



DESIGN OF MECHANICALLY ENHANCED CONCRETE USING HEMP SHIV

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Abstract

Hemp shiv ultra lightweight concretes are more and more used in European countries for their coupled insulation, mechanical and transfer properties. One of the scientific limitations of this material is related to the weakness of the interface zone between woody particles and mineral binder. A new way of approach to address the problem is the use of cellulose ether (CE) as an additive in order to explore to the potential of the improvement of the interface zone of between hemp shiv and binder. Two mixes were tested without CE and with 1%CE. An experimental work was carried out to investigate the binder's properties like the paste water retention or interstitial water surface tension and viscosity. The influence of CE addition on the interface between hemp shiv and hardened binder was investigated using a Secondary scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) were also conducted on selected surfaces. Two hemp shiv concrete mixes with a dry density of 490 (kg/m³) were tested. Bidirectional compression and thermal conductivity tests were carried out at 60 days. The effect of particles' orientation during manual casting was also studied. The results of SEM observations and EDX analysis showed that the transition zone contained the calcium ions (Ca²⁺) and had a porous thickness around hemp shiv of 200 μm. This transition zone is not visible when CE is added to the binder. As a result, the young modulus of hemp shiv concrete is increased by 2.5 to 5 times and compressive stress (ε=5%) by approximately 2.5 times, no matter the direction of compression loading. The thermal conductivity values in both directions of the aggregates were different. The results also underlined the interest of the angle parallelepiped aggregates during the compression loading direction, which affected to enhance hemp concrete rigidity.

Keywords:

Anisotropy, Cellulose ether, Hemp concrete, Interface, Mechanical properties

1 INTRODUCTION

The previous research on mixing a mineral binder and aggregates originated from lignocellulosic has been investigated more than one century ago with wood-cement concretes. Nevertheless, most of these studies are condensed in the last 40 years [1]. It was reported majority the delay problems or even a lack of cement setting. Regarding the hemp and lime concrete specifically, the hardening of the material surface is due to the carbonation process, which occurred whereas the binder in the core remained at powder state. The structure of these composites showed many aggregates lapping [2], which underlined a generalized interfacial problem. What can be considered as binder interfaces between particles indicated a thickness of a few hundreds of microns.

It was reported in the literature review [3] that the water absorption ability of lignocellulosic aggregates (LA) could be sometimes higher than 300 % by mass [3], which can induce important issues related to mixing

and hydration. Indeed, the water absorption is a responsible for LA swelling and lixiviation of its removables, especially hemicelluloses [4]. Concerning the binder, it induces a decrease in water available for the setting. The water transfers by capillarity and the particles diffusion in this water lead to a migration of ionic compounds like Ca²⁺ in the LA structure [4]. Some research works also underline a growth of the LA/binder interfacial porosity when water absorption ability of the LA is increased [5]. Starting from this point, several authors have undertaken to modify LA to make them compatible with hydrated mineral binder [1]. Numerous techniques has been employed: lixiviation under water, thermal treatments, surface treatments (NaOH, Ca(OH)₂...) [4], coatings (linseed oil, silicates, sucrose...). However, very few studies of using binder additives to solve LA hygroscopic problem were reported in the literature.

This work focused on the influence of the addition of the cellulose ether (CE) to pozzolanic binder on the interface shaped with hemp hurd aggregates. Several tests are carried out to determine the effect of the

interface quality on the mechanical properties of ultra-lightweight hemp concretes. Particles orientation effect of these orthotropic materials is also analysed. The choice of CE as additive was selected for its ability to increase the water viscosity. The prospected effects are multiple: increase binder's cohesion and water retention ability [6], decrease water absorption ability, limit the diffusion of binder's fine particles and ions [7]. In addition, Pourchez et al. [6] reported that the capacity of CE to limit liquid water capillarity absorption and permeability while water vapour permeability is increased regarding the hardened binders. These properties are a great of interest for the lignocellulosic concretes.

The main objective of this study was to evaluate the effect of use of cellulose ether on the mechanical performance, the thermal conductivity and to explore the potential improvement of the interface zone between binder and hem shiv aggregate and also the effect the orientation hemp shiv aggregates.

2 MATERIALS PROPERTIES AND MIX PROPORTIONS

The hemp shiv mixes investigated in this study were prepared with a local pumice filler and calcic lime (CL90). CL90 used was conformed to French Standard (NF EN 459-1). The chemical composition and physical properties of pumice filler and calcic lime are provided in Table 1. The materials was stored in a room at temperature of $20 \pm 2^\circ\text{C}$ and relative humidity of $35 \pm 5\%$.

Previous studies [3, 8] enabled to design a reference binder B-Ref characterized by Pumice/Lime and Water/Powders ratios (by mass) of 4 and 0.5, respectively. This pozzolanic binder is activated by an addition of 4% of Na_2SO_4 (by mass). The effect of CE of Methyl-hydroxyethyl-cellulose type (MHEC) is experimented with an addition of 1% (by mass of binder). This compound showed a degree of substitution (DS) of 1.3 and an average molar substitution (MS) of 0.3. The water retention was measured according ASTM C91 standard [6]. The characteristics of the binder and its interstitial water have been achieved and the water retention ability (WR) of the pastes is reported in Table 2.

To determine the viscosity, binders' interstitial solutions have been obtained by centrifugation of the fresh pastes. The analysis of the solutions was realized using a Couette rheometer (ARG2, TA Instruments). The dynamic viscosities η for a shear rate of 10 s^{-1} as well as the superficial tensions γ measured by mean of Wilhelmy plate, are also reported in Table 2. The addition of CE, which has an aptitude to form a hydrated network in the interstitial solution, enabled a large increase in water retention of the paste and the viscosity of the interstitial solution. It is coupled with a drop of superficial tension responsible for a better wetting ability of this liquid.

3 PREPARATION OF SPECIMENS AND TEST PROCEDURES

The characterization binder/hemp hurd interfaces was investigated using SEM microscopy (JEOL JSM-5910LV). The structure of ultra-lightweight hemp and lime concretes was too weak and shown too many overlaps between hemp particles to be a

representative sample. Cylindrical specimens ($\varnothing 20\text{mm}$ and height of 20mm) were then casted with the binder paste and one hemp hurd particle was introduced under vibration. Specimens were stored at $20 \pm 2^\circ\text{C}$ in autogenous conditions. After 60 days, each sample was resonated, sawed and polished in order to show a transversal section of the hemp hurd particle and the transition zone (ZT) with the binder.

The composition of the mixes is described in Table 3. CE quantity is adjusted with dry binder powder and homogenized. Two mixes are designed and assigned as C-Ref and C-CE. The mixing procedure is described in Nguyen thesis [2]. The fresh concrete mix obtained is introduced in the mold in three layers. Each layer is manually compacted to obtain a concrete fresh density of $800 \pm 10 \text{ kg/m}^3$. The specimens are removed from the mold after 24 hours. It was noticed that the adding of CE enabled an instant removing of the specimens. It is due to the higher yield stress of the C-CE mix creating a stiff consistency. The specimens are stored at $20 \pm 2^\circ\text{C}$ and $35 \pm 5\% \text{RH}$ during 60 days. It is sufficient to reach hydric stability and a dry density of $490 \pm 10 \text{ kg/m}^3$.

Manual compaction technique of the mix formed with a parallelepiped and highly anisotropic aggregates created an anisotropic material. Hemp concrete is then considered as an orthotropic composite with a granular skeleton being angled in the perpendicular plan to the compaction. To obtain information on the mechanical anisotropy of this material, cubic specimens of $15 \times 15 \times 15 \text{ cm}^3$ were made. Specimens were tested in the directions parallel ($//$) and perpendicular (\perp) to the casting compaction direction. To verify the repeatability of the measurement, three cubes were tested for each configuration.

4 RESULTS AND DISCUSSION

4.1 Effect of CE on the binder/hemp hurd interaction

The observations made on hemp hurd epidermis side demonstrated at the interface with L-Ref an increase in the porosity and showed a calcium concentration gradient within a $200 \mu\text{m}$ thickness (Fig. 1a). The lime hydrolysis driving to a fast release of Ca^{2+} and OH^- in the interstitial solution explains this targeted migration. In addition, Ca^{2+} ions are fixed in lignocellulosic plant cells, which underline this mobility [8]. It is to be noticed that silicium ions do not migrate because of the slow pozzolanic reaction releasing them in the aqueous medium. The addition of CE permitted to control the water filtration in the binder and diffusion process in liquid medium, which are responsible for calcium concentration gradient (Fig. 1b). Several elements can explain this phenomenon. The improvement of the binder cohesion is attributed to the better water retention of the paste made with CE, which resulted in better hydrated polymeric network. Additionally, the interstitial solution which transfers the capillary forces was well controlled.

On the other hand, free interstitial water obtained by centrifugation demonstrated a lower superficial tension when CE is added. Its ability to wet hemp hurd surface is then increased and grants binder/aggregate adhesion. Moreover, the viscosity growth of the interstitial solution explained the low diffusion of particles and ions by osmosis at the binder/hemp interface. This diffusion D of particles is described by

the Stokes-Einstein law equation (1), which indicates D is inverse proportional to viscosity [7] where T is the temperature, K_B is the Boltzman constant and r is the particle radius. A porous transition zone of $80 \mu\text{m}$ is observed at the interface when CE is added (Fig. 1a). It is clearly less marked than with reference binder B-Ref. The enhanced water retention ability of B-CE set

against capillarity forces carried out by hemp hurd particle. Higher water content close to the interface is then not achieved.

$$D = \frac{K_B T}{6\pi\eta r} \tag{1}$$

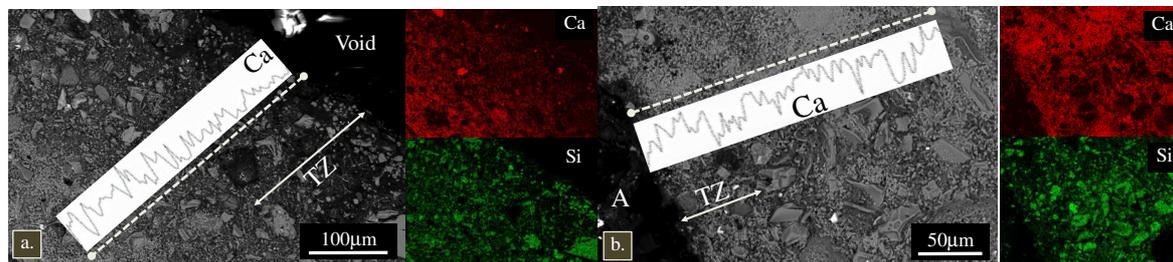


Fig. 1. Backscattered electrons observation and elementary EDX analysis (Ca, Si) of the binder/hemp hurd interface for B-Ref (a) and B-1% (b). A : Aggregate, TZ: Transition Zone

Table 1 : Chemical composition and densities of binder's raw materials.

	CaO	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	ρ_{BULK}	ρ_{ABSOLUTE}	BET surface (cm ² /g)
	% (by mass)					(kg/m ³)		
Pumice filler	1.1	62.2	17.3	5.9	6.3	1050	2750	8000
Calcic lime	95.5	-	-	-	-	450-500	2525	20000

Table 2 : Composition by densities and characteristics of fresh

Mix	Pumice filler (kg/m ³)	Calcic lime (kg/m ³)	Water (kg/m ³)	Na ₂ SO ₄ (kg/m ³)	EC (kg/m ³)	ρ_{PASTE} (kg/m ³)	WR (%)	η (mPa.s)	γ (mN.m ⁻¹)
B-Ref	866.1	216.5	541.3	43.3	--	1667	85.4	2.1±0.4*	52.9±3.0
B-CE	812.9	203.2	508.1	40.6	10.2	1575	96.3	26.9±0.6	42.3±2.7

*Confidence interval 2s (95%); **h: viscosity for a shear rate of 10s⁻¹

Table 3 : Composition by components density of C-Ref and C-CE concretes

Mix	Hemp hurd kg/m ³	Binder + (CE) kg/m ³	Water (pre-wetting) kg/m ³	Water (convenience) kg/m ³	ρ_{FRESH} kg/m ³	ρ_{DRY} kg/m ³
C-Ref	150.1	312.2 + (0)	187.6	150.1	796±16*	484±10*
C-CE	149.5	311 + (3.1)	186.9	149.5	810±12*	498±14*

*Confidence interval 2s (95%)

4.2 Compression tests perpendicular and parallel to the casting direction

C-Ref and C-CE mixes have been tested in two directions. Classically, hemp concrete is solicited mechanically in parallel direction to the casting direction. Compression load is then applied perpendicularly to the parallelepiped aggregate orientation. On the contrary, when the test is done perpendicularly to the casting direction, angled hemp hurd particles are strained widely in their longitudinal direction (having higher rigidity, Fig 2). In this configuration, the interfacial forces could be more intense. Observation of the compression behaviour in both directions // and ⊥ to the casting shows important differences (Fig. 2). A compression test in ⊥ direction of the casting resulted in an elastic modulus E and

compressive strength $f'_{c-1\%}$ (at 1% strain) 2 to 4 times higher than those in // direction of casting (Fig. 3). In other ways, a compression preferentially in the direction of the aggregates' longitudinal orientation led to an increase in stiffness. This observation can be linked to the wood behaviour for which the mechanical properties were more than 10 times higher in the direction of the stem or trunk. However, analysis of the resistances $f'_{c-5\%}$ (at a 5% strain) demonstrated values of stresses of 5 to 10% higher when the concrete is compressed in // direction of casting. In fact, the compression in this configuration generated a crushing test of the ductile particles when the binder bridges are disrupted. Conversely, ⊥ compression led to a stress plateau of 2 and 3% strain. It is due to binder/hemp interface breaking as a result of shearing.

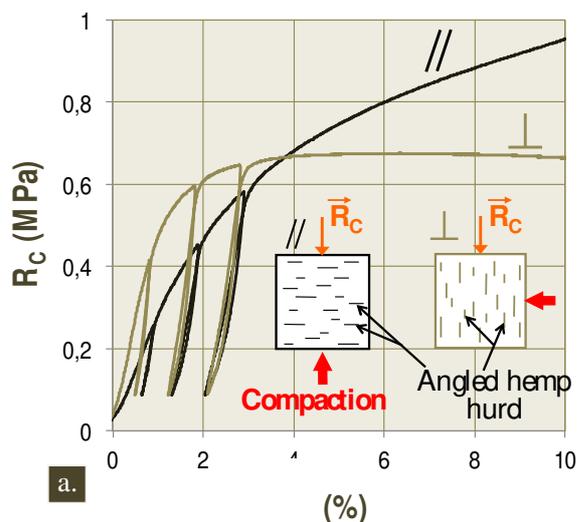


Fig. 2. Compression behaviour of mixes C-Ref and C-CE vs. load directions ($//$ and \perp) (a). Influence of loading compression direction

Hemp concretes are often used as wall filling insulation materials. Thermal conductivity has been measured with the hot wire test in the two orthotropic directions of the specimen. Results showed that when hemp

particles were oriented vertically, the thermal conductivity values were higher approximately 15 to 20% than those in \perp direction (Tab. 4).

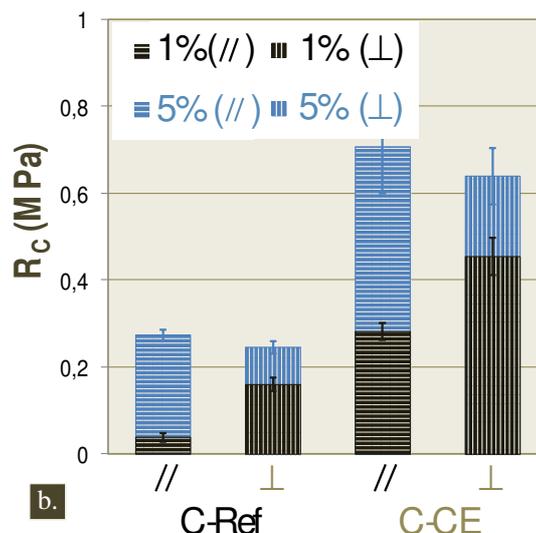


Fig. 3. Influence of loading compression direction and addition of CE at 1 and 5% strain (b).

Table 4 : Mechanical and thermal characteristics of C-Ref and C-CE concretes compressed in the directions \perp and $//$ to the casting direction

Mix	Comp. direction	f'C-1% (MPa)	f'C-5% (MPa)	E (MPa)	$\lambda_{35^{\circ}\text{HR}}^4$ ($\text{Wm}^{-1}\text{K}^{-1}$)
C-Ref	$//$ ¹	0.055 ± 0.033	0.274 ± 0.013	5.6 ± 0.3	0.131 ± 0.01
	\perp ²	0.16 ± 0.015	0.245 ± 0.015	20 ± 3.3	0.114 ± 0.02
C-CE	$//$	0.282 ± 0.02	0.707 ± 0.11	29.6 ± 3.4	0.136 ± 0.02
	\perp	0.455 ± 0.04	0.64 ± 0.066	53.2 ± 7.3	0.115 ± 0.03

¹ Compression parallel to the casting direction

² Compression orthogonal to the casting direction

³ Confidence interval 2σ (95%)

⁴ Thermal conductivity λ is measured in the direction orthogonal to the compression one, which corresponds to the usual direction if considering a hemp concrete wall

5 CONCLUSIONS

Compression tests carried out in the two orthotropic directions of hemp concretes indicated that the rigidity was 2 to 4 times higher when the compression is realized perpendicularly to the casting direction. It corresponds to a compression test parallel to the hemp particles packing principal direction. At the same time, the thermal conductivity in perpendicular direction of casting were 15 to 20% lower than those in parallel direction (orthogonal to the compression stresses when considering a wall).

The use of cellulose ether (CE) of MHEC type enabled to improve (3 times higher) the mechanical properties (f'c, E) of the hemp concrete designed during this work (490kg/m^3). This result is achieved without any rise of the thermal conductivity. Mechanical improvement of the material is mainly due to the solving the problem powdering effect of hemp concrete. The observation of the interface between B-Ref and a hemp hurd particle demonstrated a double gradient of porosity and calcium concentration of $200\ \mu\text{m}$ thickness around the particle. This weakened transition zone explains

why the interfacial and thin binder links are powdered in the final hemp concrete C-Ref.

Addition of CE enabled to reduce drastically the calcium concentration gradient and to limit the porosity growth. The analysis of binder at fresh state enabled to propose two explanations:

1) The increase of water retention ability of CE underlines the structural thickening of the free water and the improvement of the cohesion of the binder. Water losses by filtration are then prevented.

2) The interstitial water viscosity improvement by a factor of ten (for $\dot{\gamma} = 10\ \text{s}^{-1}$) leads to significant reduction of the ionic activity in the liquid medium (Stokes-Einstein law).

Finally, the simultaneous use of CE with the perpendicular direction of the hemp concrete specimens during the compaction resulted in an increase by ten time of the elastic modulus.

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6 ACKNOWLEDGMENTS

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