Impact of a microwave treatment of hemp fibers on the hemp / cement interaction

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ABSTRACT As the need to reduce grey energies is growing, materials such as hempcrete are in development. Its good thermal and acoustic insulation, combined with the very low carbon footprint of hemp make it a first choice material. However, the great amount of hemp in hempcrete, combined with de weakness of the interface fibers/cement binder, lead to poor mechanical resistance of hempcrete. This is due to a significant absorption of water by hemp and by components such as lignin, or hemicellulose, which slow down the setting of cement. This induces a poor transfer of the applied stress and therefore poor mechanical resistance in compression tests. Several chemical and chemical & physical treatments have been investigated to solve this problem. This study focuses on the impact of a treatment based on a microwave heating on the characteristics of hemp and hempcrete. The influence of such treatment was evaluated in terms of water absorption, index of crystallinity, halo of hydration, and mechanical properties.

Key-words: hemp ; hempcrete ;microwave, adhesion, treatment, cement binder.

I. INTRODUCTION

As the global warming is currently at stake, scientists around the world are working in order to find new innovative solutions to tackle this problem, and civil engineering is not left behind. Indeed, new building materials made with vegetal raw materials are expanding. For instance, hemp is being used to develop a concrete where hemp hurds – the woody fibers of the plant – will replace mineral aggregates.

Hemp is a plant which as many sustainable properties such as inexhaustible resources, low water consumption for its crop, and reduction of harmful emissions as it stores CO_2 during its whole life time (which is not case of other plants which absorb CO_2 only during their growth) [Niyigena et all. 2016]. Moreover, the main asset of hemp as a bio-aggregate in building material is its natural ability to isolate thermally and acoustically and its permeability to water vapor.

The use of such bio-aggregate material enables to conceive lightweight concrete made principally of hemp. Indeed the ratio of natural fibers over binder is about 0.4 which means that the numerous fibers induce low mechanical resistance. However, hempcrete have good thermal and acoustic insulation properties [Niyigena et all. 2016]: about 0.065 W/m.K whereas the thermal conductivity of cellular concrete is around 0.12 W/m.K. Moreover, the great ability of hemp to catch or release water is relevant in two cases: first, it allows the building made of hempcrete to regulate its water storage with respect to the air conditions in order to regulate humidity; and secondly, it has been proved that the water absorption decrease considerably with an efficient physical or chemical treatment (as by increasing the bond, the buffering effect of hemp decrease), which makes it a good indicator. Thus, hempcrete is a low grey energy material (90 kWh/m3) and its assets allows this construction material to be useful both for new constructions and rehabilitations.

However, this material and other bio-aggregates have a poor adhesion between their fibers and the cement matrix, which does not lead to sufficient mechanical properties [Schwarzova et all. 2015]. Indeed this weak adhesion is due to the high moisture sorption of this material, decreasing the hydration rate of cement, which leads to a poor transfer of the applied stress and therefore a poor mechanical resistance at compression tests: average maximum resistance around 0.405MPa [Niyigena et all. 2016].

Contrary to bast fibers (outside part of the stalk), hurds are less composed of cellulose (crystalline phase), but more of hemicellulose and lignin (amorphous phase) [Schwarzova et all. 2015]. Those two former components are responsible for the hydrophility of hemp and the capture of calcium ions (responsible for the hydration of cement) which leads poor adherence between fibers/cement binder, and some tools [Le Troedec et all. 2008] enable the monitoring of their loss such as the Fourier Transforms Infrared spectroscopy (FT-IR), X-ray Diffraction (XRD) [Dai, 2010], MEB, DTA Thermal Analysis etc.

In order to tackle the non-profitable amorphous phase, several chemical treatments were tested [Le Troedec et all. 2008] such as alkalinization with NaOH, EDTA, or PEI, etc. In addition to chemical treatments, physical ones may be used to accelerate the elimination process as the heating by microwave or ultrasound [Mistik et all. 2016]. Here, physical treatment is coupled with alkalinization treatment (NaOH) or treatment with ethanol, formic acid or acetic acid. Microwave process happened to be more efficient than ultrasound, when coupled with those chemical treatments on Kenaf fibers. Thus, it tackles the adhesion problem by removing the non-profitable amorphous phase and cleaning the surface of the particles [Mistik et all. 2016].

The aim of this paper is therefore to test the influence of a physical treatment, using as the only treatment process a microwave, on the adhesion problem and on the thermo-mechanical properties of hempcrete. In order to demonstrate the impact of micro waves on hemp fibers' surface, several tools were used: FT-IR spectroscopy on particles and pellet, X-ray Diffraction – particles and pellet as well, water absorption tests on samples of 25g and finally Diquelou tests on pellets [Diquelou et all. 2015].

II. Material and method

A 25 kg of hemp has been sampled according to the RILEM recommendations into 16 bags of 1.2 kg which were sealed. Three kinds of hemp samples have been investigated: a reference sample untreated, a dry sample treated with microwave and a saturated sample treated with microwave. Before any treatment or test, hemp shives were put in an oven at 60°C for a week to become dry. Saturated hemp was obtained by placing dry hemp into water for 6 days. After that, dry and saturated hemp fibers have been treated 2'30 in the microwave at full power (800W). Then saturated hemp fibers have been placed in an oven at 60°C for two weeks in order to dry. The table 1 sum up the parameters chosen for the two treatments. The hemp which served as a reference was only dried at 60°C.

TABLE 1. Treatment

Sample name	Reference	MO_800W_25g_2'30"_dry	MO_800W_90g_2'30"_saturated
Temperature of drying	60 °C	60 °C	60 °C
Pre-treatment	-	-	6 days in water for saturation
Quantity of treated hemp	-	25g of dried shives	90g of saturated shives
Volume of treated hemp		215 cm3	215 cm3
Microwave treatment	No	Yes	Yes
Power	-	800 W	800 W
Duration	-	2'30	2'30

In order to test the efficiency of those two treatments, several analytical tools have been investigated such as FT-IR spectroscopy, X-ray Diffraction (XRD), Optical microscope, Water Absorption tests as well as a Diquelou test [Diquelou et all. 2015] on particles and pellets. Based on FTIR and XRD analysis crystallinity index has been investigated according to [Le Troedec et all. 2008]. For Diquelou test, the cement paste is made with CEM I 52.5 R Vicat and the ratio w/c is 0.5. Finally, hempcrete (made with the same cement, with a ratio fibers / cement of 0.4 and a ratio w/c of 1) has been poured in cubic molds of 7 cm length. Six molds have been poured for each of the tree following type: the dried and unmodified hemp, the dried and treated hemp (MO_800W_25g_2'30"_dry) and the saturated and treated hemp (MO_800W_25g_2'30"_saturated). Each time, four specimens were used for the compression tests at 21 days and two were used for thermal conductivity with a Hot Disk disposal.

III. Results

From a microstructure point of view, hemp does not show any obvious modification which could be demonstrated by X-ray diffraction or by infrared spectroscopy. Indeed, the XRD diagrams remain amorphous and always present two broad peaks at 18° and 22° without additional peaks. The variation of the index of crystallinity [Dai et all. 2010], calculated from XRD analyses (Figure 1 (a)), is not significant enough to emphasize a change in the microstructure of hemp. The absence of any structural modification of hemp is also confirmed by the water absorption test (Figure 1 (b)) as it remains stable according to the reference.

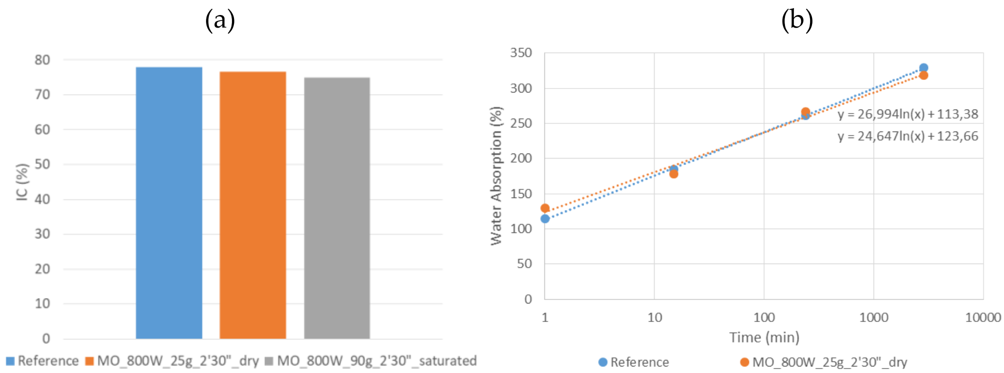

FIGURE 1. **(a) Index of crystallinity based on XRD and (b) Water Absorption of hemp fibers**

In order to investigate the interaction between hemp fibers and a cement paste, Diquelou's test has been performed with the three kind of samples. The Diquelou's test determines the perturbation area induced by hemp fibers in the hydration of the cementitious paste. In all cases, the area of halos remains similar to the reference one, which allows once again to conclude that no great modification has occurred with those two treatments. Besides, the mean maximum mechanical stress obtained for the reference is around 0.2 MPa and the thermal conductivity around 0.199/m.K.

IV. Conclusion

The whole set of tests converge to the same conclusion that there is no difference before and after a treatment of 2'30 with a microwave at 800 W. Indeed, the non-profitable amorphous phase are not altered, and the water absorption which should decrease considerably with an efficient treatment shows no difference. Finally, the Diquelou test, which requires great modification of the halo area to be relevant, shows no accurate results. For further investigations, it will be interesting to focus on the impact of the mineral binder chosen.

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