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ORIGINAL

Experimental characterization of thermal and hygric properties of hemp concrete with consideration of the material age evolution

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Abstract The incorporation of plant crops in construction materials offers very good hygrothermal performance to the building, ensuring substantial environmental and ecological benefits. This paper focuses on studying the evolution of hygrothermal properties of hemp concrete over age (7, 30 and 60 days). The analysis is done with respect to two main hygric and thermal properties, respectively: sorption isotherms, water vapor permeability, thermal conductivity and heat capacity. In fact, most of these parameters are very susceptible to change function of the age of the material. This influence of the aging is mainly due to the evolution of the microstructure with the binder hydration over time and the creation of new hydrates which can reduces the porosity of the material and consequently modify its properties. All the tested hemp concrete samples presented high moisture storage capacity and high-water vapor permeability whatever the age of such hygroscopic material. These hygric parameters increase significantly for high relative humidity requiring more consideration of such variability during the modeling of coupled heat and mass transfer within the material. By the same, the thermal conductivity and heat capacity tests highlighted the impact of the temperature and hygric state of the studied material.

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1 Introduction

Nowadays, the study of coupled heat and mass transfers in porous building materials becomes an essential part of the methodological arsenal of the modern thermal studies. It became essential step to corroborate the energetic and environmental challenges. Moreover, the preliminary selection of materials used for construction has an important role in the success of a high environmental quality project and the constitution of energy efficient systems. The research was soon directed to the use of materials based principally on vegetal particle. These materials appear as an inevitable solution in response to environmental problems. Among these materials, hemp concrete becomes more and more used in construction because of its renewable plant, not degradable with time [1–4]. It is composed of vegetable fibers, binder (mainly lime) and water, and appears as a relevant solution. This kind of material is mainly used as fill material in the construction of building envelopes. It may cover several uses in buildings that differ by the used formulation [5, 6]. This ecological material made from renewable vegetable granulates allows carbon storage during them growth [7, 8]. In addition to its good mechanical and acoustic properties [9–12], hemp concrete is lighter compared to traditional building materials, and has excellent thermal insulation properties thanks to its low thermal conductivity [13–16]. This property depends on the material composition. As the binder is more conductive, the increasing of binder content leads to increased thermal conductivity [17]. The material density can also influence the thermal conductivity of hemp concrete. In fact, more the density increases the thermal conductivity increases also [18].

From hygric point of view, the hemp concrete has many advantages. It has an excellent moisture buffer capacity

(MBV) [19], which enables it to maintain the indoor air quality. In addition, it is very porous structure [20], and its total porosity very close to the open porosity, make it enable to adsorb large quantities of water [21]. Furthermore, concrete hemp has high water vapor permeability and the ability to substantially moderate the changes in relative humidity of the surrounding air. It thus presents a very good humidity regulation capacity [22]. In general the hemp concrete can decrease the daily changes in the relative humidity inside by adsorption and restitution of moisture, reduce energy consumption [23] and maintain the building hygrothermal comfort [16].

In order to better apprehend the behavior of porous building materials and to improve the prediction of energy and environmental performance of buildings, the different heat and mass transfer phenomena require fine modeling in different parts of the building envelope. For this purposes, several transfer models were developed to predict the hygrothermal behavior of building materials [24–28]. The various input parameters of these models are evaluated experimentally. Slight divergence between the evaluated parameters can be observed because of the formulation difference, the type and nature of the constituents used for the fabrication of the material.

Most of papers on hemp concrete hygrothermal characterization neglect the durability factor where these properties vary function of the material age. Otherwise, most of these properties are generally considered constant with any dependency on temperature and water content which may conduct to dramatically lack of control of the material behavior.

The objective of this work is to propose a reliable experimental characterization procedure of thermal and hygric properties of hemp concrete used in building envelopes, to better master different model input parameters. The studied properties deal with water vapor sorption isotherms, moisture storage capacity, water vapor permeability, thermal conductivity, and finally the heat capacity. As the envelopes of buildings are exposed to variations in temperature and relative humidity, the effect of temperature and water content on the thermal properties is studied too. Moreover, the above cited properties were measured based on the material ages that aim to evaluate the hemp concrete behavior during the curing phase.

2 Material and methods

In this section, the material setting and formulation is proposed. Concerning the experimental procedure, four main parameters were evaluated, which are: sorption isotherms, water vapor permeability, thermal conductivity, and heat capacity. It is essential to evaluation the impact of the temperature, water content and the age's factors on those parameters. Tests have been realized on different samples, which were cut at 7, 30 and 60 days in order to consider age factor of material.

Fig. 1 Photo of used hemp shiv

2.1 Material setting and formulation

Hemp concrete is a construction material based on bio-aggregate of hemp shives and binder. The hemp shiv (see Fig. 1) used is an aggregate for hemp concrete shuttered, of C020 reference. Its apparent density is about 100 to 110 kg/m^3. The used lime-based binder is called Batichanvre, produced by French lime producers St. Astier. This binder is a mixture of: natural lime from Saint-Astier (Hydraulic: NHL and aerial: CL), cement CEM I 52.5, and different adjuvants to improve the rheology and permeability of hemp concretes. Its density is of 500 kg/m^3.

These products (hemp shiv and binder) are marketed by a manufacturer-distributor of hemp products and ecological insulation: Technichanvre[1] (France). Hemp-based concrete presents variability of structure depending on the selected formulation. In this study, the retained application is a wall application. It is developed from the professional rules of implementation of hemp concrete structures and corresponds to a composition of materials used on construction sites. The mass dosages of shiv, lime and water are recapitulated in Table 1.

2.2 Sorption isotherm

The water vapor sorption isotherm test characterizes the relationship between the material water content and the relative humidity of the surrounding air for different moisture conditions defined by a relative humidity ranging from 0 to 100%. The measurements were performed following the protocol described below.

[1] http://www.technichanvre.com

Table 1 Composition of the studied materials

Shiv (kg)	10
Lime (kg)	25
Water (kg)	35
Water/Lime (−)	1.4
Lime/Shiv (−)	2.5

The samples were beforehand dried in an oven at 40 °C for 24 h and then degassed under air vacuum to complete the drying. The sample was weighed to determine the dry mass and then analyzed on the Belsorp aqua-3® device. The used device method is manometric [29]. It is based on the calculation of the adsorbed water by the acquisition of vapor pressures and using the perfect gases formula. The samples analyzed are lamellae of 40 mm of height, 5 mm of width and 5 mm of thickness.

The water vapor sorption isotherms were determined by the volumetric method, and the Belsorp aqua-3® device which has three analysis ports and that allows high-throughput measurement, and therefore the adsorption isotherm is taken up more precisely. This method makes it possible to palliate for the inaccuracies of scales used in the gravimetric methods.

2.3 Water vapor permeability

The water vapor permeability is an essential property in the hygric characterization of building materials. It gives information about the migration of moisture in these materials. This migration is generally attributed to a vapor diffusion process. The measurement of water vapor permeability is performed using the cup method based on the NF EN ISO 12572 standard [30]. The test is performed under isothermal conditions (23 ° C).

The test consists on creating a partial vapor pressure gradient between the two compartments at the upstream and the downstream of the sample. This gradient is ensured by two different relative humidity, 93% inside the cup for a wet cup or 3% for dry cup, the two for 50% outside. The test was performed using the Gravitest® facility. This device (Gravitest, Gintronic, Switzerland) is a climatic chamber where temperature, relative humidity and the air speed are controlled. Six cups with automated weighing were placed in the chamber. The Gravitest® device includes a robust precision balance (0.00001 g). The sample is a disc with approximately 8 cm of diameter and 1 cm of thickness.

2.4 Thermal conductivity

The thermal conductivity is measured in stationary conditions using the standard method of guarded hot plate according to EN 12664 [31] and EN 12667 [32].

The measurement is performed using the λ-Meter® EP 500 facility. The test consists on reproducing the heat transfer conditions through an infinite surface plate with limited thickness by two parallels and isotherms planes. The tested specimen is placed between a cold and hot plates and subjected to a fixed temperature gradient. A guard ring around the measurement zone prevents transversals heat dissipations and allows directing the heat flow through the sample thickness. The thermal conductivity of the material is then calculated directly from the electrical power provided by the device. The minimum sample size is 15×15 cm^2, with a thickness which may vary (4 cm in our case).

2.5 Heat capacity

The measurement of specific heat capacity is performed using Calvet® calorimeter. It is composed of a calorimetric block enclosed in a sealed cylindrical pregnant. This calorimeter block encloses the measurement cell in which the tested sample is placed and a reference cell. The most advantage of this device is the 3D Calvet sensor that completely surrounds the sample and all the emitted heat is thus measured. A cryostat is connected to the calorimeter in order to regulate its internal temperature. Pumping canalization is also connected to the calorimeter to ensure the placed under vacuum of the enclosure calorimetric. The test is performed with a temperature ramp from −15 °C to 35 °C. The heating rate was 0.1 °C / min. The used cells are standard; they are designed specifically for studies of solid or powdery products with low vapor tension when the internal pressure must not exceed 500 kPa. The advantage of this apparatus lies in the fact that 3D Calvet sensor encircles the entire sample. This 3D sensor is more accurate in comparison with the 2D sensor used in DSC. The loss of heat is between 50 and 70% in the case of a 2D sensor and less than 10 in the case of a 3D sensor. The sample is a cylinder of 1 cm diameter and 2 cm height.

3 Results and discussion

We will present first the sorption desorption isotherms and the moisture capacity of hemp concrete for the three tested ages where the sorption evolution will be expressed through adapted classical model available in literature; the second one corresponds to the vapor permeability measurement, which is realized on wet and dry cup function of the material's age.

In second part, the thermal conductivity and heat capacity evolution at different temperature and moisture content states will be also analyzed along the material age parameter; some promising interpretations are conducted.

The sorption desorption isotherms have been achieved using a the Belsorp aqua-3. Figure 2 represents the water

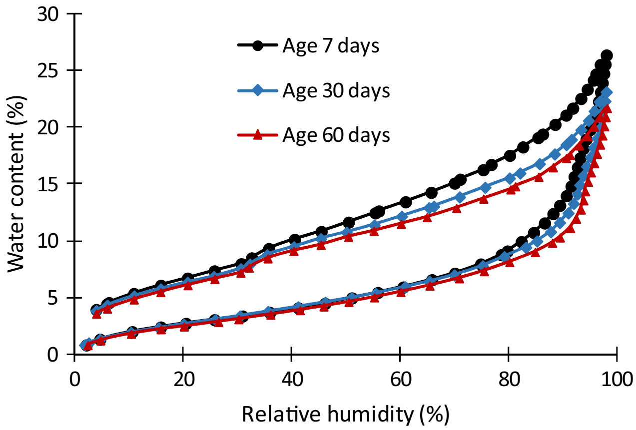

Fig. 2 Comparison of water vapor adsorption- desorption isotherm at 7, 30 and 60 days hemp concrete age at 25 °C

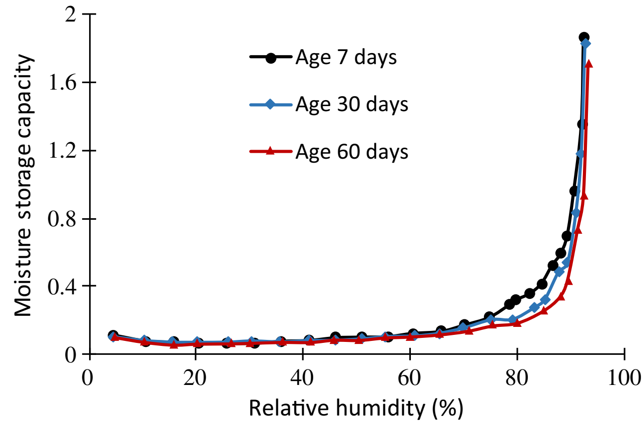

Fig. 3 Comparison of moisture storage capacity at 7, 30 and 60 days at 25 °C

vapor distribution of hemp concrete at 25 °C after 7, 30 and 60 days of age, this curve represents the change in the water content of the material depending on the water activity or relative humidity of air in equilibrium. The water content increases in a progressive manner, and becomes important for high relative humidities, due to the trigger of capillary condensation mechanisms. It is noticed that the obtained isotherms of sorption-desorption exhibit hysteresis between absorption and desorption that extends to the lower pressures. This phenomenon is mainly attributed to the complex morphology of the porous space of the studied material.

Hemp concrete has a high capacity for water absorption, mainly due to its large specific surface. The studied material has an average specific surface of 18.13 m^2/g, this latter is measured by the device ASAP 2020® based on the BET (Brunauer, Emmett and Teller) theory [33]. The obtained sorption isotherms with the volumetric method are of type II according to the classification of the IUPAC [34], which is consistent with the structure of the porosity of hemp concrete.

Furthermore, the evolution of the microscopic morphology [35] during age [36] affects considerably the behavior of this material. The effect of the material age was observed on the sorption behavior of hemp concrete. In fact, the adsorption desorption rate decreases with increasing material age. For example, at 89% of relative humidity, the adsorption decreases from 21.51% between 7 and 60 days. The total error on the variation of the adsorption and desorption isotherm curves is estimated at 0.37%, 0.51% and 0.55% for 7, 30 and 60 days respectively. At early-age, the binder hydrates do not form a connected network yet, over time, the hydrates connect together, and the volume of the solid phase increases which causes a change in the internal structure of hemp concrete and reduces the material porosity and its water adsorption capacity.

These sorption isotherms allow us to deduce the moisture storage capacity of hemp concrete shown in Fig. 3. The moisture storage capacity is an essential input parameter of coupled model of heat, air and moisture transfers. Mathematically, the moisture capacity is defined by the slope of the isotherm curve of water adsorption [37] (Eq. 1):

$$C_m = \frac{\partial u}{\partial \varphi} \tag{1}$$

Where: C_m is the moisture storage capacity [−], u is the water content of the material [kg/kg], and φ the relative humidity [%].

The analysis of the result given in Fig. 3, shows a significant increase in C_m for high relative humidities (>80%). The same behavior is observed with all material ages (7, 30 and 60 days). Therefore, it is necessary to consider the evolution of the moisture storage capacity depending on the relative humidity during hygrothermal transfers modeling, especially in high relative humidity zones. It is also shown that the C_m value decreases with the material hydration progress.

Given the importance of sorption isotherms significance for the study of the behavior of building materials, modeling of this latter curve was performed using GAB (Guggenheim-Anderson-de Boer) and GDW (Generalised D'Arcy and Watt) models [38], this allows a more accurate interpretation of experimental results. The two models listed above are the most widely used models in the literature, their expressions can be written in the following forms (Eqs. 2 and 3):

GAB model:

$$U = \frac{m.C.K.\varphi}{(1-K.\varphi)(1-K.\varphi+K.C.\varphi)} \tag{2}$$

Where, U is the water content at equilibrium, φ the relative humidity, m is the monolayer capacity, C is the kinetic constant related to the sorption in the first layer, K is the kinetic constant related to multilayer sorption.

GDW model:

$$U = \frac{m.K.\varphi}{1+K.\varphi}\frac{1-k(1-\omega).\varphi}{1+K.\varphi} \tag{3}$$

Table 2 Parameters of GAB and GDW models

Age of material	Model	*m*	*C*	*K*	*k*	*ω*	R^2
7 days	GAB	0.023	45.150	0.926	–	–	0.997
	GDW	0.044	–	6.679	0.943	0.476	0.999
30 days	GAB	0.022	52.730	0.916	–	–	0.993
	GDW	0.060	–	3.820	0.953	0.266	0.998
60 days	GAB	0.019	81.20	0.921		–	0.986
	GDW	0.068	–	2.722	0.964	0.187	0.997

Where, m is the concentration of primary active surface sites; ω represents the ratio of water molecules adsorbed on the primary sites which is converted into the secondary sorption sites. K and k are the kinetic constants related to the sorption on primary and secondary sorption sites, respectively.

These parameters of models are calculated using the curve-fitting of the experimental results (see Fig. 2), and recapitulated with the performance criteria in Table 2. We can note that the two models reproduce perfectly the sorption behavior of the tested material (see Fig. 4), but the GDW model is the most suitable to reproduce the experimentally determined sorption isotherm of hemp concrete for the three tested ages.

Concerning the water vapor permeability, it is calculated in permanent regime after mass equilibrium of the cups. Thereafter, the water vapor permeability and the diffusion resistance factor are calculated [30] and shown in Fig. 5. These quantities characterizing the vapor diffusion in hemp concrete are high, which shows the character breathing to this material.

The obtained results (Fig. 5) show a decrease in the water vapor permeability according to the age of hemp concrete. This is due to the hydration reaction, which causes the change of the structure, and affects the porosity and constructivity of the material. A decrease on the permeability values of 37.5% between 7 and 60 days was observed. The standard deviations of repeatability are $2,05.\ 10^{-12}$, $1,13.\ 10^{-12}$ and $4,36\ 10^{-13}$ for samples of 7, 30 and 60 days, respectively. The total errors corresponding to these variations are then of the order of: 6.36%, 4.74% and 2.16% for 7, 30 and 60 days of age respectively. Figure 6 represents the water vapor permeability evaluated at hygrometries of 50%–93% (wet cup) and 50%–3% (dry cup). It is noted that the value of the water vapor permeability increases with increase in the average moisture gradient as expected [39, 40].

Concerning the thermal conductivity results, the Fig. 7 shows the effect of temperature on the thermal conductivity of the studied hemp concrete, this property was determined for three different temperatures (10, 23 and 30 °C) after drying at 40 °C until mass stabilization.

It is noted that the thermal conductivity of the material changes as a function of the test temperature, and increases linearly. These results confirm those in the literature [41], where it was shown that the thermal conductivity is linearly proportional to the temperature. Furthermore, the thermal conductivity is directly related to the porous structure of the

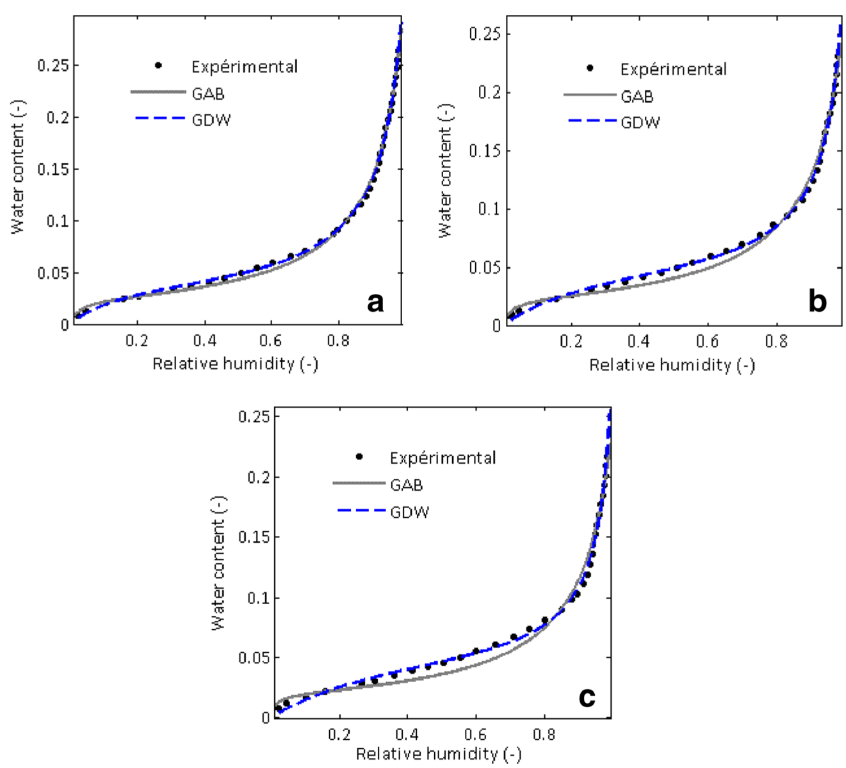

Fig. 4 Sorption isotherm of hemp concrete obtained by GAB and GDW models, at (**a**) 7 days, (**b**) 30 days and (**c**) 60 days

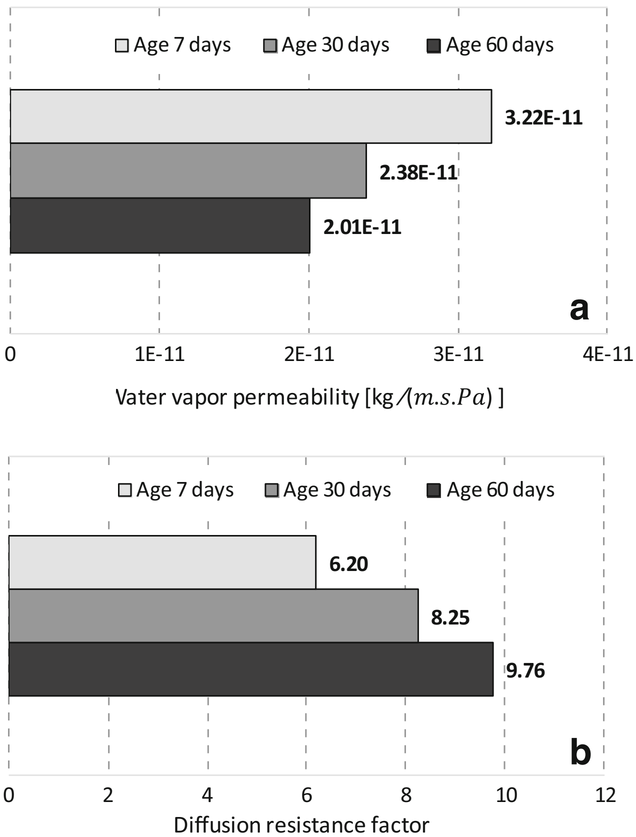

Fig. 5 Average values of various hemp concrete parameters depending on the age for the wet cup: (**a**) water vapor permeability, (**b**) water vapor resistance factor

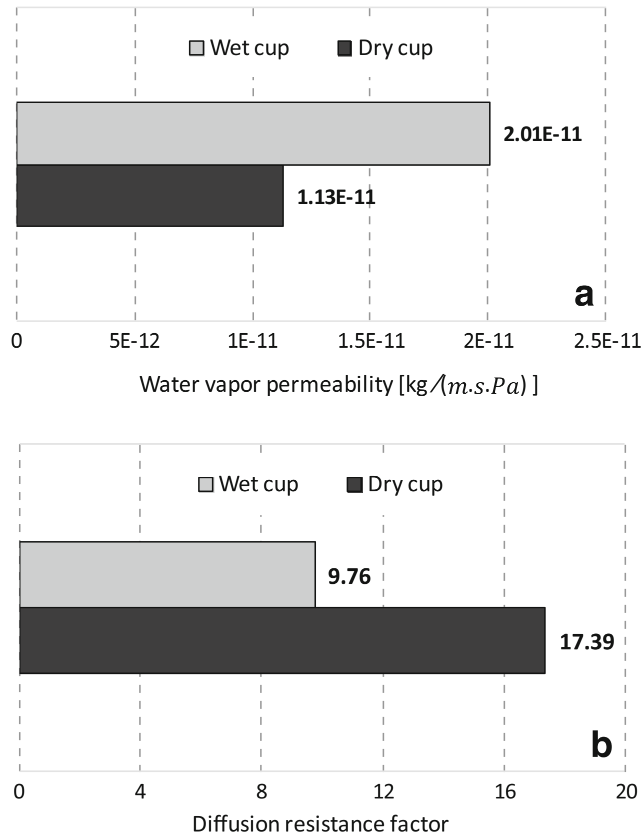

Fig. 6 Comparison between the wet cup and dry cup at 60 days of age: (**a**) water vapor permeability, (**b**) water vapor resistance factor

material, which explains the increase in this property with the age of the material. An increase in thermal conductivity of 11% was observed between 30 and 60 days. In addition, several studies provided by the literature also showed the effect of the hygric status of the material on thermal conductivity [42]. That is why we propose to take this parameter in consideration too; the thermal conductivity tests will be then reproduced for the same specimen at different water content sate.

Figure 8 represents the evolution of the thermal conductivity of hemp concrete studied as function of its water content. The results illustrate the significant effects of the hygric state of the material on the values of thermal conductivities. Indeed, an increase in thermal conductivity of 125% between the dry and saturated states has been obtained.

Another important property for the heat transfer characterization in the building materials is the specific heat. Figure 9 shows the evolution of the specific heat of the studied hemp concrete as a function of temperature at 7, 30 and 60 days of age after drying at 40 ° C until mass stabilization. In fact, the specific heat increases by about 9% between 5 and 35 °C.

Concerning the influence of the age of the materials on this propriety, the specific heat increases by about 25% between 7 days and 60 days, this increase is mainly due to the hydration of the binder, and aging of samples. The specific heat is sensitive to changes in the water content of the material, the Fig. 10 shows the significant effect on the hygric state of hemp concrete on the value of its specific heat, this value changes from 1161 *J*/*kg*. *K* dry to 3021 *J*/*kg*. *K* in the saturated state. an increase of 160% between the dry and saturated states was observed.

In general, the proportion of air decreases when the water content increases in the material. Therefore, the thermal conductivity and specific heat of the material increase and the insulating power of the material decreases.

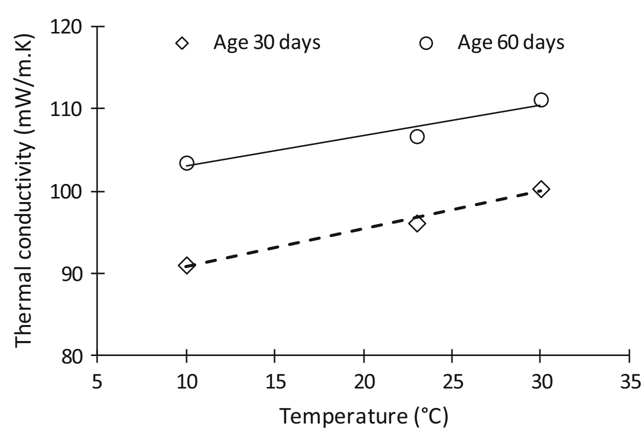

Fig. 7 Comparison of thermal conductivity as a function of hemp concrete temperature at 30 and 60 days of age

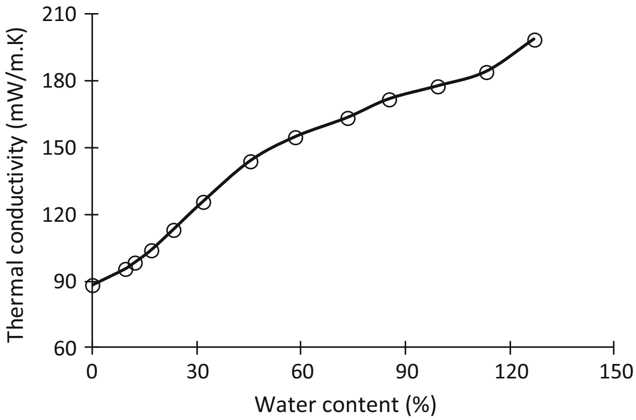

Fig. 8 Evolution of thermal conductivity depending on the water content of the material at 23 °C, and 30 days of age

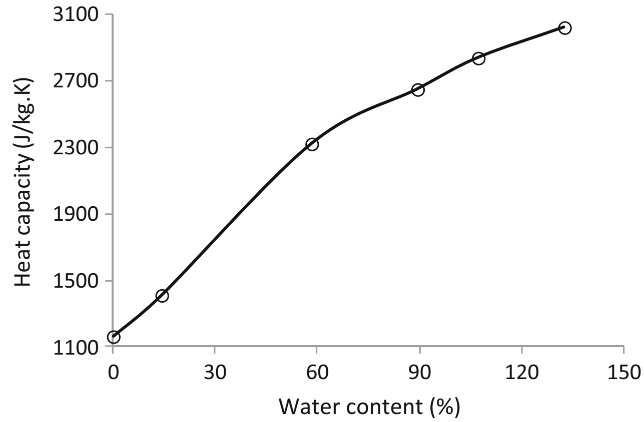

Fig. 10 Evolution of heat capacity depending on the content of the material at 25 °C, and 30 days of age

4 Conclusions

This work presents several experimental approaches that have been developed for hygric and thermal characterization of building materials based on vegetable fibers. This experimental campaign is essential for a good prediction of hygrothermal behavior of hemp concrete.

Particular attention has been paid to the evolution of intrinsic properties as a function of the age of the material. All hygrothermal properties are directly related to the material microstructure and evolves with its age. The hemp concrete aging reduces:

- The rate of adsorption and desorption,
- The moisture storage capacity,
- The water vapor permeability,

And increases:

- Thermal conductivity,
- Specific heat of the material.

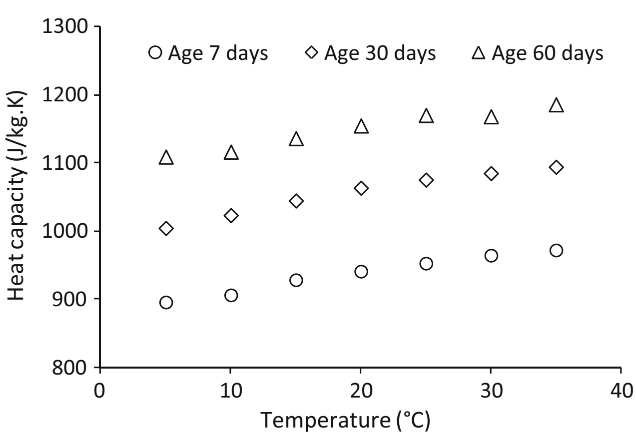

Fig. 9 Comparison of heat capacity as a function of hemp concrete temperature at 7, 30 and 60 days of age

The experimental sorption isotherms exhibit a hysteresis between adsorption and desorption. Moreover, the curves obtained by GAB and GDW models give a good approximation of sorption isotherms.

The sorption isotherm curves allow us to deduce the moisture storage capacity of hemp concrete. This property is defined by the slope of sorption isotherms curves, it increases significantly in the super-hygroscopic zone.

The vapor permeability of hemp concrete measured by the cup method leads to a high water vapor exchange capacity; this property is sensitive to hygric variations and increases with increasing humidity gradient. In fact, the water vapor permeability of a wet cup is greater than that of the dry cup.

The vapor permeability calculated in steady state makes it possible to emphasize the breathing quality of hemp concrete, which is justified by the low value of the coefficient of resistance to the diffusion of water vapor.

Concerning the thermal conductivity and the specific heat capacity, they are highly dependent on the temperature evolution. Also, the obtained value of the thermal conductivity (0.15 W/m.K) shows a good thermal insulation capacity for hemp concrete. Furthermore, the thermal properties were measured as a function of the water state of the material. it is important to note that the hygric status of the material has significant influence on the thermal properties.

This study provides the literature and the future works with promising results, especially concerning the hygrothermal characterization of the hemp concrete properties where the material age should be carefully considered. These parameters are used as input parameters in the simulations models of coupled heat and mass transfer.

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