Impact of the Accelerated Aging Protocols on the Hemp Concrete Durability

Kamilia Abahri, Alexandra Bourdot, Sylvain Langlois, and Ghaith Alhaik

Abstract Hemp concrete has been widely recommended as bio-based material to limit carbon emissions and energy consumption of buildings. This material presents interesting hygrothermal and acoustic performances. It is produced of hemp particles embedded in a natural cement that forms a very heterogeneous and porous component. Few works have studied the evolution of the properties of hemp and flax concrete, over time. This research aims to study the evolution of the compressive strength, the microstructure and porosity of hemp concrete over the time. Thus, three accelerated aging protocols were conducted on the formulated hemp concrete before undertaken properties tracking experimentations. Results shows that compressive strength increases up to 58 days for hemp concrete. The hemp shiv deformation depending on the accelerated aging in hemp concrete is calculated by 2D image analysis. Optical investigation was used for this experimentation. Microscopic results confirm the high degradation of such heterogeneous materials which is confirmed by the porosity results.

Keywords Immersion/drying · Microstructure · Porosity · Compressive strength

1 Introduction

Nowadays, the use of materials with a low environmental impact such as bio-based mortars, already recognized for their thermal and acoustic insulating qualities and their low environmental impact, is gaining more and more place in the construction sector. In addition, they present a renewable resource, derived from plant biomass, in comparison with other building materials. They also have a weak shot and are responsible for low CO_2 emissions, which respects the life cycle.

In fact, the hygrothermal properties of bio-based mortars and especially hemp concrete has been widely studied and demonstrated. From a hygric point of view,

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E. B. Pereira et al. (eds.), *Proceedings of the 3rd RILEM Spring Convention and Conference (RSCC 2020)*, RILEM Bookseries 32,
https://doi.org/10.1007/978-3-030-76547-7_17

hemp concrete has many advantages. It has an excellent moisture buffering capacity (MBV) to maintain the quality of indoor air [1, 2]. It's very porous structure, and its total porosity close to the open porosity, make it capable of absorbing significant quantities of water [3]. In addition, hemp concrete has a high permeability to water vapor, and the property of substantially moderating the relative humidity changes of the surrounding air [4]. In general, this material can reduce daily variations in indoor relative humidity by absorbing and returning moisture, reducing energy consumption, and maintaining hygrothermal comfort in the building [5].

Thermally, hemp shives present low thermal conductivity which makes them good insulating materials that are well adapted to be associate with binder [6, 7]. The porosity of hemp concrete ranges from 60 to 85% and for linseed concrete from 70 to 90% [7, 8].

Moreover, the use of these materials is hampered, in particular, by the unavailability of databases relating to their intrinsic properties and by lack of knowledge of their behavior over time (durability). This use requires taking into account several criteria that make them vulnerable; the problem of swelling-shrinkage at the cement-fiber plant interface and fungal growth are the most important criteria for assessing the degradation of these materials. Being hygroscopic materials, they are very sensitive to changes in relative humidity, which can cause dimensional variations, which in some cases lead to their deterioration. It is therefore essential to evaluate the behavior of these materials with regard to water demands.

Regarding literature, some researchers have studied the evolution of the hygrothermal properties [9, 10]. They use different aging protocol. The most reliable protocol for measuring the evolution of compressive strength over time is to age samples in situ. This kind of protocol can take several months, even several years. To answer this problem, different accelerated aging protocol, inspired by concrete, have been put in place.

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 the degradation has been performed. Indeed, a companion of compressive strength has been carried out for referenced samples and after aging. From these tests, crucial conclusions on the durability of this material are drawn.

2 Materials and Methods

This paper proposes different accelerated aging protocol for the study of the durability of hemp concrete. First, we will have to manufacture hemp concrete samples using literature formulations. Then evaluate the porosity, the microstructure and compressive strength at a young age. Next, we will quantify these properties after accelerated aging. Compressive strength has been done after 28 days of manufacturing and at the end of each accelerated aging protocol.

2.1 Hemp Concrete Formulation

The chosen formulation is based on the formulation of the hemp concrete samples already used by researcher in order to make relevant comparison [11]. The water/binder ration is W/B = 1.45, and the aggregate/binder ration is A/B = 0.42 In addition to concrete manufactured, samples of hemp concrete with the same formulation dating from April and May were given to us in order to characterize a more aged concrete. The concrete is placed in specimens of 10 × 10 × 40 cm. After 28 days we cut cubic samples of 5 × 5 × 5 cm. The binders used in the formulation of linseed concrete must ensure a desired cohesion between the different components and thus provide a certain rigidity and mechanical strength. The choice of binder used in the composition of the assembly must consider: ease of mixing while ensuring good coating of the constituents, facilitate implementation in the fresh state and finally guarantee the mechanical properties after hardening. The benchmark binder for applications involving linseed concrete is Tradical PF70 lime, a mixture of air lime, hydraulic lime and pozzolan [12]. This hydraulic lime is characterized by a hydraulic price declaring between 3 and 5 h depending on the mixing rate. It has many physical, chemical and plastic qualities. Also, it easily adheres to different supports; It promotes water exchange, air permeability and impermeability to liquid water [9]. It is a mixture This binder consists of 75% aerated lime, 15% hydraulic binders and 10% pozzolanic binders. Its density is of 500 kg/m^3.

First of all, we prepared the necessary quantities of each component (linen, binder, water); Mixed the water and the binder until the lime swells, which corresponds to the appearance of air bubbles (estimated time between 2 and 3 min); Gradually add the flax shives while mixing. The mixture is judged close as soon as a homogeneous paste is obtained; Stop the mixer and empty the concrete into the wheelbarrow (mixing speed is 90 rpm); Start directly to fill the test pieces with a light compaction so as to have a homogeneous sample.

Rectangular samples of 40 × 10 × 10 cm were obtained after manufacturing the different sizes (medium, large, bulk) which will then be stored in a macroscopic room (50% of relative humidity and 23 °C) until the age of 28 days.

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.

Table 1 Composition of the studied materials

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

2.2 Methods

This paper proposes several accelerated aging protocols for the study of the durability hemp concrete. This step required the analysis of hemp samples porosity and microstructure and young age to perform comparison with the same samples which have been aging.

First, the NF P 18-459 standard was adopted for the water porosity measurement on a minimum of three cubic hemp concrete samples. Measuring this porosity involves determining by weighing the: apparent mass in water after immersion in water (hydrostatic weighing) of a concrete test body previously impregnated with water under vacuum; dry samples masses. The standard required to put samples in desiccators under vacuum at a constant pressure of 25 mbar (±5 mbar) during 4 h and then gradually inject water inside the desiccator ensuring that samples are covered by a minimum of 20 mm of water level during 48 h. Hydrostatic whetting at 0.01% were carried out (M_w) accompanied with air weighing (M_a). Finally, the mass after drying (M_d) (at 100 °C) was determined for all samples and when the mass becomes stable (no variation up then 0.05% between two successive weighing in an interval of 24 h) we calculate the porosity according Eq. (1):

$$\varepsilon = \frac{M_a - M_d}{M_a - M_w} \cdot 100 \tag{1}$$

Further, a microscopic characterization was done using scanning electron microscopy (SEM) to identify the hemp shiv structure and the interface between the fibers and the binders inside hemp concrete. The Keyence VHX-2000 microscope at LMT Laboratory was used to quantify the hemp concrete swelling/shrinkage and their variation through time for all samples before and after aging: at 58 days for samples stocked in normal conditions of temperature and relative humidity (50% RH and 23 °C) and also for the same samples after 30 days of accelerated aging. This comparison needs to conserve the same samples position before and after observations under the microscope.

2.3 Mechanical Tests

At the end of each protocol of aging, mechanical tests have been done on 3 samples of each formulation. The compressive strength was determined by crushing three cubes of 5 × 5 × 5 cm.

For this, the INSTRON (Fig. 1) is used with a loading speed of 5 mm/min. Before mechanical tests, microscopic observations are performed on each formulation and for each protocol. These observations focus on the evolution of space between the aggregate and the binder, also the proliferation of molds.

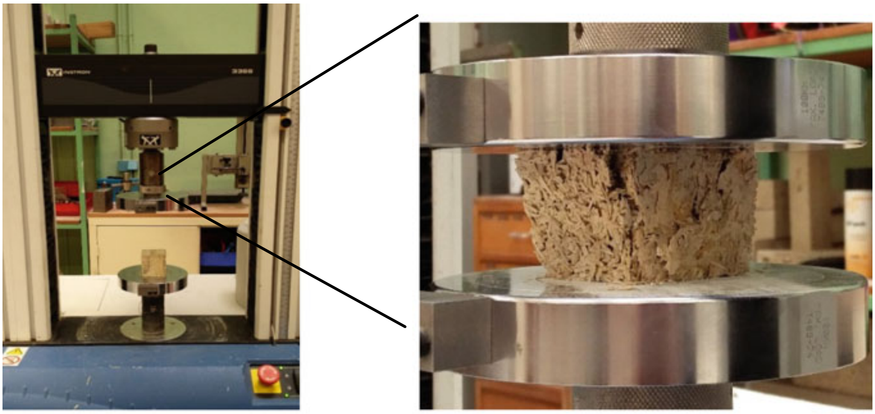


Fig. 1 INSTRON dispositive for mechanical tests: compressive strength

2.4 *Accelerated Aging Protocols*

The accelerated aging protocols were inspired from conventional protocols applied on concrete. To quantify the evolution of compressive strength over time three accelerated aging protocols were conducted in parallel on 3 specimens of each formulation as presented on the Fig. 2.

Fig. 2 Hemp concrete sample conditioned in the oven for drying (left) and immersed in water (right)

Table 2 Description of used aging protocols

Protocol	Description
Immersion/drying	This test consists of a series of immersion cycles of the sample in water for 48 h, followed by drying in an oven at 50 °C for 72 h [9, 11]
Immersion/freeze/drying	The first protocol is retained by adding a gel step between immersion and drying at −10 °C for 24 h. This protocol permits the flaking of the sample
Climatic chamber	The relative humidity is varied between 33 and 85% at a constant temperature T = 30 °C. The duration of a complete cycle is 10 days. 5 days for wetting (RH = 85%) and 5 days for drying (RH = 33%). This protocol favors the apparition of mold [11]

The immersion/drying test [9, 11], the immersion/freeze test, and the climatic chamber protocol [11]. These protocols make it possible both to make aggressive accelerated aging tests, and less-aggressive with aging in the climatic chamber. The description of used aging protocols in this study was presented in Table 2.

3 Results and Discussion

As expected in literature, hemp concrete offers a high porosity [1, 7, 8]. Concerning the studied formulation, the results are indicated in Table 3. These results obtained from 5 hemp concrete samples. This step made it possible to verify the repeatability of the tests for the different samples from the same formulation.

Microscopic observations were investigated function of the hemp concrete age. By the same as porosity, various samples were observed to guaranty the repeatability of the results. The aims of this part are to study the dimensional variation caused by the aging at microscopic scale.

During the 119 days of accelerated aging, the eventual presence of microorganisms has been detected on hemp and flax concrete. The development of mold has been observed on all formulation and for the immersion/drying and immersion/freeze protocols. Several types of mold are visible with the optic microscope. They can take the form of white “mushroom” and black/gray spots. They are visible on Fig. 3.

By the same the microscopic degradation was analyzed for different ages. Images from Fig. 4 show the morphology evolution from samples conditioned at 58 days in normal conditions of temperature and relative humidity and for the same sample after aging: 30 days of immersion. The corresponding dimensional variation were quantified the graphs of Fig. 5.

Table 3 Hemp concrete porosity

Porosity (%)	Deviation (%)	Variation coefficient (%)
77.41	0.7	0.91

Fungi appears at 119 days only for immersion protocols

Fungi appears at 60 days and visible for all protocols

Fig. 3 Fungi degradation of hemp concrete

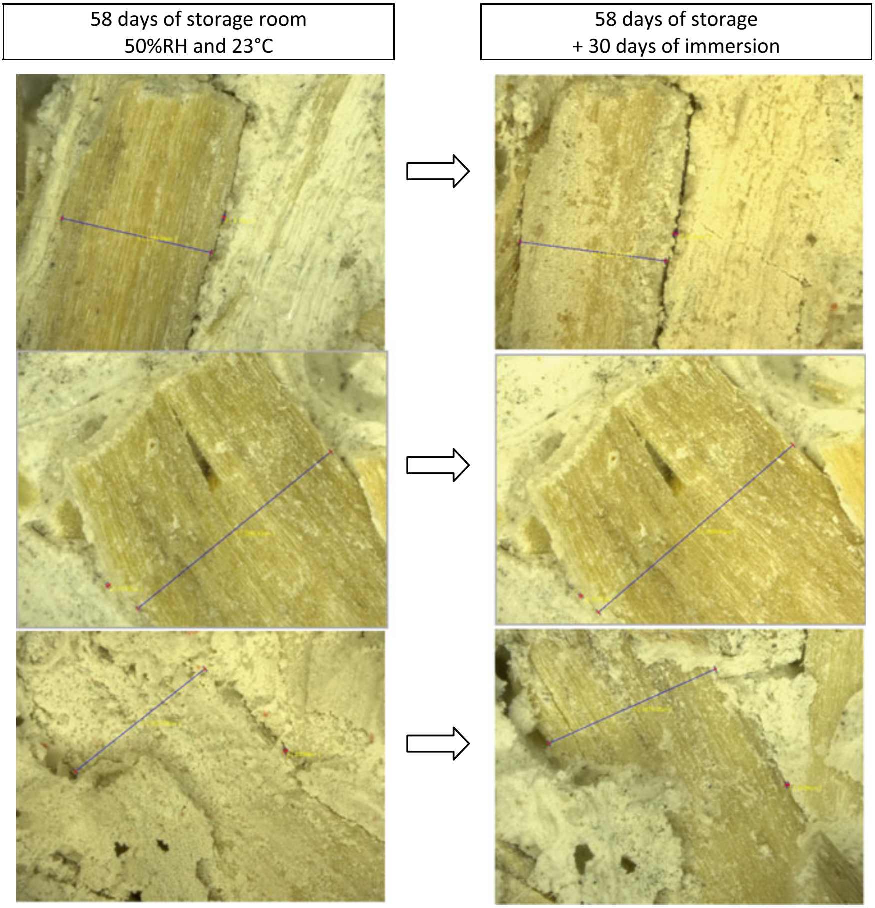


Fig. 4 Microstructural deformations before and after degradation

Fig. 5 Dimensional variation of hemp shiv after 58 days of hydrothermal aging

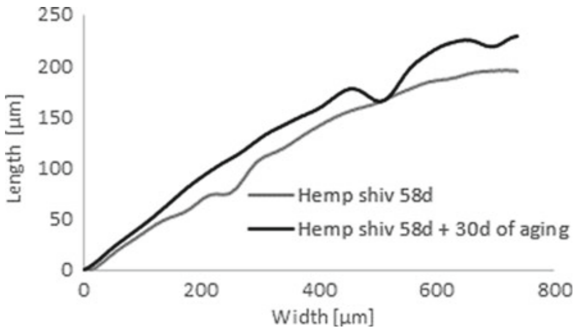


Figure 5 shows the microstructural variability of granulates profiles after 58 days of normal conditioning of temperature and relative humidity. The same position of the samples was again observed by the microscopic after 30 days of aging (without immersion) to performed correctly comparison.

From these curves we can clearly notice a difference of the hemp shiv dimension before and after aging. After aging the specimen swell and shrink representing more distance between the wooden fibers and the cementitious interface.

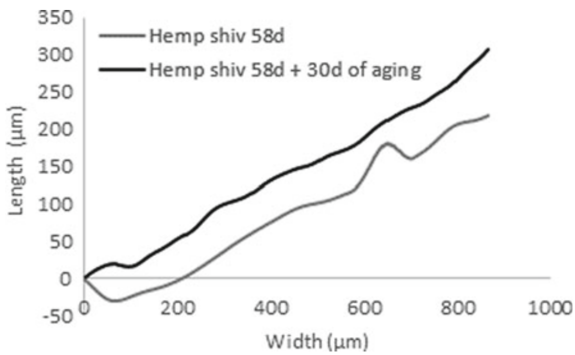
By the same Fig. 6 indicates the hemp shiv evolution, this time, after 30 days of immersion and drying from the 58 days of aging. The comparison has been done her for hemp shiv conserved at 58 days of normal conditioning and the same hemp shiv after 30 days of accolated ageing by immersion and drying.

The difference between the two cases can easily observed. For the 58 days of normal conditioning, the hemp aggregates present shrink and swelling zoon. When following this test by immersion and drying cycle the distance between the shives widens and becomes more regular because it is in the limit of the cementitious binder.

Compressive strength results

Before every test all samples are weight and the density is measured. During the experiments, the samples usually break in the direction parallel to the loading. The mean compression stresses are shown on Fig. 7 for each formulation and for each

Fig. 6 Dimensional variation of hemp shives after 58 days of immersion and drying



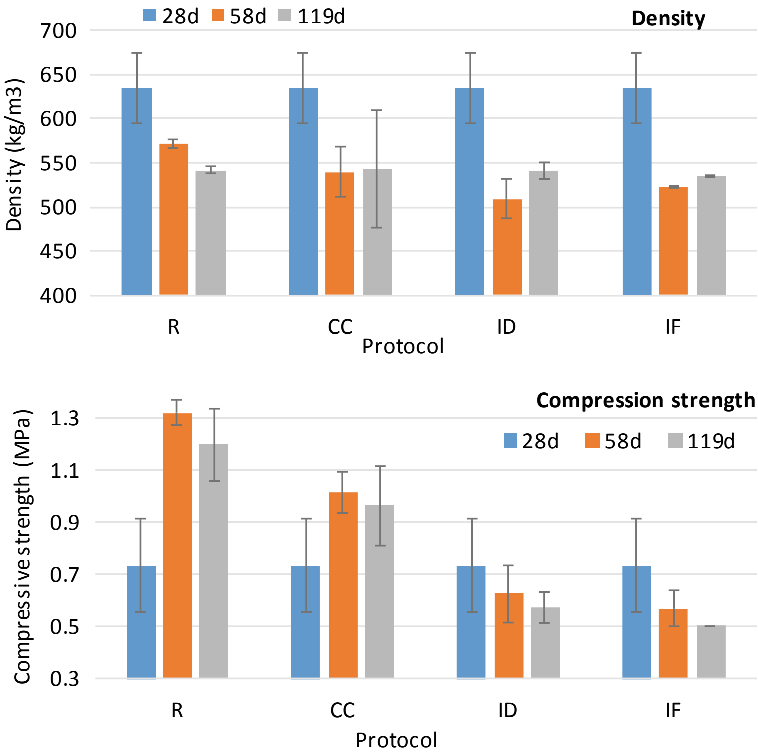


Fig. 7 Compressive strength of tested specimens over time according to accelerated protocols

aging protocol.

Literature indicate that the compressive strength of hemp concrete ranges from 0.1 to 0.7 MPa [10].

Based on results presented this figure, hemp concrete has the higher compressive strength at 28 days. Up to 58 days, compressive strength increases for all mix except for hemp concrete for the immersion/drying and immersion/freeze protocol. Up to 119 days, the strength continues to increase for flax concrete.

4 Conclusion

In this report, we have attempted to provide new information on mechanical and microstructural properties of hemp concrete under aging. The monitoring of the evolution of these properties following accelerated ageing type stresses has been attentively presented in this paper.

The microstructure of the concretes has been observed by optic microscopy. These observations show the porosity of both hemp and flax concrete, as well as the quality of the granulate-binder interface. A good cohesion is observed for hemp and flax concrete no matter their formulation.

The less aggressive accelerated aging protocol was the climatic chamber, which lead to compressive strength just under the reference one (aging in storage room). Immersion/drying and immersion-freeze lead to close value in term of compressive strength for both hemp and flax concrete.

In this article, we observed that bio-based concretes reach their maximum resistance after 28 days. Hemp concrete has a significant gain of 44% in in situ aging in compressive strength between 28 days and 58 days. Among all types of concrete seems to be the one that resists best after accelerated aging, regardless of the type of stress. Finally, it would have been interesting to consider the compaction direction. We observe that compaction of hemp concrete induces an orthotropy in the material which can cause the material to crash and not crack under a load.

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

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