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FORMULATION OF BINDER TO IMPROVE MECHANICAL PROPERTIES OF HEMP CONCRETE AT EARLY STAGE. EFFECT ON THERMAL AND HYGRIC PROPERTIES IN USE.

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Abstract

Hemp concrete is a bio-based material generally made of lime-based binder and hemp shiv currently used for building envelop. This lightweight material shows a thermal conductivity about 100 mW/(m.K). On hygric point of view, hemp concrete is strongly hygroscopic, with high moisture transfer and storage capacities. These properties allow hemp concrete to moderate ambient relative humidity variations. It is an excellent hygric regulator with moisture buffer values globally higher than 2 g/(m².%RH).On mechanical point of view, hemp concrete is a non-load bearing material. It gains mechanical resistance over the first few weeks after manufacturing. These mechanical performances depend, among other factors, on the properties of components and on the mix proportioning. In order to improve the mechanical resistance of hemp concrete at early stage, this study investigates new formulations starting from reference formulation. The binder composition varies by increasing the substitution rate of lime-based binder for selected calcium sulfate-based binder. This binder leads to interesting hardening at early stage. The effect on mechanical performances at long term appears effective, without large influences on thermal and hygric properties (maximal variation about 20% and 15%). Effective proportioning tool is then available.

Keywords:

Binder formulation, mix proportioning, thermal conductivity, moisture buffer value

1 INTRODUCTION

Hemp concrete is a bio-based material used to build building envelop. It is generally made of lime-based binder and hemp shiv. This composition allows carbon and makes hemp sequestration environmentally friendly [lp 2012][Pretot 2013]. This lightweight material shows a quite low thermal conductivity (about 100 mW/(m.K)) [Amziane 2013]. On hygric point of view, hemp concrete is strongly hygroscopic, with high moisture transfer and storage capacities. These properties allow hemp concrete to moderate ambient relative humidity variations. It is an excellent hygric regulator with moisture buffer values globally higher than 2 g/(m².%RH) [Collet 2013].

On mechanical point of view, hemp concrete is a non-load bearing material. It requires large time before drying and gains mechanical resistance over the first few weeks after manufacturing. These mechanical performances depend, among other factors, on the type of components and on the mix proportioning. In order to improve the mechanical resistance of hemp concrete at early stage, this study investigates new formulations. This study focuses on coupling of two types of hydraulic binders: lime and gypsum. Starting

from traditional lime-based composition, influence of the introduction of gypsum in the formulation on hemp concretes performances are analyzed. The binder formulation varies by increasing the substitution rate of lime-based binder for calcium sulfate-based binder without changes of water and hemp shiv contents. The effect of binder formulation on mechanical properties is evaluate at short term and long term. Then, the thermal and hygric qualities of produced hemp concretes are evaluated, expecting few modification of performances. Firstly, this paper details raw materials selection and formulation of hemp concrete. Then, experimental procedure are presented. Finally, mechanical, thermal and hygric characteristics are presented and discussed.

2 RAW MATERIALS SELECTION, HEMP CONCRETE FORMULATIONS AND PRODUCTION

2.1 Binder compatibility

The lime-based binder used as reference binder is a commercial product (Tradical BCB PF70 - BCB France) commonly used to produce hemp concrete. This binder is a formulated lime composed of air-slake

lime (75%), hydraulic lime (15%) and Pozzolane: 10%. Content of surfactants is suspected. The setting time of such binder starts between 3 and 5 h, depending on the water content. Hydration of this lime leads to compression strength ranging between 2 and 15 MPa for water to binder mass ratio (W/B) ranging between 1 and 0.4 respectively. The specific density of hydrated binder is evaluated to 2300 kg/m³.

The selected calcium sulfate binder is a commercial product (Microcem 7 – Microcem France). This binder is obtained by heating of natural gypsum. The mineral phase corresponds to 50% of anhydrite β AIII and 50% of hemihydrate β . This binder is highly reactive with water. The mean grain size is 15 μm and the specific surface is 8000 cm²/g. The hydration leads to the formation of gypsum. Compressive strengths of such binder evolve from 1 to 40 MPa for W/B ranging between 1.5 and 0.35 respectively. Formulation of Microcem 7 includes setting agent leading to acceptable setting time. The specific density of hydrated binder is evaluated to 2400 kg/m³.

2.2 Shiv selection

The shiv is a commercial product (Chanvribat – LCDA France) commonly used to produce hemp concrete. Its bulk density is about 100 to 110 kg/m³. The mean width of particles (D50) is 2.2 mm and the mean length

is 8 mm. The maximal width is 5 mm and the maximal length is 19 mm.

2.3 Hemp concrete formulations and production

The proportion in mass of dry component of realized hemp concretes are presented in table 1. The two types of binders are mixed in various proportions. The proportion of Hemp shiv (HS) remains the same for all formulation with HS/B = 0.5. The water content added to the mixes is the same for all the formulation with a ratio W/B of 0.83. The flow ability of all the mixes is quite not influenced by change of binder. Then, the production process of concretes is the same for all formulations. Water and binders are mix together in a laboratory Hobart mixer (20 liters) for 2 minutes. Then hemp shiv are added progressively at low rotation speed until homogeneity of mix (maximum of 3 minutes). The hemp concrete is then poured in prismatic wood molds (30x30 cm² and 15 cm height) in two layers. Each layers is compacted with constant loading (0.05 MPa as proposed by [CenC 2007]). The molds are sealed by wood layer and placed in climatic room (23°C, RH 50%) for one week. The molds are removed and natural drying of hemp concrete blocks is started. The blocks stay in climatic room (23°C, RH 50%) and their mass evolution is recorded. After mass stabilization, samples are collected in bocks for analysis.

Tab. 1: Hemp concretes formulations (proportion in mass).

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Formulation	Binders types	Binders proportions [%]	Hemp Shiv / Binder [%]	Water/binder [%]
F0	Tradical BCB PF70	100	50	83
F1	Tradical BCB PF70 Microcem 7	66 33	50	83
F3	Tradical BCB PF70 Microcem 7	50 50	50	83
F4	Tradical BCB PF70 Microcem 7	33 60	50	83

3 EXPERIMENTAL PROCEDURES

3.1 Physical characterization

Density and open porosity of the hemp concrete are calculated from weighing of samples, measuring their sizes and from hydrostatic weighing in water after saturation. After crushing, same samples are used to measure the specific density and total porosity by immersion in acetone. SEM analysis are realized on hemp concretes to understand the effect of binder proportioning on mineral matrix structure.

3.2 Mechanical characterization

Mechanical tests are realized with a universal machine (Instron®) of capacity 400 kN controlled in displacement (cyclic loading following the rate indicated on figure 1) with a data recording at 0.10 Hz. Mechanical tests are done onto 14x14x7cm3 samples collected in hemp concrete blocks. For each composition, 8 samples are stabilized in climatic room (23°C, 50% RH). For compression tests, 14x7 cm² face of samples are loaded. The mechanical behavior of hemp concrete is characterized by large post pick plastic domain as presented on figure 2. Cyclic loading permits the analysis of the effect of loading and unloading on elastic behavior. Apparent modulus E_a and elastic modulus E_e are then distinguished.

Maximum compressive strength R_{c} and associated strain are quoted. For large strain, the residual strength level σ_{r} remains high.

3.3 Thermal characterization

The thermal characterization investigates the variation of thermal conductivity with water content. A transient method was used to not induce (or to limit) water migration during the test. The device consists in a commercial CT-meter device with a five-centimeterlong hot wire. The measurement is based on the analysis of the temperature rise versus heating time. For cylindrical geometry, Blackwell [Blackwell 1953] and Carslaw and Jaeger [Carslaw 1959] solve the equation of heat conduction for a two media system including (i) the probe, assumed as an ideal infinitely thin and long line heating source, and (ii) the studied material, that constitutes an infinite surrounding and is supposed to be homogeneous and isotropic. For a sufficiently long time, there is a proportional relationship between temperature rise ΔT and logarithmic heating time (ln(t)) (figure 3):

$$\Delta T = \frac{q}{4\pi \lambda} (\ln(t) + K) \tag{1}$$

where q is the heat flow per meter (W/m) and K is a constant including the thermal diffusivity of the material.

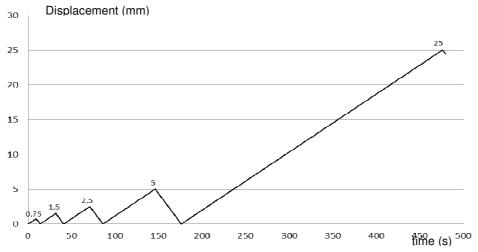


Fig. 1: Loading instruction for compression test.

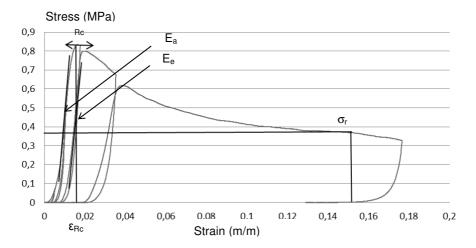


Fig. 2: Example of stress-strain curve obtained with tested hemp concrete.

The heat flow and heating time are chosen to reach high enough temperature rise (>10°C) and high correlation coefficient (R^2) between experimental data and equation (1). Consequently, the heat flow is 4.6 W/m and the heating time is 120 seconds. According to the manufacturer, the hot wire is well adapted for the

measurement of thermal conductivities ranging from 0.02 to 5 W/(m.K) and the expected accuracy is 5%.

Previously to the measurement, the specimens are conditioned at 23°C to several ambient relative humidity (dry point, 33%RH, 50 % RH and 81%RH) using dry chamber or climatic chamber Vötsch VC4060.

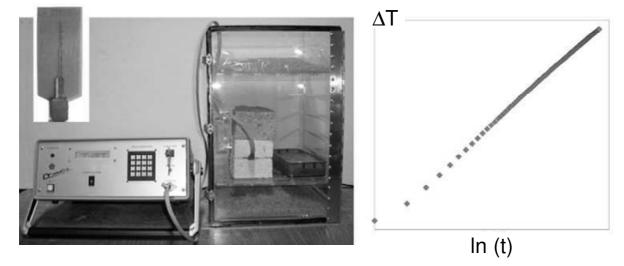


Fig. 3: Measurement of thermal conductivity at dry state left: CT Meter, Hot wire and dry chamber, right: experimental thermogram.

Tab. 2: Characteristics of specimens used for MBV test.

Formulation	Number of specimens	Average apparent density [kg.m-3]	Average exchange area [cm²]	Average thickness [mm]
F0	5	432	224.22	77.24
F1	3	423	197.56	70.19
F3	3	417	197.34	82.61
F4	3	418	197.09	70.00

3.4 Hygric characterization

The hygric characterization is based on the measurement of the moisture buffer value of materials which characterizes their ability to moderate the variations of indoor humidity in buildings.

The moisture buffer value is measured following the Nordtest protocol [Rode 2005]. Specimen are sealed on all but one surfaces. After stabilization at (23°C; 50%RH), they are exposed to daily cyclic variation of ambient relative humidity (8 hours at 75%RH and 16 hours at 33 %RH) in a climatic chamber (Vötsch VC4060). The moisture buffer value is thus calculated from their moisture uptake and release with:

$$MBV = \frac{\Delta m}{A(RH_{high} - RH_{low})}$$
 (2)

Where:

MBV: Moisture Buffer Value (kg/(m2.%RH)),

∆m : moisture uptake / release during the period (kg),

A: open surface area (m2),

RH_{high} / RH_{low} : high/low relative humidity level (%).

During the test, temperature and relative humidity are measured continuously with Sensirion SHT75 sensors and with the sensor of the climatic chamber. The air velocity is measured in the surroundings of the specimens: the vertical velocity is in the range 0.07 to 0.14 m/s and the horizontal one is 0.1 to 0.4 m/s. The specimens are weighed out of the climatic chamber

five times during the absorption period and eight times during the desorption period. The weighing instrument reading is 0.01 g, and its linearity is 0.01 g. According to the NORDTEST protocol, stability is reached when the change in mass is the same between the last three cycles with a discrepancy of less than 5 %.

Specimens are cut from blocks and are chosen to be representative of the material (density and homogeneity). The size and the density of specimens are given Table 2. According to the NORDTEST requirement, the thicknesses are chosen to be higher than the penetration depth during the test and the total exposed surface area is higher than 300 cm² for each material. The specimens are then sealed on all but one surface with aluminium tape.

4 RESULTS

4.1 Physical characteristics

Average values of densities and porosities of hemp concretes are presented in table 3. Total porosities remain quite the same for all formulations but open porosities evolve with the formulation. A minimum value of open porosity is expected between formulation F0 and F1. Figure 4 shows the state of the matrix obtained by SEM for the same magnification. Open porosity of F0 is significantly different from the others. The shape of gypsum grain is clearly visible for formulation F1 to F4. It appears that the matrix is more closed in the case of F1 formulation.

Tab. 3: Hemp concretes physical properties.

Formulation	Apparent density [kg/m³]	Open porosity [%]	specific density [kg/m³]	Total porosity [%]
F0	421.6	60.2	1996.5	78.9
F1	392.7	50.3	1937.8	79.7
F3	393.8	54.2	2025.8	80.6
F4	394.4	57.0	2051.3	80.8

Tab. 4: Average mechanical parameters obtained at 90 days, conservation at 23°c and 50RH.

Formulation	Apparent density [kg/m³]	R₅ [MPa]	E _a [MPa]	E _e [MPa]
F0	420.0	0.52	-	-
F1	428.9	0.76	95.4	155.1
F3	415.3	0.62	71.6	114.6
F4	409.9	0.56	59.2	98.4

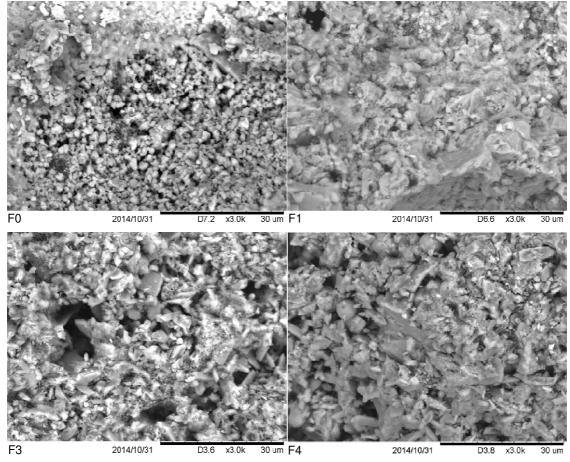


Fig. 4: SEM analysis of matrix structure.

4.2 Mechanical characteristics

Introduction of calcium sulfate binder in the hemp concrete formulation is investigated to obtain faster hardening of hemp concrete at early stage without noticeable effects on other performances of hemp concrete. The analysis of compressive strength evolution of F1 hemp concrete (with density of 405 kg/m³) is presented on figure 5. Compressive strengths remain lower than 0.6 MPa, justifying the non load-bearing ability of this material. Two hardening kinetics are visible. The hardening during the first 10 days is linked to the calcium sulfate binder hydration and drying. The second kinetic appearing after 100 days is linked to the carbonation of the lime binder.

Other tests show that for substitution rate lower than 20 %, it appears that the calcium sulfate content is too low to induce a binding effect and the short term hardening is not noticeable.

The mean mechanical parameters obtained at 90 days for all the tested formulations are given table 4.

The evolution of compressive strength versus density is presented on figure 6 (results obtained with all samples from two blocks quoted C and NC). The representative curve of reference formulation F0 stays bellow the results obtained for F1, F3 and F4 formulations. The substitution ratio of 33 % appears as the more effective to increase the compressive strength. Increase of calcium sulfate content induces a decrease of density, probably due to the change of flow ability of the mixes at fresh state leading to the increase of the open porosity.

Apparent and elastic modulus of hemp concrete

decreases as the calcium sulfate content increases. This is mainly due to the concomitant decrease of the density. The F1 formulation leads to the more ductile hemp concrete. The links between compressive strength and apparent modulus is presented on figure 7. All the results are adjusted on a linear tendency. Same type of results are obtained for compressive strength and elastic modulus. Analysis of mechanical tests results shows that a limited substitution of lime binder by calcium sulfate binder increases the mechanical performances of hemp concrete. But a too high substitution rate penalizes the properties, inducing too large density decrease. The optimum substitution rate is expected between 0 and 33%.

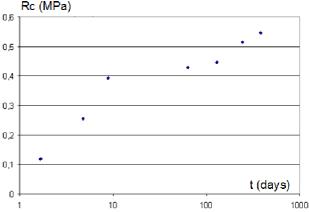


Fig. 5: Compressive strength versus time of F1 type hemp concrete with density of 405 kg/m³.

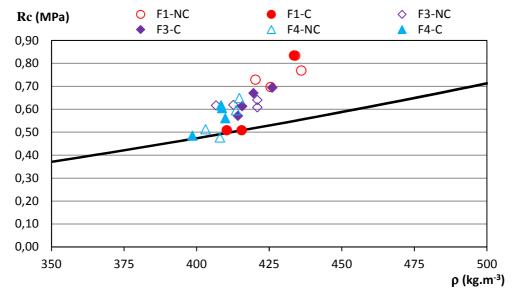


Fig. 6: Rc versus apparent density - conservation at 23°C 50% RH.

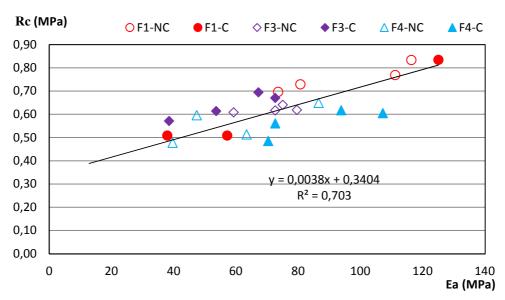


Fig. 7: Rc versus apparent modulus – conservation at 23°C 50% RH.

4.3 Thermal characteristics

The thermal conductivity of studied hemp concretes are given figure 8 versus ambient relative humidity.

At dry point, the thermal conductivity ranges from 109 to 139 mW/(m.K). The reference formulation F0 shows the highest thermal conductivity while the formulation F1, with the lowest substitution of lime-based binder for calcium sulfate-based binder, shows the lowest thermal conductivity. The thermal conductivity is thus reduced by 21%. Then, the thermal conductivity increases with the substitution rate. On the one hand, the variation of thermal conductivity is related to the density. On the other hand, it is also related to the kind of binder itself. With similar density, the formulation F0 shows higher thermal conductivity than the formulation F4. When ambient relative humidity increases, thermal conductivity increases as the water content of material becomes higher. The increase between dry point and 81%RH is about 21 % for the reference formulation F0, 33 % for formulation F1 and 25 % for formulations F3 and F4. The thermal conductivity of formulation F1 is thus slightly more impacted by ambient relative humidity.

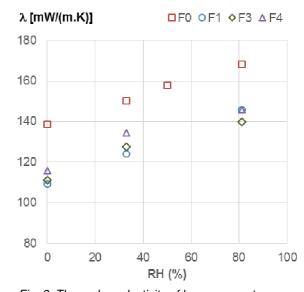


Fig. 8: Thermal conductivity of hemp concretes versus ambient relative humidity.

4.4 Hygric characteristics

Figure 9 shows the ambient relative humidity in the climatic chamber during the test. The average value of relative humidity (RH) is slightly lower than 75 % during absorption (about 71.6 %) and slightly higher than 33% during desorption (about 33.7 %) because the door of the climatic chamber is regularly open to weigh specimens (peak on the curve). The step changes in relative humidity are achieved within 30 minutes from low to high RH and within one hour from high to low RH. This induces a nearly square wave of relative humidity. It was shown in [Roels 2005] that the time needed to achieve the step in relative humidity has a limited influence on the moisture buffer value (less than 5 %) even when it takes one hour and a half to achieve the required relative humidity.

For all formulations, the measures performed on the three (or five) specimens give similar results for moisture uptake and release; and thus for Moisture Buffer Value. The declining tendency of the mass is due to the initial conditions where specimen are stabilized to 23°C 50%RH and then exposed to 75% RH. This conditions are higher than the average of the quasi steady state conditions that arise after some cycles. As shown figure 9, the cyclic amounts of moisture uptake and release are approaching each other and the change in moisture buffer value is less than 5 % within each cycle from the third cycle. The moisture buffer value is thus calculated from cycles 3 to 7.

For all formulations, the average Moisture Buffer Values range from 1.99 to 2.53 g/(m².%RH). According

to the NORDTEST project classification, the moisture buffering capacity of these formulations is thus good (1<MBV<2 g/(m².%RH)) or excellent (MBV > 2 g/(m².%RH)).

The moisture buffer value of the reference formulation F0 is 2.15 g/(m².%RH). This value is in agreement with the values given by Tran Le for similar formulation (experimental value of 1.99 g/(m².%RH) and analytical value of 2.35 g/(m².%RH)) [Tran Le 2010]. On the other hand, these values are more scattered with those given by Evrard [Evrard 2006] whose experimental value is much lower (1.75 g/(m².%RH)). This may be due to the thickness of his samples. Actually, it was shown in [Roels 2005 that the moisture buffer value drops when the thickness is lower than the penetration depth.

The moisture buffer value of formulation F1, with the lowest substitution rate, is slightly lower than the MBV of the reference (figure 10). Then, the MBV of hemp concrete made of mix binder increases with increasing substitution of lime-based binder for calcium sulfate-based binder. The variation of MBV is due to the variation of the porous network. When substituting lime-based binder for calcium sulfate-based binder, the permeability of porous network decreases for low substitution rate. Then, with higher substitution rate, the permeability increases. Finally, above a given substitution rate (50%), the increase in calcium sulfate-based binder induces an increase in Moisture Buffer Value.

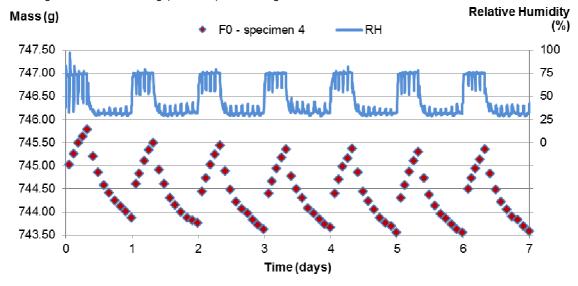


Fig. 9: Ambient relative humidity during the test and example of moisture uptake and release.

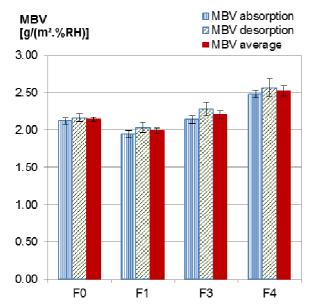


Fig. 10: Moisture Buffer Value of hemp concretes F0, F1, F3 and F4 – average value and standard deviation.

5 CONCLUSIONS

This paper relates the effect of binder composition on physical, mechanical, thermal and hygric properties of hemp concretes. The reference binder is a commercial product (Tradical BCB PF70). In order to achieve faster hardening of hemp concrete at early age, the investigated binder formulations are based on substitution of lime-based binder for calcium sulfate-based binder. A minimum rate of substitution of 20% is necessary to ensure an early stage increase or mechanical strength. Then formulations focus on substitution rate of 33, 50 and 66 %.

Such substitution impacts the porous structure of hemp concretes and thus mechanical, thermal and hygric properties. It is shown that, on mechanical point of view, the optimum substitution rate is expected to be between 0 and 33%. On thermal point of view, similar results are obtained: low substitution rates also seem to be best. Finally, on hygric point of view, opposite conclusions are outlined: highest substitution rates lead to highest moisture buffering capacity. From these results, the choice of binder formulation can be made, depending on the properties required for the final product.

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