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Experimental investigation on the influence of immersion/drying cycles on the hygrothermal and mechanical properties of hemp concrete

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Highlights

- Accelerated aging of hemp concrete was experimentally performed by a succession of immersion/drying cycles during 40 days.
- Immersion/drying cycles significantly affected the microstructure of the hemp concrete.
- A 50% decrease in compressive strength and a 38% increase in water vapour permeability were noted for weathered hemp concretes.
- Chemical analyses highlighted the ion leaching of the hemp concrete pore solution into the immersion solution during aging.

Abstract

Hemp concrete is the most commonly used bio-based material for construction because of its hygrothermal behaviour and its contribution to reduce the environmental impacts and energy needs of buildings. Apart from these performances and environmental qualities, there is still a lack of information concerning hemp concrete durability and the evolution of its physical properties over time. That is why this study investigates the influence of accelerated aging on the properties of this material through a succession of immersion/drying cycles. Microscopic observations highlighted morphology changes of this bio-based material and porosity variations caused by swelling and shrinkage phenomena. A significant decrease of 51% in compressive strength was noted for the weathered hemp concrete. Furthermore, functional hygrothermal properties were also affected, and a 38% increase in water vapour permeability was noted. Finally, chemical and thermogravimetric analyses revealed no effect of aging on the pH and thermal decomposition ranges of hemp concrete. From these analyses, crucial conclusions on the durability of this material were drawn.

Keywords: hemp concrete, accelerated aging, durability, microstructure, vapour permeability, thermogravimetric analysis.

1. Introduction

The building sector is one of the most important sources of energy savings and reducing greenhouse gases emissions. This sector is responsible for 40% of energy consumption in the OECD countries (Organisation for Economic Co-operation and Development) and emissions of CO₂ of around 36.2 trillion tonnes. It is therefore responsible for the depletion of natural resources and the damage that our environment undergoes because of greenhouse gas emissions [1]. That is why the current thermal regulation recommends building structures that are more and more thermally insulated. Doing this may reduce the energy and environmental impact. Unfortunately, this may alter summer comfort and indoor air quality because the Thermal Regulations (TR 2012) require minimum air renewal values and a very low level of air permeability of the building envelope of about 0.6 m³/(h·m²) in individual houses and 1 m³/(h·m²) in collective buildings. These new issues have significant consequences since they can seriously affect the health of occupants [2]. In order to reduce these adverse effects, researchers [3–10] tested bio-based materials for construction, that came from either

agriculture, forestry or recycling. These materials are characterised by low density and complex microstructure. Low density is associated with low compressive strength, but also with low thermal conductivity. The use of bio-based materials, recognised by their low environmental impact and their hygrothermal quality [11], allows to reduce the energy impact and therefore to meet the requirements of the thermal regulations.

In this work, the studied bio-composite material is hemp concrete. It is very widespread in France and has a regulatory framework for its use and formulation in the construction sector [12]. Hemp shives are an innovative material and have particularly interesting properties. The mixture of these hemp shives with lime improves the insulating properties of the final material [4]. Admittedly, this material lacks quality in terms of compressive strength. Its resistance varies between 0.1 to 0.8 MPa at the age of 28 days [13], but the advantage is in its thermal, hygric and acoustic performances [14]. That is why its current main use is limited to a infill material [15]. Studies have shown that hemp concrete is a good insulation material with good hygrothermal and acoustic properties [6,16], and it has a low thermal conductivity due to its porosity and low density. From a hygric point of view, hemp concrete is also an aerated material with high water vapour permeability [6,11,15]. In addition, it is considered to be an excellent moisture regulator that is able to absorb relative humidity in the case of surplus in the living environment and to restore it in the inverse case [17,18]. This strong absorbing power allows to moderate daily variations in the relative humidity and to ensure good indoor air quality [19]. Despite all these advantages, the use of bio-based materials is limited by the lack of a guarantee as to the evolution of their properties over time.

Moreover, hemp concrete is a highly heterogeneous and anisotropic material [20]. When this material is exposed to outside climatic conditions, dimensional variations appear [21] and modify its morphological behaviour, which consequently affects the hygrothermal behaviour of the material [22]. In addition, hemp concrete is a hygroscopic material. Its sensitivity to moisture variations causes swelling/shrinking of the hemp shives. In general, aging can modify the material microstructure with the hydration of the binder over time and generate dissolution/precipitation of novel compounds [6]. This could, consequently, modify the hygrothermal and mechanical properties of the material. Few

studies have been conducted for this purpose, and most of them only describe the reaction of hemp concrete against accelerated aging tests [23].

Among the few works on hemp concrete durability, Bessette *et al.* [24] studied the effect of aging and mould growth during the manufacture of cement-based hemp concrete and during its exploitation. Their results show that the outer coating must be well chosen. They showed that the use of a waterproof render that is also permeable to moisture does not exhibit mould growth. Marceau *et al.* [25] studied the effect of humidification/drying cycles on the physical properties of hemp concrete and the mould growth at high hygrometry. They highlighted that the aging cycles modify the material porosity. However, this evolution is not enough to modify the functional properties of the materials. Moreover, Walker *et al.* [13] investigated the influence of the binder on hemp concrete resistance to freeze/thaw cycles, exposure to salt and biodegradation. They showed that hemp concrete formulated with a hydraulic binder is less sensitive to freeze/thaw action. They also showed that salt exposure in the short term resulted in the precipitation of salt layers but did not impact compressive strength at one year. Furthermore, Arizzi *et al.* [23] analysed hemp concrete samples in three different climates: Mediterranean, tropical and semi-arid climates. Macroscopic properties (density and chromatic variations) and the modification of the chemical properties before, during and after accelerated aging cycles were analysed. They showed that the hemp concretes studied have a good durability towards the various stresses and climatic conditions. However, it is advisable to take some specific preventive and maintenance measures to ensure greater durability of these materials. Viel *et al.* [26] studied the resistance of bio-based materials to fungal developments. The tested materials were regularly weighed and photographed in order to monitor weight evolutions and to measure the contaminated surface by moulds. In addition, chemical analyses highlighted that the pH is an important factor for predicting mould susceptibility. Indeed, materials with pH of about 10 are resistant to fungal development. However, those with pH less than or equal to 6 exhibit significant fungal development. Delannoy *et al.* [27] studied the evolution of the functional properties of hemp shives by keeping their material for two years under three different conditions: a static reference environment, humidification/drying conditions and external aging (*in situ*). Based on these different cases of aging, the authors identified four phenomena that are part of aging mechanisms, namely: mass loss, volume variation, opening of

the porosity of hemp shives and modification of the pore size distribution. In addition, they have shown that fungal attacks can alter the microstructure and properties of the hemp shive. For this purpose, the authors suggested monitoring the fungal development of hemp concrete over time.

The aim of this paper is to study the effect of accelerated aging (immersion/drying cycles) on the properties of hemp concrete. This allows to quantify the changes of the hygric, thermal and mechanical properties of hemp concrete due to the immersion/drying cycles (aging). The characterisation was focused on the functional properties of this material, namely: microstructure evolution, water porosity, thermal conductivity, water vapour permeability, water vapour sorption, moisture buffer capacity and compressive strength. The cases of hemp concrete (weathered and reference) were characterised at the same conditions in order to deduce the effect of immersion/drying cycles on the properties of this material.

2. Materials and methods

The innovative material studied is hemp concrete, which can be used in both renovated and new buildings. An experimental campaign was carried to study the effect of aging (immersion/drying cycles) on the hygric, thermal and mechanical properties of this material. The experimental protocol followed is described below.

2.1. Materials

Hemp concrete is obtained by mixing defined quantities of hemp shives, binder and water. The hemp shives used for this study is the Chanvribat (see Fig. 1). It is provided by the manufacturer Ecohabitat (Ecological Materials for Habitat). It has an average density of about 100 kg/m^3 .



Fig. 1. Photo of used hemp shives.

Size and geometry of hemp shives can have an important role in the determination of the final properties of hemp concrete. The particle size analysis of the used hemp shive was done by image processing and is based on 713 hemp shive particles randomly sampled. Particles are then glued on an A3 sheet of black paper avoiding any overlap to facilitate segmentation operations as shown in Fig. 2.



Fig. 2. Used hemp shives for particle size analysis.

The distributions of lengths and widths are given in Figs. 3(a) and 3(b), respectively.

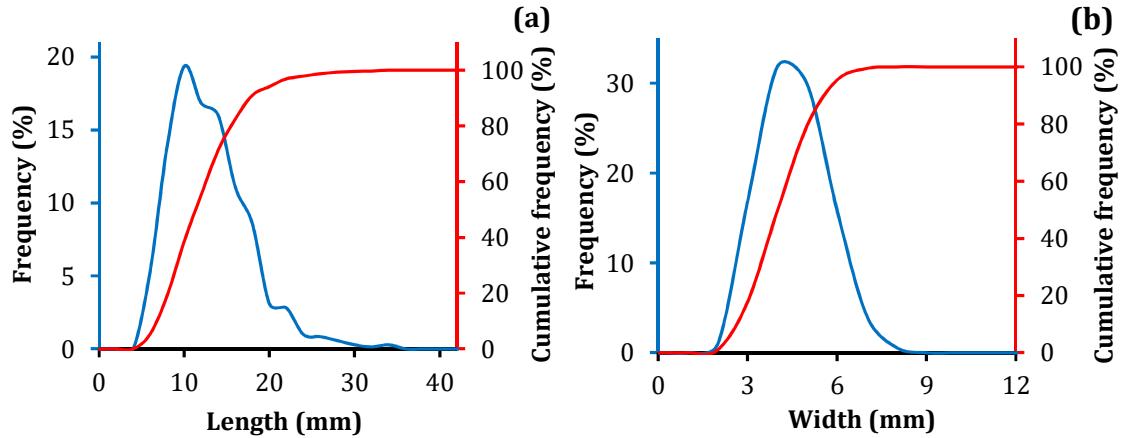


Fig. 3. Characterisation of hemp particle size by image processing: (a) length distribution and (b) width distribution.

Fig. 3 shows that the hemp shives used have different size ranges in length and width. This heterogeneous distribution of hemp shives in the material implies a wide pore size distribution and increases the complexity of the microstructure of hemp concrete, which has a significant impact on the final material properties [28,29].

The binder used for this study is Tradical PF70 (compatible with the used hemp shives) provided by Ecohabitat. It is composed of 75% air lime with 15% hydraulic and 10% pozzolanic binders. This type

of binder can be used alone [30,31] or with other alternative binders based on unfired clay [3]. It often gives good results, especially in terms of compressive strength and thermal conductivity [3].

The application chosen for the formulation of the samples studied is a wall application. It has been set in accordance with the professional rules for the execution of hemp products which were definitively validated in 2012. The used formulation is given in Table 1.

Table 1 Proportion of lime binder, hemp shives and water by mass for concrete.

Hemp shive (Chanvribat)	Binder (Tradical PF70)	Water
16%	34%	50%

After the manufacture of the hemp concrete samples, they were conserved for four days in their moulds in a climate chamber regulated at a temperature (T) of 20 °C and a relative humidity (RH) of 50%. Then, the moulds were disassembled to allow drying of samples, and from this moment, a mass monitoring was carried out with a period of 24 hours in order to obtain the kinetics of drying. Investigations were started after sample mass stabilisation (mass variation less than 0.1%).

2.2. Accelerated aging test and experimental characterisation

The exposure of building materials to outside weather conditions (rain, snow, sun, etc.) can affect their microstructure and consequently modify their hygrothermal behaviour. Assessment of the durability of materials can be done *in situ* (exposure of the material to natural climatic conditions) or in laboratory by means of accelerated aging tests.

In this work, an accelerated aging test was chosen and applied to the hemp concrete. It consists of a succession of immersion cycles in water for 48 hours followed by drying in an oven at 50 °C for 72 hours, over a period of 40 days. This type of aging is significantly more severe than the surrounding conditions of materials during their life cycle, but it gives an answer to the behaviour of the material in its state of extreme degradation. First of all, hemp concrete samples have undergone aging (40 days of immersion/drying cycles). In parallel, other reference hemp concrete samples (unweathered) are stored in a climatic chamber (T = 23 °C and RH = 50%). At the end of the aging process, all the materials (reference and weathered) were collected in the climatic chamber for 15 days before their

characterisation in order to have the same state of hygric and thermal equilibrium at the moment of characterisation [6].

Total porosity

The measurement of the water porosity has been measured using the water porosity test according to the NF P18-459 standard [32]. It is based on vacuum saturation followed by different weighing of the samples. To ensure repeatability measurement, three cubic samples ($5 \times 5 \times 5$ cm) were tested each time.

Microstructure characterisation by digital microscope

To better interpret our results, the microstructure of weathered and unweathered hemp concrete was analysed by using a digital microscope from Keyence®. The dimensional variations of the hemp concrete were monitored by microscopic observations of the same viewing area before and after aging. The digital microscope used produces high-resolution images, allowing to clearly observe the morphological changes of the hemp concrete.

Thermal conductivity

Thermal conductivity was measured under stationary conditions using the λ -Meter Ep500e® device. The principle is based on the guarded hot plate method according to the standards EN 12667 [33] and EN 12664 [34]. This property was measured on six samples of $15 \times 15 \times 5$ cm at a temperature of 23°C .

Water vapour permeability

Water vapour permeability characterises the ability of a material to transfer moisture under a vapour pressure gradient. The measurement was performed by using the Gravitest® device, which is based on the cup method according to the standard NF-EN-12572 [35] (see Fig. 4). The test is performed on three cylindrical samples of 8 cm in diameter and 2 cm thick.

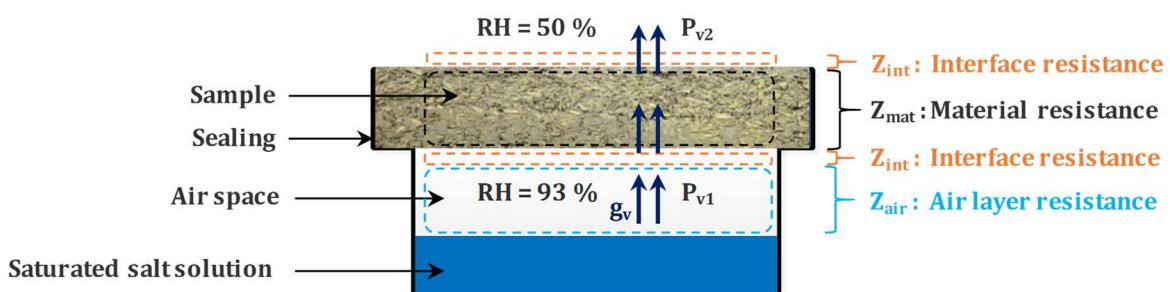


Fig. 4. Standard cup method for determination of water vapour permeability.

Sorption isotherm

The measurement of the water vapour sorption isotherms is performed at 23°C by using the ProUmid device, which is based on the principle of the gravimetric method. Before beginning the test, the cubic samples (1 cm^3) were dried in an oven at 40 °C for 24 hours and then degassed under vacuum in order to complete the drying. To ensure the repeatability of the results, three samples were tested for each case.

Moisture buffer capacity (MBV)

The moisture buffer capacity characterises the ability of the material to moderate the relative humidity changes of the surrounding air. In this study, the measurement of this parameter has been performed in accordance with the Nordtest project [36]. This project proposes to subject the samples of hemp concrete to cyclic step-changes in humidity between 75% for 8 hours and 33% for 16 hours at a constant temperature of 23 °C. The samples were initially preconditioned at 50% relative humidity.

Three samples of 10 cm sides and 5 cm thick were tested for each case.

The Nordtest project proposed a classification of materials according to their moisture buffer capacity.

The different moisture buffer value (MBV) ranges are given in Fig. 5.

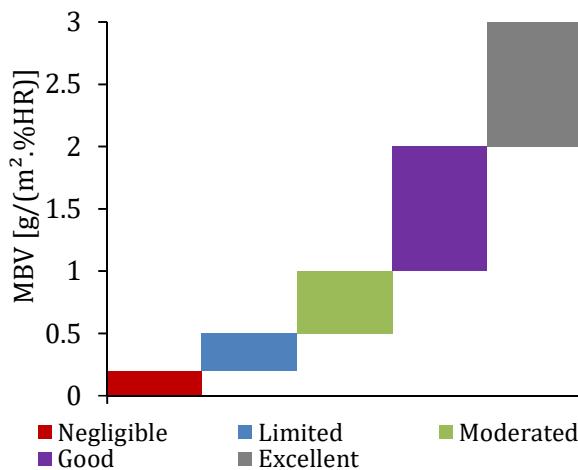


Fig. 5. Moisture buffer value classes.

Penetration depth

The penetration depth ($d_{p,1\%}$) provides a reference value that can be used to evaluate the thickness of the sample for measuring the MBV value [37]. This property defines the depth where the amplitude of moisture content variation does not exceed 1% of the surface amplitude [36]. It is given by Equation 1.

$$d_{p,1\%} = 4.61 \sqrt{\frac{D_w t_p}{\pi}} \quad (1)$$

where t_p is the period, and D_w is the moisture diffusivity.

Compressive strength

The compressive strength test is performed according to European standard EN 826 [38] and ASTM C109 [39] as well as the current studies on this type of material [40,41]. Three cubic samples of 5 cm sides were tested for each case using the Zwick Roell® test apparatus, with a displacement rate of 5 mm/min and a maximum load of 100 kN. ASTM C109 [39] requires that the maximum permissible range, between specimens (5×5×5 cm) from the same material at the same test age, is 8.7% of the average when three cubes represent a test age and 7.6% when two cubes represent a test age.

pH measurement

After hemp concrete degradation, there is a high probability that the pH will change [25], and hence there is the necessity to measure this evolution function of the aging. The adopted measuring technique is the production of an aqueous solution [42], which consists firstly in reducing the hemp concrete samples to powder. Then, this latter was mixed with distilled water with a liquid/solid ratio of 9 ml/g to obtain an aqueous suspension. Finally, pH values were measured by using the pH meter 780 from Metrohm®. Three solutions were prepared and dosed three times for each one.

Thermogravimetric analysis

The thermogravimetric analysis (TGA) test allows to quantify the material mass evolution function of the temperature. Each mass variation reflects a chemical, physical or physico-chemical phenomenon due to the effect of temperature. This test aims to verify whether the decomposition temperature ranges of the components remain the same after the aggressive aging applied. The measures were performed by using the SETSYS Evolution 16/18 device from SETARAM®. The test is performed in a controlled helium atmosphere in a temperature range of 25–500 °C for hemp shives and of 25–900 °C of binder with a heating rate of 5 °C/min.

3. Results and discussions

3.1. Microstructural and chemical degradation

Fig. 6 shows a comparison of the hemp concrete morphology before and after aging. The microscopic observations highlighted significant changes in the microstructure of the material after aging. These changes usually appear as cracks in the binder/hemp shive interface as surrounded in the microscopic images in Fig. 6. This is due to the swelling and shrinking phenomena and the poor adhesion between the fibre and the binder of the hemp concrete. To better show the sensitivity of these hemp shives to moisture, the expansion coefficient is calculated from the ratio of the hemp shive deformation and the difference in moisture content between RH=0% and 85%. Firstly, moisture content was deduced from literature results between a dry state (RH=0%) and a wet state (RH=85%), which corresponds to a difference in moisture content of 10% [28]. Secondly, the deformation of the hemp shives between dry and wet states was measured by ImageJ image processing software, and it is about 9.81%. Finally, an expansion coefficient of 0.95 ($\%\epsilon / \%w$) was found. This coefficient is higher than that of wood, which is about 0.4 [43]. This expansion coefficient confirms the sensitivity of hemp shives to moisture and better explains the cracks observed after aging due to several swelling/shrinkage cycles of hemp shives and their interaction with the binder.



Fig. 6. Images of hemp concrete microstructure by Keyence microscope. On the left: before aging, on the right: after aging.

In order to confirm these observations, microstructural changes were quantified. Firstly, the water porosity of both unweathered and weathered hemp concrete was measured. The results are presented in Fig. 7. The reference samples show a total porosity of 71.51%. This is in accordance with the literature [5,30]. Indeed, the porosity proportionally depends on the quantity of plant-based aggregates of the hemp concrete. In addition, the inter-particle porosity of hemp concrete depends on the size and distribution of the used hemp shives in the material [28]. This heterogeneous distribution of hemp shives in the material, as shown by particle size analysis (Fig. 3), implies a wide pore size distribution and increases the complexity of the microstructure of hemp concrete.

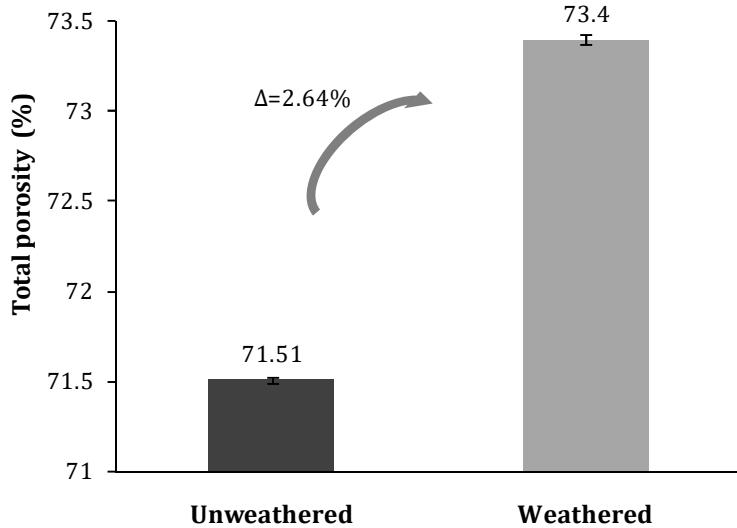


Fig. 7. Total porosity of weathered and unweathered hemp concrete.

According to the results shown in Fig. 7, the immersion/drying cycles seem to have an influence on the total porosity. An increase of 2.64% of the weathered samples compared to the reference ones is noted. This is in accordance with microscope observations that confirm the increase of the porosity that could be explained by cracks on the binder/hemp shive interface and an eventual dissolution of the hydrates initially present in the dough [44].

Indeed, a colour change of the immersion solution was observed after immersion/drying cycles. In order to confirm the hydrate dissolution hypothesis and better understand the origin of the colour change, chemical analysis of the immersion solution was therefore performed by ionic chromatography. The latter allows to identify and quantify perfectly the cations and anions present in the solution. Results of the chemical analyses performed on three solutions are presented in Table 2. They highlight the presence of calcium, potassium, sodium and magnesium in the immersion solution. These species are leached from hemp concrete (binder and/or hemp shives).

Table 2 Chemical composition of the immersion solution obtained by ionic chromatography.

Immersion solution	Ions	Calcium (Ca ²⁺)	Magnesium (Mg ²⁺)	Potassium (K ⁺)	Sodium (Na ⁺)
Without hemp/lime leachate (tap water)	Concentration (mg/L)	26.29	3.66	1.2	12
	Number of solutions tested	3	3	3	3
	Standard deviations (mg/L)	± 6.98	± 1.21	± 0.11	± 1.09
With hemp/lime leachate (tap water after aging)	Concentration (mg/l)	625.76	12.48	314.59	17.34
	Number of solutions tested	3	3	3	3
	Standard deviations (mg/l)	± 51.85	± 1.54	± 5.81	± 1.55

The pH values of weathered and unweathered hemp concrete were presented in Table 3. These results confirm the basicity of these materials. A slight reduction of 1.4% of pH is obtained after aging. In fact, literature studies have shown that fungal growth appears from pH values less than 10 [25]. In our case study, a pH value higher than 10 was obtained for both cases of hemp concrete. This confirms that immersion/drying cycles do not significantly affect the pH of hemp concrete and consequently do not promote the growth of mould.

Table 3 pH of weathered and unweathered hemp concrete.

Hemp concrete	Unweathered	Weathered
pH	11.81	11.64
Number of simples tested	3	3
Standard deviation	± 1.13× 10 ⁻²	± 1,72× 10 ⁻²

Thermogravimetric analysis (TGA) was also performed for both weathered and unweathered hemp shives and binder in order to identify the different decomposition phases of the hemp concrete (hemp shive and binder). The aim was to compare the hemp concrete phases before and after aging. Fig. 8(a) and Fig. 8(b) compare, respectively, the TGA and the dTG curves of the weathered and unweathered hemp shives.

From Fig. 8(b), a first peak of phase change between 50 and 150 °C is noted, which corresponds to the evaporation of the free water contained in the hemp shives. We also observe a main peak due to the decomposition of cellulose between 300 and 380 °C [45]. The shoulder upstream of this main peak is characteristic of the thermal depolymerisation of hemicellulose at around of 250 °C. In addition, the

TGA curve presented in Fig. 8(a) shows that the mass loss of the weathered hemp shives is slightly higher than that of the reference one. However, the peaks occurred in the same temperature ranges with slightly different amplitudes, reflecting the difference in the amount of the dehydrated compound in question.

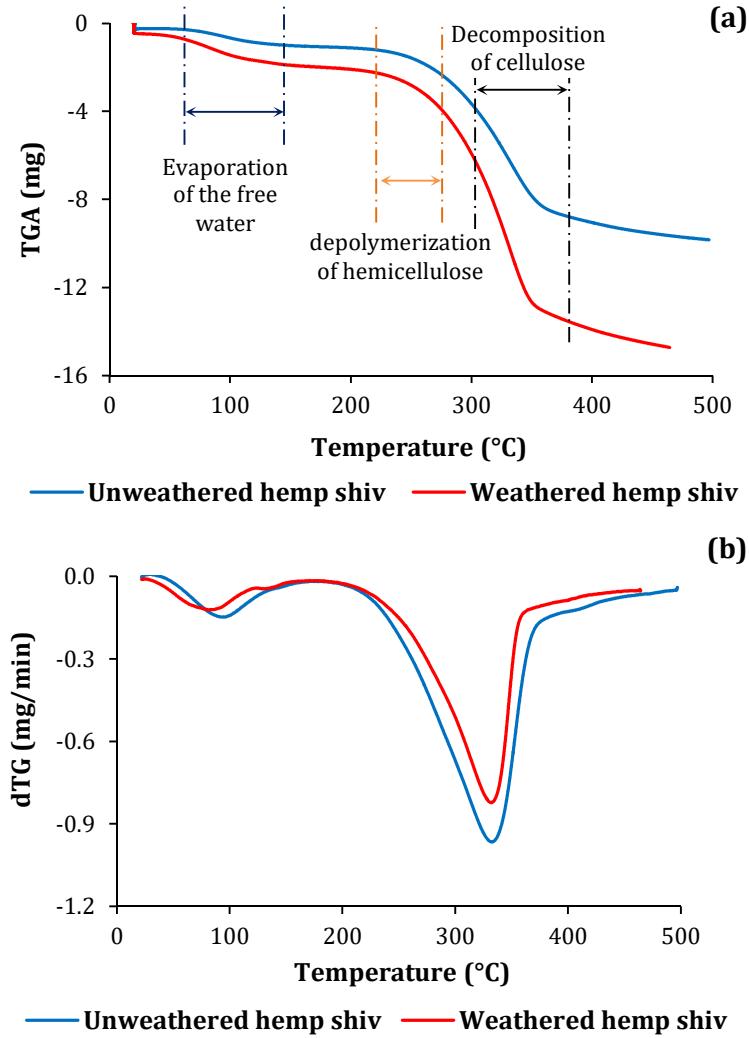


Fig. 8. TGA (a) and dTG (b) of the weathered and unweathered hemp shives.

In the same way, results of Fig. 9 compare TGA and the dTG curves of the weathered and unweathered binder. The TGA and dTG curves shown in Fig. 9(a) and Fig. 9(b), respectively, are nearly the same and exhibit identical behaviour with some slight variations in peak amplitudes. The temperature ranges of these peaks are also identical. The first peak corresponds to the departure of the free water in the pores at around 100 °C and water chemically bound to hydrates of C-S-H in continuous form between 100 and 400 °C [46]. At 480 °C, a second peak was observed. The latter is

due to the dehydroxylation of $\text{Ca}(\text{OH})_2$. Finally, a last peak occurred between 750 and 850 °C. This is due to the decarbonation of CaCO_3 .

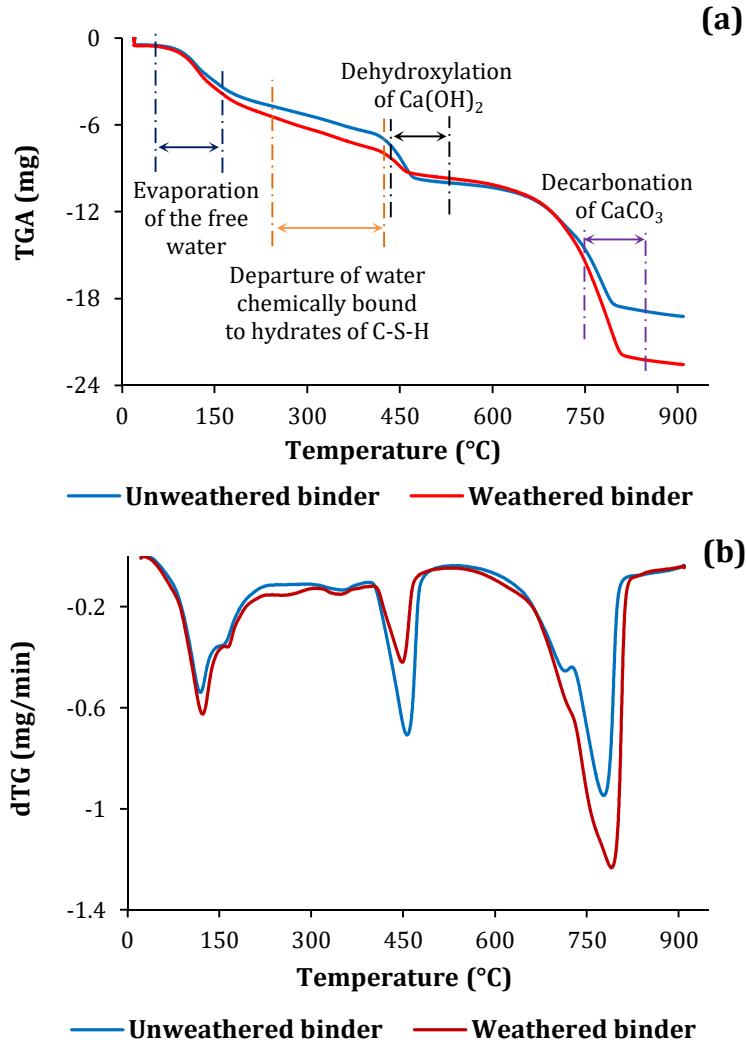


Fig. 9. TGA (a) and dTG (b) curves of the weathered and unweathered binder.

3.2. Hygrothermal characterisation

Hemp concrete is known for its low thermal conductivity, making it a good thermal insulator. This low thermal conductivity depends mainly on the type of the plant-based aggregates but also on their size (see Fig. 3) and their distribution in the material because the heat transfer in the longitudinal direction of the hemp shives is higher than that in the transverse direction [6,28,29]. The average of these measures is illustrated in Fig. 10. Results show a thermal conductivity value of 100.94 mW/(m·K) at the reference state and 94.25 mW/(m·K) at the aging one. The standard deviations are 1.28 mW/(m·K) and 0.34 mW/(m·K) for the reference and weathered samples, respectively. A decrease of 6.63% of

thermal conductivity was observed between the two states. This decrease in thermal conductivity is in agreement with the increase of the total porosity [47,48]. This is due to the modification of the microstructure of the material and the increase of its total porosity after aging as shown above (see Fig. 6 and Fig. 7). Indeed, the conductivity of air is much lower than that of hemp shives and binder, and this caused a dampening of the transmission of heat, which resulted in a decrease in the thermal conductivity of the weathered samples.

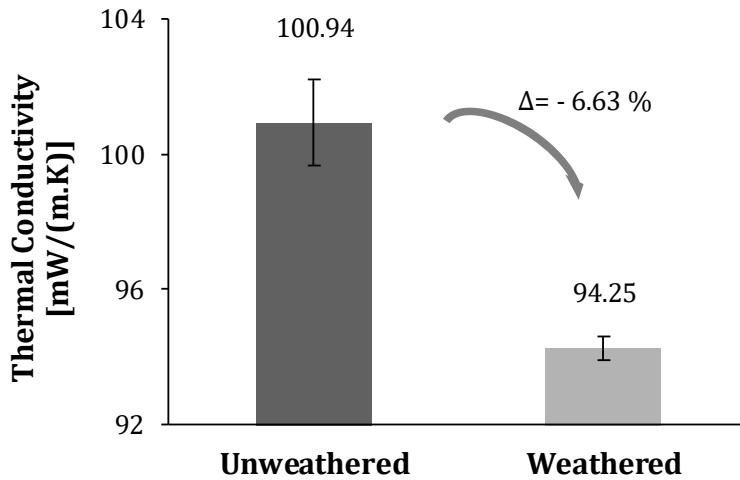


Fig. 10. Thermal conductivity of weathered and unweathered hemp concrete [mW/(m·K)].

Furthermore, the evolution of water vapour permeability was measured. Fig. 11(a) and 11(b) show, respectively, the comparison between the water vapour permeability and the diffusion resistance factor obtained. Firstly, the results obtained for unweathered samples are in agreement with the literature [6] and with the microstructure change as shown in Fig. 6 and Fig. 7.

After aging, an increase in water vapour permeability of 38.24% was observed in the weathered materials. The standard deviations of this property are 8.07×10^{-13} kg/(m·s·Pa) and 2.16×10^{-12} kg/(m·s·Pa) for the reference samples and the weathered ones, respectively. This increase led to a 27.47% decrease in the diffusion resistance factor with a standard deviation of 0.261 and 0.186 of the samples without and with aging, respectively. This is due to the modification of the material morphology and the degradation of hemp shives quality due to the immersion/drying cycles. Moreover, this can also be attributed to the increased porosity of materials after aging, which ultimately resulted in a more hygroscopic material and therefore a more permeable one.

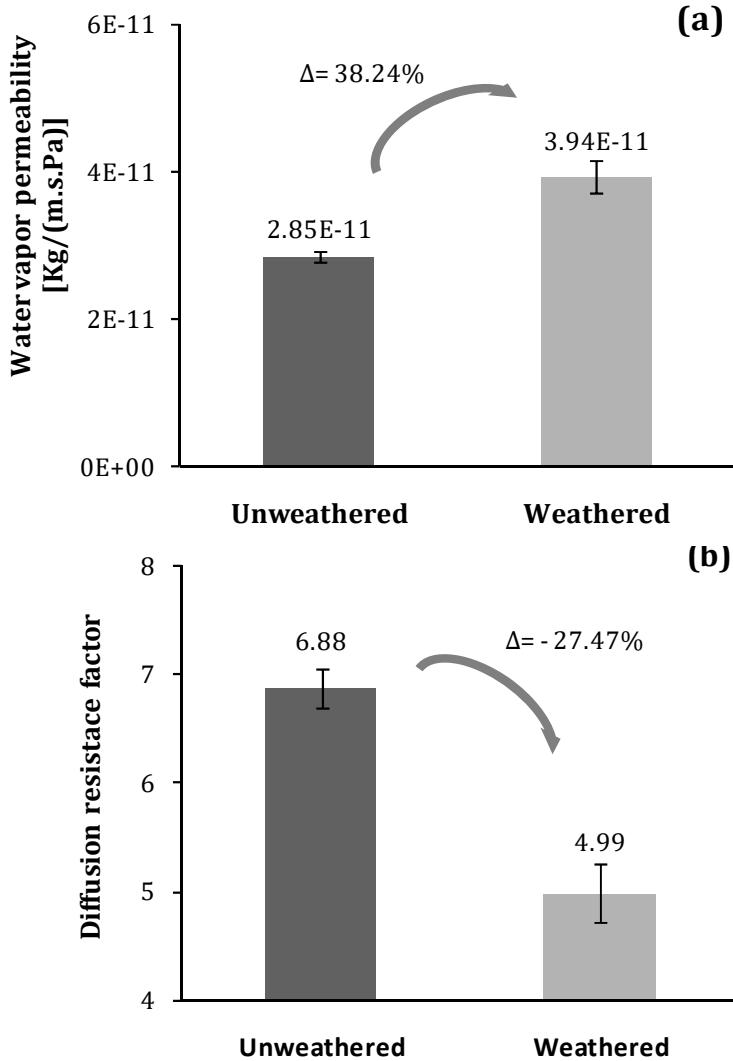


Fig. 11. Parameter values characterising the water vapour diffusion of weathered and unweathered hemp concrete: (a) water vapour permeability, (b) diffusion resistance factor.

To perform Moisture Buffer Capacity (MBV) measurements, the penetration depth was first calculated to define the thickness of the sample to be characterized. Table 4 shows the results of penetration depths of hemp concrete obtained in each case. An increase of 17.69 % of the penetration depth of weathered materials compared to reference ones is noted. This is related to the increase of porosity and water vapour permeability as shown above [49] (see Fig. 7 and Fig. 11). The increase of these two properties (porosity and water vapour permeability) promotes the mass transfer kinetics as well as the moisture variations in the material and increases consequently the penetration depth.

Table 4 Penetration depth of weathered and unweathered hemp concrete.

Material	Weathered	Unweathered
$d_{p,1\%}$ (cm)	3.74	4.39

Number of simples tested	3	3
Standard deviation (cm)	$\pm 4.91 \times 10^{-4}$	$\pm 1.14 \times 10^{-3}$

Therefore, for the study of MBV, a thickness of 5 cm, which is greater than the penetration depth, was chosen.

Fig. 12 illustrates MBV results for the two states of hemp concrete samples studied. Firstly, the MBV results of the unweathered materials are in agreement with the literature [26]. Indeed, hemp concrete has an MBV value of 2.27, which allows it to be classified as an excellent moisture regulator according to the Nordtest project classification (see Fig. 5). This MBV value may depend on the size and distribution of hemp shives in the material (Fig. 3), as these affect the moisture storage capacity of the material [29].

Furthermore, results show that the moisture buffer capacity of weathered materials decreased slightly compared to unweathered ones. This decrease is due to the aging of the hemp shives, which has caused a degradation of its capacity to adsorb and restore moisture (natural moisture regulation). However, despite a decrease of 11%, the weathered hemp concrete remains, according to the Nordtest project classification, an excellent moisture regulator with an MBV index of 2.02.

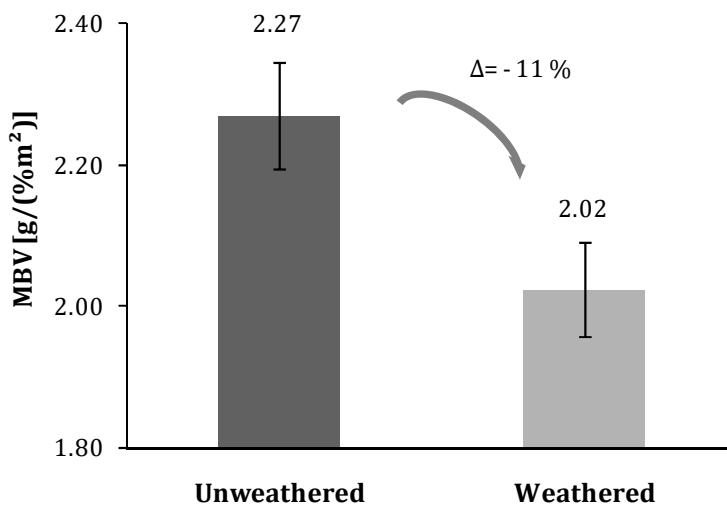


Fig. 12. Moisture buffer value (MBV) of weathered and unweathered hemp concrete.

Moreover, sorption isotherms were also evaluated for each case of hemp concrete. Fig. 13 shows moisture content as a function of the relative humidity of the weathered and reference materials. The moisture content at saturation state of the reference material (unweathered) was found to be higher

than that of the weathered one. This can be explained, on the one hand, by the size and random distribution of the hemp shives (see Fig. 3 and Fig. 6) and, on the other hand, by the aging of the material and especially by the reduction of the adsorption capacity of the weathered hemp shives and the decrease of moisture storage capacity of the weathered hemp concrete. This result confirms the decrease observed in the MBV after aging [29]. In addition, a slight difference in the hysteresis loop was noted between the two materials. The area of hysteresis loop was slightly higher for the weathered material. This is due to the microstructure modification of the material, which becomes more complex after aging. Indeed, the irregular pore size distribution can cause water entrapment in small pores because of the ink-bottle effect [50,51]. That is why the emptying of large pores, during desorption, begins only after the emptying of small pores under high capillary pressure. Therefore, this water can remain trapped in small pores as long as the capillary pressure necessary for the evacuation is not reached, and hence the difference between the hysteresis loops is noted.

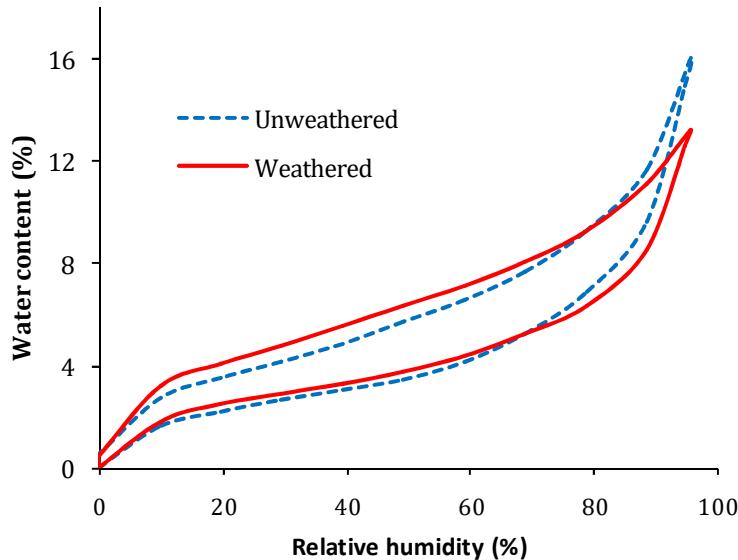


Fig. 13. Comparison of water vapour adsorption-desorption isotherms of the weathered and unweathered materials.

3.3. Compressive strength evolution

The average measurements of the compression strength are shown in Fig. 14. The results are consistent with the literature [13,40]. The compressive strength is influenced by the accelerated aging of the material. A significant decrease of 51% was recorded on the weathered materials after aging, with a standard deviation of repeatability of 0.089 MPa and 0.013 MPa for the reference and

weathered samples, respectively. This is due, on the one hand, to the degradation and debonding of the binder/hemp shive interface (cracks) after the swelling and shrinkage of hemp shives as shown above on the digital microscope images (see Section 3.1) and, on the other hand, to the porosity increase caused by the immersion/drying cycles, which significantly weakens the mechanical behaviour of the material.

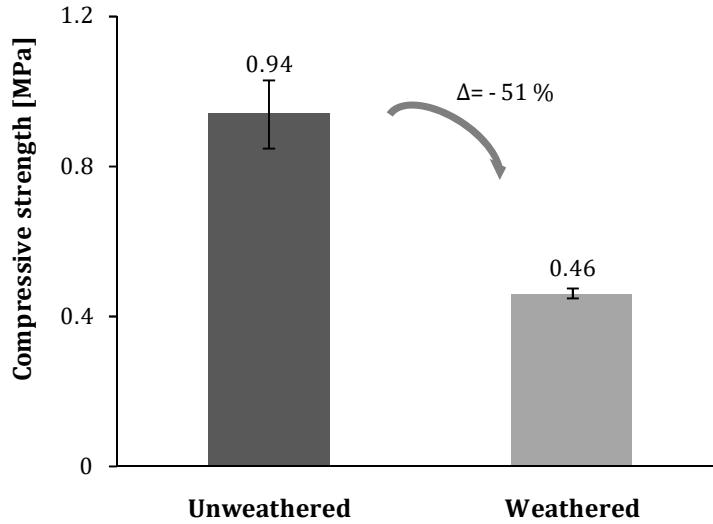


Fig. 14. Compressive strength of weathered and unweathered hemp concrete [MPa].

4. Conclusion

This work presents an experimental investigation of hemp concrete behaviour vis-à-vis the climatic conditions (simulated by immersion/drying cycles) in order to better understand its durability. For this purpose, microstructural, hydrothermal and mechanical properties of weathered and unweathered materials were evaluated. The following conclusions are drawn:

- The studied thermo-hydro-mechanical properties of hemp concrete depend on its microstructure and, mainly, the adhesion of binder/hemp shive interface. Observations with a digital microscope (Keyence) and porosity measurements showed the effect of aging on the microstructure of hemp concrete. Several cracks were found in the binder/hemp concrete interface, and an increase of 3% in total porosity was recorded. These microstructure changes engender: (i) a reduction in the diffusion resistance factor, the moisture buffer value (MBV) and the compressive strength of 51%, and (ii) an increase in the water vapour permeability and the penetration depth of the hemp concrete of 38% and 18%, respectively. However, we have

noted a positive impact of aging on the thermal conductivity of hemp concrete. A decrease of 6% of this property was noted for the weathered materials.

- Chemical analysis performed on the immersion solution confirms the presence of calcium, potassium and magnesium, which may result from the dissolution and leaching of some hydrates. This too has contributed to the microstructure changes of hemp concrete after aging. In addition, thermogravimetric analysis (TGA) has shown that the weathered and unweathered hemp shives and binder have nearly the same behaviour at high temperature. Small variations of peak amplitudes due to the difference in the amount of dehydrated compound were noted.
- Finally, this investigation showed that immersion/drying cycles do not affect the pH of hemp concrete. Both materials (weathered and unweathered) presented a basic pH of about 11, which according to the literature does not promote mould growth. However, the fungal development of the material should be monitored over time to avoid any risk of this type of material degradation.

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References

- [1] L. Melchert, The Dutch sustainable building policy: A model for developing countries, *Build. Environ.* 42 (2005) 893–901. <https://doi.org/10.1016/j.buildenv.2005.10.007>.
- [2] M. Qin, R. Belarbi, A. Aït-Mokhtar, A. Seigneurin, An analytical method to calculate the coupled heat and moisture transfer in building materials, *Int. Commun. Heat Mass Transf.* 33 (2006) 39–48. <https://doi.org/10.1016/j.icheatmasstransfer.2005.08.001>.
- [3] R. Haik, G. Bar-Nes, A. Peled, I.A. Meir, Alternative unfired binders as lime replacement in

hemp concrete, Constr. Build. Mater. 241 (2020) 117981.

<https://doi.org/10.1016/j.conbuildmat.2019.117981>.

- [4] A. Arizzi, M. Brümmer, I. Martín-Sánchez, E. Molina, G. Cultrone, Optimization of lime and clay-based hemp-concrete wall formulations for a successful lime rendering, *Constr. Build. Mater.* 184 (2018) 76–86. <https://doi.org/10.1016/j.conbuildmat.2018.06.225>.
- [5] B. Seng, C. Magniont, S. Lorente, Characterization of a precast hemp concrete. Part I: Physical and thermal properties, *J. Build. Eng.* 24 (2019). <https://doi.org/10.1016/j.jobe.2018.07.016>.
- [6] F. Bennai, N. Issaadi, K. Abahri, R. Belarbi, A. Tahakourt, Experimental characterization of thermal and hygric properties of hemp concrete with consideration of the material age evolution, *Heat Mass Transf.* 54 (2017) 1189–1197.
<https://doi.org/https://doi.org/10.1007/s00231-017-2221-2>.
- [7] F. Bennai, C. El Hachem, K. Abahri, R. Belarbi, Influence of hydric solicitations on the morphological behavior of hemp concrete, *RILEM Tech. Lett.* 4 (2019) 16–21.
<https://doi.org/10.21809/rilemtechlett.2019.80>.
- [8] T. Alioua, B. Agoudjil, N. Chennouf, A. Boudenne, K. Benzarti, Investigation on heat and moisture transfer in bio-based building wall with consideration of the hysteresis effect, *Build. Environ.* (2019) 106333. <https://doi.org/10.1016/j.buildenv.2019.106333>.
- [9] B. Seng, C. Magniont, S. Lorente, Characterization of a precast hemp concrete block. Part II: Hygric properties, *J. Build. Eng.* 24 (2019). <https://doi.org/10.1016/j.jobe.2018.09.007>.
- [10] E. Troppová, M. Švehlík, J. Tippner, R. Wimmer, Influence of temperature and moisture content on the thermal conductivity of wood-based fibreboards, *Mater. Struct.* 48 (2015) 4077–4083. <https://doi.org/10.1617/s11527-014-0467-4>.
- [11] T. Jami, S.R. Karade, L.P. Singh, A review of the properties of hemp concrete for green building applications, Elsevier Ltd, (2019). <https://doi.org/10.1016/j.jclepro.2019.117852>.
- [12] AQC, Commission Prévention Produits Mise En Œuvre (C2P): Prévention des désordres liés aux produits et procédés de construction, Publication Semestrielle., (2018).
- [13] R. Walker, S. Pavia, R. Mitchell, Mechanical properties and durability of hemp-lime concretes, *Constr. Build. Mater.* 61 (2014) 340–348. <https://doi.org/10.1016/j.conbuildmat.2014.02.065>.

- [14] L. Arnaud, E. Gourlay, Experimental study of parameters influencing mechanical properties of hemp concretes, *Constr. Build. Mater.* 28 (2011) 50–56. <https://doi.org/10.1016/j.conbuildmat.2011.07.052>.
- [15] P. Brzyski, D. Barnat-Hunek, Z. Suchorab, G. Lagód, Composite materials based on hemp and flax for low-energy buildings, *Materials* (Basel). 10 (2017) 510. <https://doi.org/10.3390/ma10050510>.
- [16] R. Fernea, D.L. Manea, L. Plesa, R. Iernuțan, M. Dumitran, Acoustic and thermal properties of hemp-cement building materials, *Procedia Manuf.* 32 (2019) 208–215. <https://doi.org/10.1016/j.promfg.2019.02.204>.
- [17] E. Latif, M. Lawrence, A. Shea, P. Walker, Moisture buffer potential of experimental wall assemblies incorporating formulated hemp-lime, *Build. Environ.* 93 (2015) 199–209. <https://doi.org/10.1016/j.buildenv.2015.07.011>.
- [18] U. Dhakal, U. Berardi, M. Gorgolewski, R. Richman, Hygrothermal performance of hempcrete for Ontario (Canada) buildings, *J. Clean. Prod.* 142 (2017) 3655–3664. <https://doi.org/10.1016/j.jclepro.2016.10.102>.
- [19] T. Colinart, D. Lelievre, P. Glouannec, Experimental and numerical analysis of the transient hygrothermal behavior of multilayered hemp concrete wall, *Energy Build.* 112 (2015) 1–11. <https://doi.org/10.1016/j.enbuild.2015.11.027>.
- [20] R. Belarbi, F. Bennai, M. Ferroukhi, C. Hachem, K. Abahri, Multiscale modelling for better hygrothermal prediction of porous building materials, 02005 (2018) 1–6. <https://doi.org/10.1051/matecconf/201814902005>.
- [21] K. Abahri, C. EL Hachem, F. Bennai, T. Ngoc, R. Belarbi, Prediction of Hemp Concrete Morphological Deformation by X-ray Tomography, American Concrete Institute, ACI Special Publication. 320 616–625., (2017).
- [22] F. Bennai, K. Abahri, R. Belarbi, A. Tahakourt, Periodic homogenization for heat, air, and moisture transfer of porous building materials, *Numer. Heat Transf. Part B Fundam.* 70 (2016) 420–440. <https://doi.org/10.1080/10407790.2016.1230393>.
- [23] A. Arizzi, H. Viles, I. Martín-Sánchez, G. Cultrone, Predicting the long-term durability of

hemp–lime renders in inland and coastal areas using Mediterranean, Tropical and Semi-arid climatic simulations, *Sci. Total Environ.* 542 (2016) 757–770.
<https://doi.org/10.1016/j.scitotenv.2015.10.141>.

- [24] L. Bessette, B. Trémerie, T. Béjat, A. Piot, A. Jay, L. Barnes Davin, Study the development of mould on prompt natural cement-based hemp concrete, in: First Int. Conf. Bio-Based Build. Mater., Clermont-Ferrand, France, (2015) pp. 142–148.
- [25] S. Marceau, P. Glé, M. Guéguen-Minerbe, E. Gourlay, S. Moscardelli, I. Nour, S. Amziane, Influence of accelerated aging on the properties of hemp concretes, *Constr. Build. Mater.* 139 (2017) 524–530. <https://doi.org/10.1016/j.conbuildmat.2016.11.129>.
- [26] M. Viel, F. Collet, Y. Lecieux, M.L.M. François, V. Colson, C. Lanos, A. Hussain, M. Lawrence, Resistance to mold development assessment of bio-based building materials, *Compos. Part B Eng.* 158 (2018) 406–418. <https://doi.org/10.1016/j.compositesb.2018.09.063>.
- [27] G. Delannoy, S. Marceau, P. Glé, M. Gueguen-minerbe, D. Diafi, I. Nour, Aging of hemp shiv used for concrete, *Jmade.* 160 (2018) 752–762. <https://doi.org/10.1016/j.matdes.2018.10.016>.
- [28] F. Bennai, C. El Hachem, K. Abahri, R. Belarbi, Microscopic hydric characterization of hemp concrete by X-ray microtomography and digital volume correlation, *Constr. Build. Mater.* 188 (2018) 983–994. <https://doi.org/10.1016/j.conbuildmat.2018.08.198>.
- [29] B. Mazhoud, F. Collet, S. Pretot, J. Chamoin, Hygric and thermal properties of hemp-lime plasters, *Build. Environ.* 96 (2016) 206–216. <https://doi.org/10.1016/j.buildenv.2015.11.013>.
- [30] M. Chabannes, F. Becquart, E. Garcia-Diaz, N.E. Abriak, L. Clerc, Experimental investigation of the shear behaviour of hemp and rice husk-based concretes using triaxial compression, *Constr. Build. Mater.* 143 (2017) 621–632. <https://doi.org/10.1016/j.conbuildmat.2017.03.148>.
- [31] M. Fourmentin, P. Faure, P. Pelupessy, V. Sarou-Kanian, U. Peter, D. Lesueur, S. Rodts, D. Daviller, P. Coussot, NMR and MRI observation of water absorption/uptake in hemp shives used for hemp concrete, *Constr. Build. Mater.* 124 (2016) 405–413.
<https://doi.org/10.1016/j.conbuildmat.2016.07.100>.
- [32] NF P18-459: Essai pour béton durci - Essai de porosité et de masse volumique (mars 2010).
- [33] NFEN12667, Performance thermique des matériaux et produits pour le bâtiment -

Détermination de la résistance thermique par la méthode de la plaque chaude gardée et la méthode fluxmétrique - Produits de haute et moyenne résistance thermique, (2001).

- [34] NFEN12664, Performance thermique des matériaux et produits pour le bâtiment - Détermination de la résistance thermique par la méthode de la plaque chaude gardée et la méthode fluxmétrique - Produits secs et humides de moyenne et basse résistance thermique, (2001).
- [35] European Standard ISO 12572 Building materials – determination of water vapor transmission properties (ISO/DIS 12572:1997) PrEN ISO 12572., (1997).
- [36] C. Rode, R. Peuhkuri, B. Time, A. Gustavsen, T. Ojanen, J. Ahonen, K. Svennberg, L.-E. Harderup, J. Arfvidsson, Moisture Buffering of Building Materials, Technical University of Denmark, Department of Civil Engineering, (2005).
- [37] C. Rode, R. Peuhkuri, B. Time, K. Svennberg, T. Ojanen, Moisture Buffer Value of Building Materials, J. ASTM Int. 4 (2006) 100369. <https://doi.org/10.1520/JAI100369>.
- [38] EN 826, Thermal Insulating Products for Building Applications—Determination of Compression Behavior, (1998).
- [39] ASTM Committee, ASTM C109/C109M-02 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars, Annu. B. ASTM Stand. 04 (2002) 1–6. https://doi.org/10.1520/C0109_C0109M-20A.
- [40] E. Sassoni, S. Manzi, A. Motori, M. Montecchi, M. Canti, Experimental study on the physical-mechanical durability of innovative hemp-based composites for the building industry, Energy Build. 104 (2015) 316–322. <https://doi.org/10.1016/j.enbuild.2015.07.022>.
- [41] J. Page, M. Sonebi, S. Amziane, Design and multi-physical properties of a new hybrid hemp-flax composite material, Constr. Build. Mater. 139 (2016) 502–512. <https://doi.org/10.1016/j.conbuildmat.2016.12.037>.
- [42] J. A. Grubb, H. S. Limaye, A. M. Kakade, Testing pH of Concrete: Need for a standard procedure, in: Am. Concr. Inst., (2007): pp. 78–83.
- [43] C. El Hachem, K. Abahri, R. Bennacer, Original experimental and numerical approach for prediction of the microscopic hygro-mechanical behavior of spruce wood, Constr. Build.

Mater. 203 (2019) 258–266. <https://doi.org/10.1016/j.conbuildmat.2019.01.107>.

- [44] D. Damidot, F.P. Glasser, Thermodynamic investigation of the CaO-Al₂O₃ CaSO₄-H₂O system at 50°C, Cem. Concr. Res. 23 (1992) 1195–1204. [https://doi.org/10.1016/0008-8846\(92\)90047-Y](https://doi.org/10.1016/0008-8846(92)90047-Y).
- [45] U. Benitha Sandrine, V. Isabelle, M. Ton Hoang, C. Maalouf, Influence of chemical modification on hemp-starch concrete, Constr. Build. Mater. 81 (2015) 208–215. <https://doi.org/10.1016/j.conbuildmat.2015.02.045>.
- [46] H. Fares, S. Remond, A. Noumowé, A. Cousture, Microstructure et propriétés physico-chimiques de bétons autoplâçants chauffés de 20 à 600°C, Eur. J. Environ. Civ. Eng. 15 (2011) 869–888. <https://doi.org/10.1080/19648189.2011.9695278>.
- [47] P. Glé, E. Gourdon, L. Arnaud, Acoustical properties of materials made of vegetable particles with several scales of porosity, Appl. Acoust. 72 (2011) 249–259. <https://doi.org/10.1016/j.apacoust.2010.11.003>.
- [48] A. Bourdot, T. Moussa, A. Gacoin, C. Maalouf, P. Vazquez, C. Thomachot-Schneider, C. Bliard, A. Merabtine, M. Lachi, O. Douzane, H. Karaky, G. Polidori, Characterization of a hemp-based agro-material: Influence of starch ratio and hemp shive size on physical, mechanical, and hygrothermal properties, Energy Build. 153 (2017) 501–512. <https://doi.org/10.1016/j.enbuild.2017.08.022>.
- [49] M. Rahim, O. Douzane, A.D. Tran Le, G. Promis, B. Laidoudi, A. Crigny, B. Dupre, T. Langlet, Characterization of flax lime and hemp lime concretes: Hygric properties and moisture buffer capacity, Energy Build. 88 (2014) 91–99. <https://doi.org/10.1016/j.enbuild.2014.11.043>.
- [50] M. Bart, Proceedings of the 4th Congrès International de Géotechnique - Ouvrages -Structures, 8 (2018). <https://doi.org/10.1007/978-981-10-6713-6>.
- [51] M. Wu, B. Johannesson, M. Geiker, A study of the water vapor sorption isotherms of hardened cement pastes: Possible pore structure changes at low relative humidity and the impact of temperature on isotherms, Cem. Concr. Res. 56 (2014) 97–105. <https://doi.org/10.1016/j.cemconres.2013.11.008>.