



ASSESSMENT OF THE DURABILITY OF BIO-BASED INSULATING MATERIALS

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Abstract

The use of vegetal concretes, such as hemp concrete, is growing for building insulation. They are constituted by vegetal aggregates embedded in a mineral binder. Hemp concretes are multifunctional materials: they have good thermal and acoustical insulating properties and their hygrothermal behavior enables a natural moisture regulation. Unfortunately, the use of hemp concrete is limited by its lack of guarantees in terms of durability. This paper focuses on the study of the evolution of the properties of hemp concretes after accelerated ageing in laboratory. The samples are submitted to wetting/drying cycles and their performances are analyzed periodically. In this article, the properties of the materials after 75 days of ageing are reported. It is shown that this period is too short to observe any significant variation of the properties of the hemp concretes, except for their porosity. The biological ageing of the materials is investigated too. It is shown that the mold growth occurs when the relative humidity is high (98% RH) and when the pH of the binder has decreased due to its carbonation reaction.

Keywords:

bio-based materials, durability, multifunctional materials, multiscale analysis

1 INTRODUCTION

Bio-based materials are increasingly used in the construction industry. Partially or totally derived from biomass, their thermal and acoustical insulation properties are likely to improve the energy efficiency of buildings, whether for new or renovated buildings.

In particular, materials containing plant aggregates, mainly hemp in France, have been developed and are commonly used for the insulation of buildings [Amziane 2013].

Several studies have been published concerning the thermal, hygrothermal, acoustical and mechanical properties of these materials [Elfordy 2008, Nguyen 2009, Glé 2013, Gourlay 2014, Nozahic 2011]. However, their development requires providing guarantees to users in terms of durability and very few studies exist about this topic [Walker 2014].

Indeed, these materials are constituted from vegetal aggregates (hemp shiv) and a mineral binder (based on lime or cement). They are highly porous and present interesting hygroscopic properties [Collet 2014]. Therefore, their properties could evolve over time depending on their conditions of use (temperature, humidity, ventilation...) [Gourlay 2015]. For example, the growth of mold or the variation of the microstructure can be observed [Hellebois 2013] under

certain circumstances, which will have consequences on the functional properties of the materials.

The overall objective of our project is to investigate the durability of these vegetal concretes after ageing in different exposure environments corresponding to their conditions of use (variations of humidity and temperature, growth of molds...). Therefore, different types of formulations (roof, wall, render) containing various binders and vegetal granulates will be studied. Accelerated ageing protocols will be developed according to the properties of bio-based materials and to realistic environmental conditions. The evolution of vegetal concretes performances will be related to biological ageing, namely the growth of microorganisms in concretes, or be the result of environmental ageing, corresponding to temperature and humidity variations. As a result, this study will allow the development of technical recommendations both to optimize the durability of bio-based materials and to quantify the lifetime of such materials according to their exposure conditions.

This article deals with the results of the first part of the study. The influence of the type of vegetal aggregate on the properties of the hardened material is investigated. The evolution of these characteristics after a short time of accelerated ageing is presented. Finally, some parameters that may affect the mold development on the materials are examined.

2 MATERIALS AND METHODS

2.1 Raw materials

One type of binder has been used in this study: prompt natural cement (PNC). The setting of this binder is very quick and the use of a retarding agent is necessary.

Two hemp shiv, SA and SB, from different origins in France were tested (fig. 1):

- SA is obtained by mechanical defibration of hemp straw. The particles are sieved and without dust.
- SB is fibred hemp shiv.

The main characteristics of these vegetal aggregates are reported on table 1.

Tab. 1: Characteristics of the hemp shiv

| Hemp shiv | SA | SB |
|---------------------------------------|----------|----------|
| Bulk density (kg/m ³) | 95.5 ± 3 | 69.6 ± 2 |
| Initial rate of water absorption (%) | 159 | 138 |
| Skeleton density (kg/m ³) | 780 | 1080 |

Due to the presence of fibers, the bulk density is lower for shiv SB. The initial rate of water absorption is also lower for this shiv. It corresponds to the water uptake after one minute of immersion.



Shiv A Shiv B
Fig. 1: Two types of hemp shiv in bulk

2.2 Composition of concrete

Two batches of hemp concretes were manufactured according to the procedure defined in the construction rules [RP2C 2012]. Each batch contains one type of shiv and PNC in the same proportions (Tab. 2). The compaction of the samples is adjusted to obtain a density of the fresh paste of 570 kg/m³.

Tab. 2: Mix formulation of the concretes

| Component | Mass content |
|-----------------|--------------|
| PNC | 33.6% |
| Shiv | 16.8% |
| Water | 49.6 |
| Retarding agent | 0.1% |

After their manufacture, the specimens are stored in the following conditions:

- 7 days in their sealed mold
- 83 days in the laboratory atmosphere (20±2°C, 50±10% RH) after opening of the ends of the molds.
- 10 days in a drying oven at 40°C after demolding until a daily variation of mass lower than 0.1%.

2.3 Ageing protocols

After drying, the samples are submitted to two types of environments:

- the first part of the samples is stored in the laboratory (20±2°C, 50±10% RH). These samples will be used as references.

- the second part is submitted to 8 wetting/drying (W/D) cycles at 30°C and 40 and 98% RH. This temperature is chosen because it is a temperature favorable to the growth of molds. The duration of a complete cycle is 4 days and the ageing lasts 75 days.

The properties of the concrete samples are analyzed before and after these periods of ageing (100 and 175 days after their manufacturing).

2.4 Microstructure

The microstructure of the vegetal concrete is analyzed by SEM observations using a PHILIPS XL30 microscope. The samples are coated with gold.

The backscattered electrons mode has been used on polished surfaces in order to characterize the degree of hydration of the binder, the quality of the aggregate/binder interface and the porosity of the material.

An air porosimeter is used to measure the open porosity and the skeleton density of the samples [Leclair 2003, Gourlay 2015].

2.5 Thermal conductivity

The thermal characteristics of hemp concretes were determined using a transient measurement technique (hotdisk method, Fig. 2). This technique enables to measure the thermal conductivity λ . The measurements were done at 25°C and 0% RH.



Fig. 2: hotdisk apparatus

2.6 Acoustical properties

The sound absorption coefficient α and the transmission loss (TL) are measured with a Kundt tube (Akustikforschung AFD1000/AFD1200) at normal incidence between 100 and 2000 Hz [Glé 2013].

2.7 Biological ageing

Different formulations of hemp concretes have been stored at 30°C and 98% RH during several months. The eventual development of mold on the samples has been observed. The pH of the surface of the concretes has also been measured during this analysis.

Tab. 3: Characteristics of the hemp concrete formulations

| Formulation | Density of the fresh mix (kg/m ³) | Density at 100 days (kg/m ³) | Skeleton density (kg/m ³) | Porosity (%) |
|-------------|-----------------------------------------------|------------------------------------------|---------------------------------------|--------------|
| PNC-SA | 578 ± 20 | 327 | 1 535 | 79 |
| PNC-SB | 573 ± 17 | 325 | 1 210 | 73 |

3 MICROSTRUCTURE OF THE MATERIALS

3.1 Density

The densities of the concretes are reported in table 3 for the fresh mix and after 100 days of drying. These densities are very close regardless of the shiv type. However, the value of the skeleton density, measured by air porosimetry, is higher for concrete containing shiv A. Accordingly, the porosity of this concrete is higher, even if the bulk density of this shiv is higher.

The microstructure evolution of the two types of hemp concretes after W/D cycles is reported on table 4. It can be observed that the global density of the two materials decreases slightly. The same result is observed for their porosity and skeleton density.

This result could be due to the continued hydration reaction of the binder in presence of water or to the swelling of the vegetal aggregates with humidity. Chemical analyses of the materials are required to confirm these hypotheses.

Tab. 4: Characteristics of hemp concretes after W/D cycles

| Formulation | PNC-SA | PNC-SB |
|---------------------------------------|--------|--------|
| Density | 342 | 335 |
| Porosity | 75.5 % | 71.5 % |
| Skeleton density (kg/m ³) | 1 402 | 1 177 |

3.2 Microscopic observations

The microstructure of the concretes after hardening has been observed by scanning electron microscopy (fig. 2 and 3). These observations show the porosity of hemp particles, the quality of the granulate-binder interface and the binder microstructure.

Generally, a good cohesion is observed for concretes containing shiv A (fig. 2), whereas decohesions are more commonly observed with shiv B. A typical illustration of these decohesions is given on fig. 3.

These observations do not allow highlighting the different values of porosity measured by air porosimetry. No evolution of the concretes microstructure with wetting/drying cycles has been evidenced either with these observations.

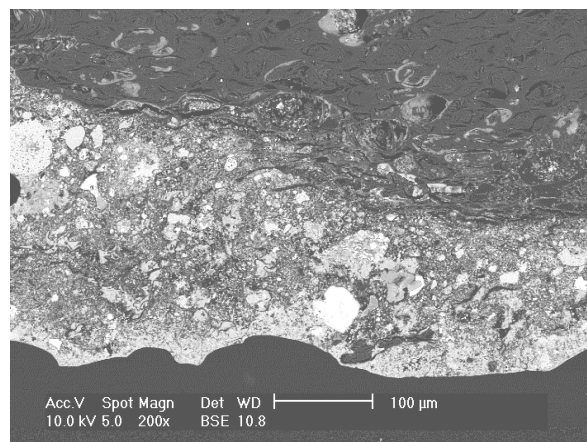


Fig. 2: Interface between shiv A and PNC observed by scanning electron microscopy (magnification: x200)

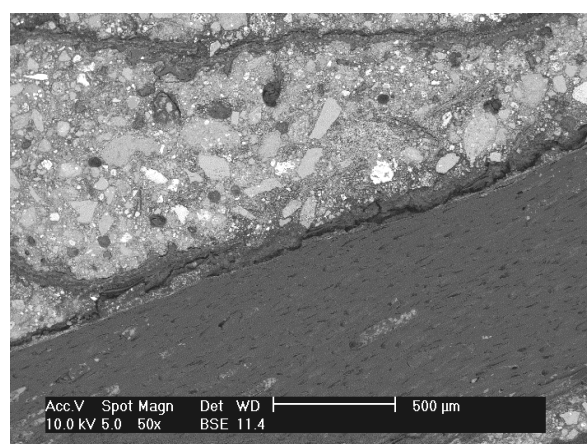


Fig. 3: Interface between shiv B and PNC observed by scanning electron microscopy (magnification: x 50)

4 FUNCTIONAL PROPERTIES

The evolution of the functional properties of the hemp concretes (thermal and acoustical performances) has been analyzed before and after wetting/drying cycles.

4.1 Thermal properties

The thermal conductivity of the concretes was measured before and after W/D cycles (tab. 5).

We can observe that the thermal conductivity is the same for the two types of concretes, because they have the same density. W/D cycles don't have any influence on these parameters.

Tab. 5: Thermal conductivity (in W/(m.K)) of hemp concretes before and after W/D cycles

| Formulation | PNC-SA | PNC-SB |
|---------------|---------------|---------------|
| Before cycles | 0.102 ± 0.006 | 0.105 ± 0.005 |
| After cycles | 0.103 ± 0.003 | 0.112 ± 0.008 |

4.2 Acoustical performances

The acoustical properties of hemp concretes have been characterized before W/D cycles, after stabilization at 0% RH (fig. 4).

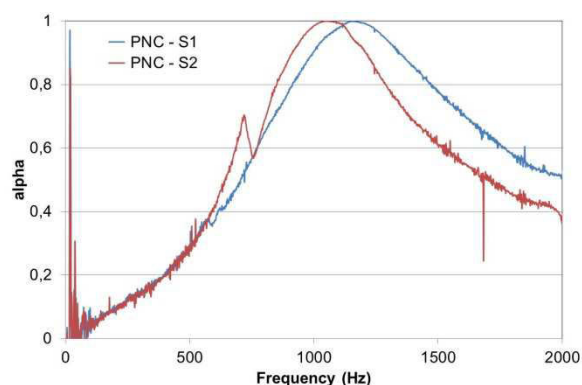


Fig. 4: Sound absorption of samples at 0% RH before W/D cycles

There is only a little difference between the two formulations of hemp concretes because their densities are very close. Generally, significant effects appear for different densities of hemp concretes [Glé 2013]. The same measurements have been performed after W/D cycles but no differences of acoustical performances are observed.

5 BIOLOGICAL AGEING

The objective of the study of mold development on hemp concrete is to analyze the conditions that can allow this growth at the surface of the materials. First, the natural growth of molds on shiv and concretes has been studied when these materials are submitted to the wetting/drying cycles.

Then, concretes aged of 14 and 120 days have been inoculated by suspensions containing three bacterial strains isolated from hemp concrete. The samples are then incubated in environmental conditions favorable to microorganisms development (30°C, 98% RH) and the presence of molds is observed periodically.

5.1 Natural growth during W/D cycles

During the 75 days of wetting/drying cycles, the eventual presence of microorganisms has been observed on hemp concretes and on samples of shiv.

The development of molds has been observed only on the two samples of shiv (fig. 5). In a previous study [Hellebois 2013], only an exposition to 30°C and 98% RH during several months led to the growth of molds on the surface of hemp concrete. Therefore, a high value of the relative humidity during a long period seems to be necessary for the molds to appear. This could explain why Walker *et al.* [Walker 2014] did not observe any microorganism development after 7 months at 30°C and 80% RH.

However, molds are present on shiv particles after these cycles. Therefore, the presence of the binder appears to limit their growth.



(a) Hemp shiv A



(b) hemp shiv B

Fig. 5: Development of molds on hemp shiv after W/D cycles

5.2 Microorganisms growth on inoculated samples

To investigate the role of the binder on the development of molds, samples of hemp concretes containing PNC binder and SA and SB shiv has been inoculated with a mineral suspension containing three bacterial strains 14 and 120 days after their manufacturing. It is known that PNC carbonates by reacting with atmospheric CO₂, leading the decreasing of the material pH. This value of pH could be an important parameter controlling the mold growth.

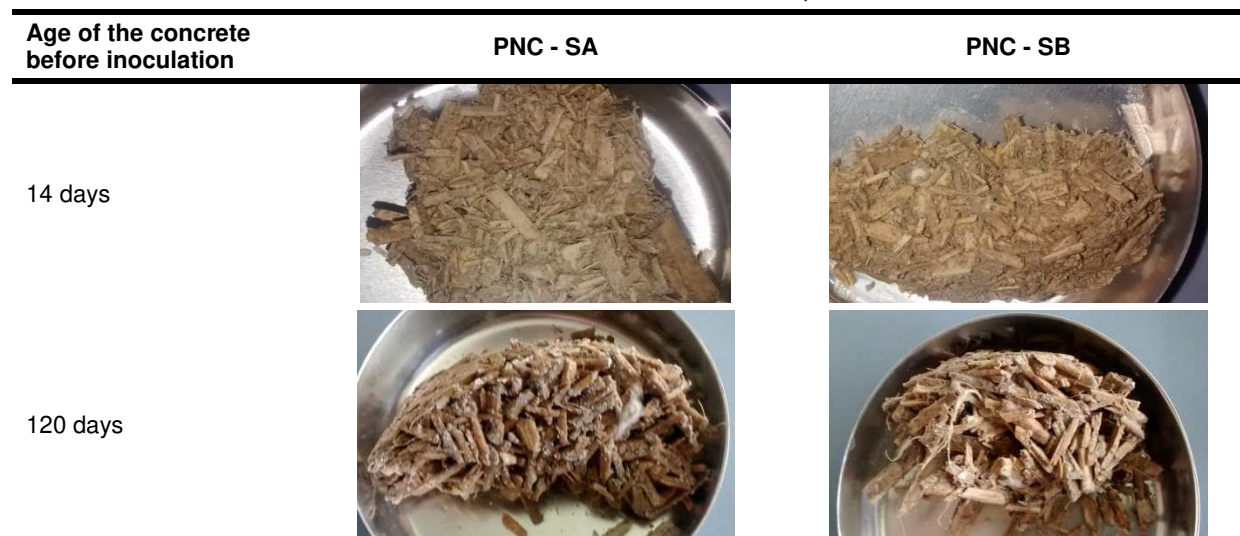
100 days after their inoculation, the presence of microorganisms has been observed and the pH of the surface of the materials measured (Tab. 6).

In table 6, it can be observed that the pH of the samples manufactured simultaneously is the same, and that it decreases with time. This result shows the carbonation of the binder. Secondly, we can notice that molds are present only on samples cast 120 days before their inoculation. The concretes have a lower pH and this value is low enough to allow the growth of microorganisms. The appearance of the molds is visible on table 7.

Tab. 6: pH of the hemp concrete samples and presence of molds

| Age of the concrete | 14 days | | 120 days | |
|---------------------|---------|------|----------|-----|
| | SA | SB | SA | SB |
| pH | 10.5 | 10.4 | 8.7 | 9.2 |
| Presence of molds? | No | No | Yes | Yes |

Table 7: Growth of molds on inoculated hemp concretes



6 CONCLUSION

In this article, the first results of a more general study on the durability of vegetal concretes for building insulation are presented. Wetting/drying cycles have been applied to hemp concretes during 75 days. After this accelerated ageing in laboratory, the performances of the materials have been analyzed. Among the measured properties, this ageing has a significant influence on the porosity of the material and on its skeleton density. No variation of the thermal conductivity or of the acoustical performance has been observed. Further measurements will be performed after one year of accelerated ageing in laboratory to observe evolution of the materials properties and validate our ageing protocol. This study has also shown that different conditions were required in order to observe mold growth: high relative humidity conditions (98% RH in this study) and a value of pH surface of the binder lower than 10. The decreasing the pH of the material is due to the carbonation of the binder. This is observed at the surface of the hemp concrete several months after their manufacturing and may lead to mold growth. All these results will be confirmed and generalized with physic-chemical analysis of the materials and other types of characterization to obtain a multiscale analysis of the ageing of vegetal concretes.

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