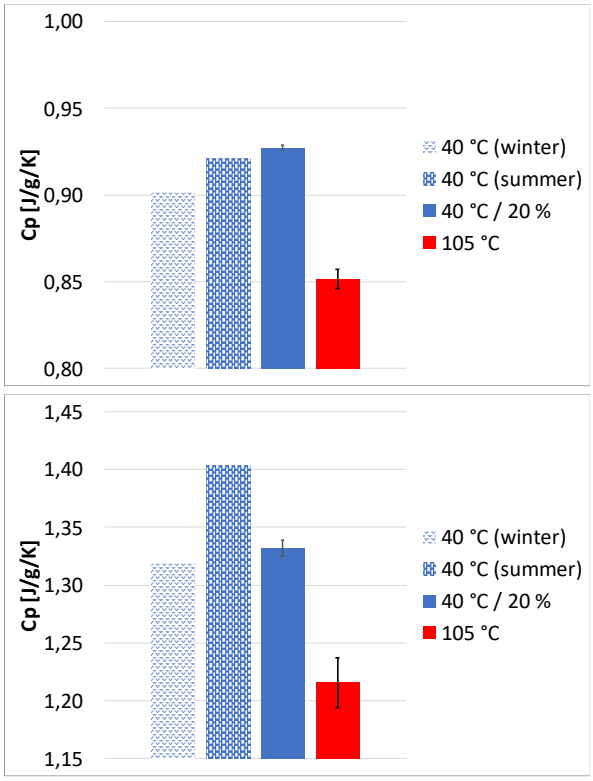

Fig. 3: Dry specific heat capacity of earth slip and hemp shiv.

Specific heat capacity

Dry specific heat capacity measured at 23 °C is presented in Fig. 3. First, we note a good repeatability of the measurement, the uncertainty being always lower than 2 %. Second, the sample conditioning before experiment also influences the results, but to a lower extend compared to sorption experiment: the absence of relative humidity control during drying at 40 °C lead to differences of 5.5 %, while they are less than 9% due to drying temperature. Last, contrary to sorption experiment, similar trends are observed for earth slip and hemp shiv.

3.2 Light earth building materials

Moisture content of light earth building materials was measured by SSS method. Since experiments takes several months, only one drying protocol was tested, namely initial drying at 40 °C and final drying at 105 °C in a ventilated oven. Experimental results are presented in Fig. 4.

First, repeatability of the measurement is rather good: uncertainty is always lower than 10 % and show similar levels than the previous measured one on constitutive material. This indicates that the sampling of materials is correct and that the volume of samples is *a priori* larger than the Representative Element Volume (REV). Second, we still observe that drying temperature is the most influencial parameter on moisture content: difference can be up to 150 %. In addition to the moisture content level, drying temperature change also the sorption capacity, *i.e.* the slope of sorption isotherm.

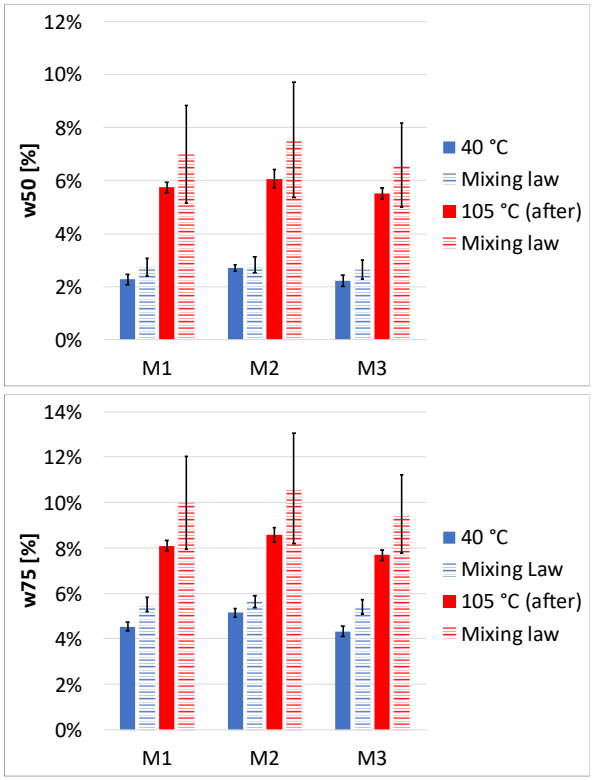

Fig. 4: Moisture content w50 and w80 of light earth building materials: measurement and mixing law.

Last, these experimental data are compared to results obtained from a mixing law defined as:

$$w = f_{hemp} w_{hemp} + f_{earth} w_{earth} \quad (1)$$

With f and w respectively the mass fraction and the moisture content of each constitutive material. Error bars include uncertainties related to the measurement

of moisture content of hemp shiv and of earth slip, but also the ones related to the mass fraction. Considering all uncertainties, a rather good agreement can be observed between predicted and measured data, the difference being always lower than 25 %.

A similar approach is adopted for dry specific heat capacity measured at 23 °C, the mixing law being defined as:

$$c_p = f_{hemp} c_{p,hemp} + f_{earth} c_{p,earth} \quad (2)$$

Results are presented in Fig. 5. As for the constitutive materials, the repeatability is excellent in spite only a few milligrams of material can be tested. Increasing the drying temperature reduces the dry specific heat capacity by 8 %. Last, predictions with a mixing law match well with the experimental data.

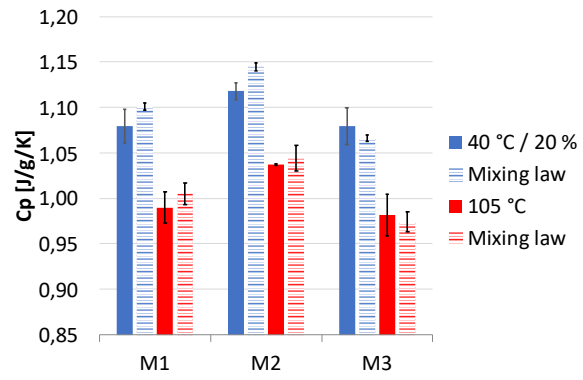

Fig. 5: Dry specific heat capacity of light earth building materials: measurement and mixing law.

4 DISCUSSION

The above-presented results underline that initial conditioning of sample and choice of characterization method is of high importance in the evaluation of thermal and hygric storage properties. Particularly, as the observed uncertainties are much larger than the intrinsic ones to each characterization method, it highlights that literature data can be compared if, and only if, the protocols are rigorously identical.

A second feature concerns the consequence of above-observed discrepancies, particularly on hygrothermal simulations. To date, sensitivity analyses are usually performed when evaluating the hygrothermal behavior of bio-based building materials [Tran Le et al. 2010]. For instance, sorption isotherm can be modified or rescaled, but not necessary based on physical consideration. Here, we proposed a sensitivity analysis considering the previous results on thermal and hygric storage properties.

The case study concerns an existing 20 cm thick stone wall insulated on its interior side with 10 cm of light earth building material. Hygrothermal simulations were conducted using WUFI Pro 5.3 software. For stone, material properties are taken from WUFI material database. For light earth building material, above measured dry density, dry specific heat capacity and sorption isotherm obtained by the mixing laws are used. Heat and moisture transfer properties were also measured in the lab. Their values are gathered in Table 3 and are kept constant during the simulation for sake of clarity.

Tab. 3: Heat and moisture transfer properties of light earth building materials.

Property	Value
Thermal conductivity	0,115 $W.m^{-1}.K^{-1}$
Vapor diffusion resistance factor	3,5
Water absorption coefficient	0,02 $kg.m^{-2}.s^{-0,5}$
Free water saturation	250 $kg.m^{-3}$
Porosity	0.85

Simulations are run for north-oriented wall located in Grenoble, France. Interior climate is derived from the exterior climate considering a “high moisture load” defined in the standard EN 15026 [EN 15026 2008]. Initial relative humidity is set to 80 %. Six simulations are then performed for two years. Analysis is focused during the second year on the predicted relative humidity at the interface between stone wall and light earth building material. Their mean daily variations are presented in Fig. 6 with a focus on the influence of drying relative humidity and temperature.

Whatever the simulation, relative humidity varies in the range [60 – 85 %], meaning that retrofitting is safe in the present case. However, results are spread depending on the sorption curve used. As expected, the drying temperature has the largest influence on the results: difference up to 7 % are observed in the predicted relative humidity; nevertheless, the curves seem only to be shifted. On the other hand, controlling relative humidity during drying has less influence on the simulation, the differences being less than 2.5 %, the shape of the results is significantly different. Note that these differences are larger than the measurement uncertainty of usual relative humidity sensors. Regarding the thermal behavior, temperature differences lower than 0.1 °C are always noted, which is less than the usual temperature sensor accuracy.

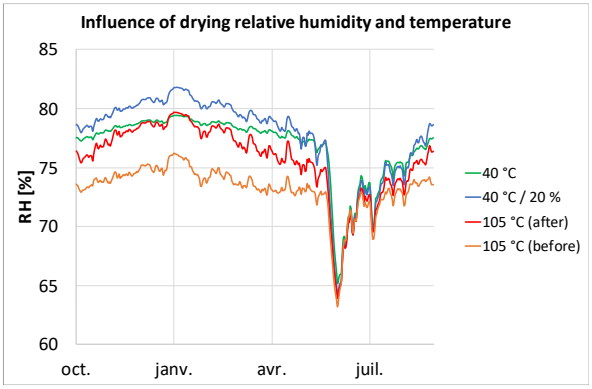

Fig. 6: Evolution of simulated relative humidity at the interface between stone wall and light earth building material: influence of drying relative humidity and temperature.

Fig. 7 focus rather on material variability by comparing prediction for the three tested materials. As sorption isotherm are rather similar (see Fig. 4), we note almost no differences between the simulations.

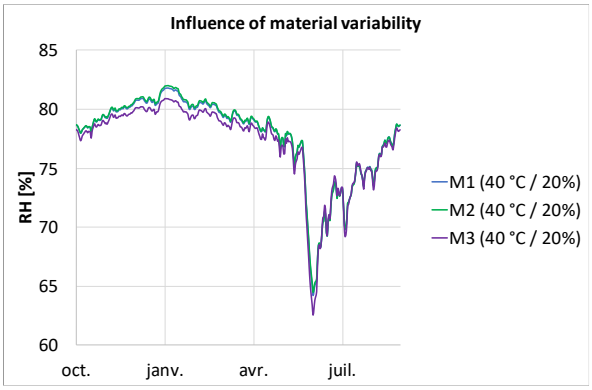

Fig. 7: Evolution of simulated relative humidity at the interface between stone wall and light earth building material: influence of material variability.

5 CONCLUSIONS

This work focused on the evaluation of uncertainties during experimental characterization of thermal and hygric storage properties. Particularly, attention is paid to the intrinsic uncertainty of each characterization method, to the influence of initial conditioning of the sample and to the comparison between different methods. This was applied to light earth building materials and their constitutive materials hemp shiv and earth slip.

Results indicates that drying temperature and drying relative humidity (to a lesser extend) are the most influencing parameters, particularly on the moisture content determination. Consequently, simulated hygrothermal behaviors are largely impacted: differences up to 7 % were observed in the relative humidity prediction for a case study.

As expected, different characterization methods lead to different results since their initial conditioning protocols are not strictly identical. However, no conclusions can be drawn regarding the best method.

Last, it was found that using a mixing law lead to satisfying results. This point is interesting in the view of speed up the characterization, since the measurement on constitutive materials takes less time.

6 ACKNOWLEDGMENTS

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