



**HAL**  
open science

# INVESTIGATION ON THE PERFORMANCE AND DURABILITY OF TREATED HEMP CONCRETE WITH WATER REPELLENT

C Sentenac, M Sonebi, S Amziane

► **To cite this version:**

C Sentenac, M Sonebi, S Amziane. INVESTIGATION ON THE PERFORMANCE AND DURABILITY OF TREATED HEMP CONCRETE WITH WATER REPELLENT. ICBBM 2017, 2nd International Conference On Bio-Based Building Materials, Sofiane AMZIANE And Mohammed SONEBI, Jun 2017, Clermont Ferrand, France. hal-01576193

**HAL Id: hal-01576193**

**<https://hal.science/hal-01576193>**

Submitted on 22 Aug 2017

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

## INVESTIGATION ON THE PERFORMANCE AND DURABILITY OF TREATED HEMP CONCRETE WITH WATER REPELLENT

C. Sentenac<sup>1,2</sup>, M. Sonebi<sup>1\*</sup>, S. Amziane<sup>2</sup>

<sup>1</sup> Queen's University Belfast, School of Planning, Architecture & Civil Eng., Belfast, BT7 1NN, UK

<sup>2</sup> Université Blaise Pascal, Institut Pascal, Polytech Clermont-Ferrand, 63174, Aubière, France

\*Corresponding author; e-mail: m.sonebi@qub.ac.uk

### Abstract

Hemp concrete is an organic based building material. Hemp concrete principal weakness point is the mechanical performance such as the compressive strength but the lack of information concerning the durability performance of this material can also be an explanation for the professional's reluctance to use bio-based concrete.

This study focused on the effects of aggregate treatments, water repellent and two different binders. The mechanical performance such as the compressive strength at 7, 14 and 28 days were done. The results shows that with the same density treated samples get a lower strength in compression. Then hydraulic performance was tested with the capillarity test and highlighted the great decrease of sorption coefficient on treated samples. Finally the durability performance tests included the full water immersion test, and the carbonation test in accelerated chamber. Weathering results are better for treated sample, which are less absorbent and were less destructive for specimens. Treated samples carbonated slightly more slowly than no-treated ones.

### Keywords:

Aggregates treatment, Hemp shiv, Durability, Mechanical performances, Water absorption, Water Repellent

## 1 INTRODUCTION

Hemp concrete principal weakness point is the strength in compression but the lack of information concerning the durability performance of this material can also be an explanation for the professional's reluctance to use bio-based concrete [Amziane 2016]. These ten last years have conducted several studies on hemp concrete to improve its properties this included the binders effect on mechanical strength [Lawrence 2007], [Pavia 2014] but also the compaction effects [Nguyen 2009] [Sonebi 2013]. Matrix additions and aggregates treatments were also conducted to improve hemp concrete performances [Magniont 2010], [Nozahic 2012]. The durability and cure conditions stay the less studying properties [Chabannes 2015], [Sonebi 2015], [Castel 2016] [Amziane 2016] but this lack of information can be an explanation for the professional's reluctance to use bio-based concrete

The selected factors considered in our study to be investigated were the effects of aggregates treatment: water repellent (WR). Few studies have already be conducted on this treatment [Falchi 2015], [Izaguirre 2015] but in our case hemp concrete has been made with two different binders: Cement Vicat (V) and a

composite binder based on hydraulic lime (C). The mix composition for this study was similar for all mixes and each treated aggregates type was compared to a normal hemp (NH) concrete sample for every performance. In total, four mixes were investigated:

- WRV : Vicat binder with water repellent treatment on hemp shiv
- NHV : Vicat binder with normal hemp shiv
- WRC : Calcic lime binder and additions with water repellent treatment on hemp shiv
- NHC : Calcic lime binder and additions with normal hemp shiv.

## 2 MATERIALS, MIX PROPORTIONS AND METHODS

### 2.1. Raw materials

#### ▪ Natural particles

Hemp shiv was used for this work. Hemp shiv used in the experimental program was Yorkshire hemp in UK. It is a hemp aggregate made from the inner woody core of the hemp plant's stem. Hemp is chopped, graded and de-dusted to give a natural, sound and breathable product. This type of boon is compatible

with lime-based binders and is marketed for individual housing construction in hemp concrete.

- Mineral binders

This investigation used two different binders:

The first binder used in this study was Vicat Cement which had an NF: P15-314 (natural prompt cement) and NF P15-317 (offshore work).

This binder has been chosen for its great properties of setting and hardening, this cement showed a highly hydraulic binder, contrary to ordinary cement.

The chemical composition is the originality of Vicat Cement even if it is very close to the Portland Clinker composition. Clay proportion is about 22% to 35%. The second binder is a hydraulic lime (75%) mixed with fly ash (15%) and cement Portland (10%). This composite binder was chosen to be compare to Vicat cement, calcic lime is very crumbly by is one so fly ash and cement Portland can improve the hardening

### Treatment

The water repellent used for experimentation is a white cream based on silane/siloxane. It supposes to penetrate deeply into silicate based mineral substrates like brick, mortar and roofing tiles. The one used in the investigation is a one coat application, it is supposed to reduce the rain penetration protects against freeze and thaw damage. The surfaces, which will welcome the treatment, have to be dry before the application and after it should dry uniformly during 24h to 48 hours.

## 2.2. Test methods

### 2.2.1 Mix design

According to the French professional rules for hemp concrete structures, Construire en Chanvre [Construire 2012], and also Amziane and Arnaud dosages based on experience [Amziane 2013] shuttered wall mix composition was tested in this investigation.

The proposed mix proportions are based on the ratios presented in Fig. 1. These ratios are used for each mixture presented previously: ERV, NHV, WRC and NHC.

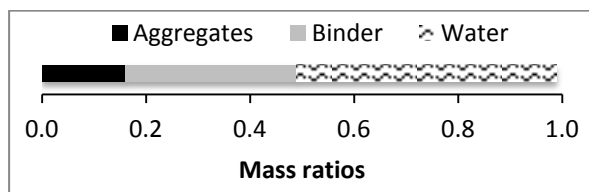


Fig. 1 : Ratios used in mixtures

### 2.2.2 Particle size distribution

There are two ways to carry out the particle size distribution :

- Mechanical sieving

A horizontal circular motion has been used and a vertical motion is created by a tapping impulse. The vibration process needs more time for vegetal particles than minerals aggregates because of their elongated shape

The minimum time of vibration should then be 30 minutes, however the vibration frequency does not influence the results. According to the literature, hemp shiv obtained by mechanical sieving method was not exact because there were many hemp particles passing through a given sieve that had length much larger than the sieve aperture. This is caused by

particles form much longer than larger [Kashtanjeva 2015].

- Image analysis

This method consisted to scan the particles of hemp shiv and use the picture in software Images Analysis in order to calculate different parameters (diameter, area etc.) [Amziane 2017]. This method was very accurate but it took a long time to carry out the experiments.

### 2.2.3 Water absorption of particles

Water absorption test is particularly interested in our case to compare treatments effects on particles water absorption.

According to working group RILEM TC 236 "Bio aggregates based buildings materials" experimental protocol for measuring the water absorption of vegetable aggregates tests have to be done three times on each specimen.

### 2.2.4 Bulk density

The bulk density of hemp shiv is linked to the porosity of the particles and to the inter-particle porosity. On her study, F. Collet use different type of mould, relative humidity and laboratory to compare the bulk density. The results are close to each other and give a bulk density value of 112kg/m<sup>3</sup> (6.7kg/m<sup>3</sup> deviation)

$$\gamma = \frac{M_s}{M_w} \times \rho_w$$

The bulk density of hemp shiv was the mean value of the measurements made on three different samples. The RILEM technical committee protocol involves measuring the weight and volume of a sample of bulk bio-aggregates to deduce the density.

### 2.2.5 Water repellent treatment process

The water repellent (WR) used is a cream so to make easier the application on aggregates it was mixed with water. Therefore for 50g of WR 50g of water is added, substance concentration is 50%. As the linseed oil a 0.5 mass ratio is used for aggregates application that means 0.25 efficient ratio of WR to aggregates mass. The following process is :

- 1) Weight 100g of dry hemp shiv
- 2) Put the sample in the mixer bowl and add 50g of Water Repellent (50%)
- 3) Mix the mix until all particles are emerged by WR
- 4) Keep the mix in a tray and wait 5 days for complete drying, do not forget to mix the sample every days if it is possible to ensure the homogenous of drying process

Repeat the stage 1 to 3 until the particle quantity is obtained.

### 2.3. Mix procedure, casting and curing conditions

In the total, four mixes were prepared in the laboratory using a mixer. The materials of mixing include hemp shiv, binders, and water. The mixing procedure is listed below:

- 1) Firstly, wipe the mixing bowl with wet tissue to moist the surface prior to mixing.
- 2) Then introduce the hemp shiv in the recipient and add progressively ¾ of the water quantity during the mixing (2min30s)
- 3) After the binder is added and mix during 30sec with the hemp wet
- 4) Finally the rest of water is added and mix during 2min to get hemp concrete.

- 5) The samples were mixed in total of 5 minutes.
- 6) The mix can be put in the different moulds, it have to be done carefully in three layers and each layers is compacted with 25 strokes distribute homogenously on the layer.
- 7) In order to prevent from excessive loss of water due to evaporation, the specimens in the mould was covered with plastic cling. Lastly, once the casting is done the specimens are stored in the control room of  $20 \pm 2^\circ\text{C}$  and its relative humidity is about 55 to 65%. Demoulding is carried out after 3 days of casting.

The concrete was poured in the different moulds (50 x 50 x 50 mm, 50 x 50 x 200 mm or 100 x 100 x 100 mm) and compacted in three layers by using a steel manual device (Fig. 2). The height of a single layer is equal to one-third of the total height of the concrete specimen (50 or 100 mm). The first and second layers have been scratched to obtain a good grip surface for the next layer. Specimens were demoulded after 48 h and stored in a climate-controlled room at  $20^\circ\text{C}$  and 60%RH.

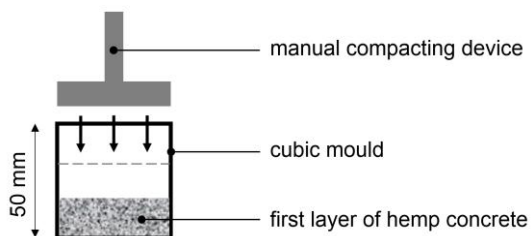


Fig. 2: Compaction process [Page 2015].

## 2.4. Test procedures

### 2.4.1. Compressive strength testing

The compressive strength was determined by crushing three cubes of 50 mm size, by using an electromechanical testing machine (Zwick). Displacement control tests were conducted with a loading rate of 3 mm/min.

### 2.4.2. Capillarity absorption test

Capillary absorption is used as an indicator of the degradation of building material. It is defined as residual spaces occupied by original kneading water and capillary coefficient in cementitious and depends upon the water/binder ratio. In our case this ratio is the same so this test will enable us to compare the real effects of treatments on aggregates.

The test method consists in expose only one face of the sample to the water to determine the rate of absorption of water by the hemp concrete.

Capillary absorption is made on  $100 \times 100 \times 100$  mm cube specimens, which are tested at 14, 28 and 60 days after manufacture. During this curing time samples are placed in a control room at  $20^\circ\text{C}$  of temperature and a relative humidity of 55%.

The protocol of capillarity test is presented as follow:

- 1) Samples are prepared by applying one waterproof tape around the circumference of the future expose face it enables water to be absorb only by one face of the sample.
- 2) Two steel bars with the same diameter are placed in a plastic tray, then the sample is put on these bar with the face exposed to water

downwards. It also enables water to be absorbed by this unique face.

- 3) Then the tray is filled with water until cubes are immersed in a constant level of water: 8mm.
- 4) After that cubes are weight at different timing 1min, 2min, 3min, 5min, 15 min, 30 min, 1h, 2h, 3h and 5h. The mass allowed determining the quantity of water absorbed throughout the test.

### 2.4.3. Weathering test

Basically, this test is used to measure the volume and weight change of the hemp concrete under several drying and wetting cycles. It enables to simulate the weather conditions, at the end of all the cycles the influence of these cycles on the compressive strength is measured.

#### • Full immersion test

Samples used in this case are 100mm cubes, four samples for each mixed are needed: two in the air and two for weathering cycles. The protocol used is describes as follow:

- 1) After 3 days of casting, the specimens are removed from the mould and cured in the control temperature room for 28 days.
- 2) Firstly, the test procedure was to place the weathering cubes in ventilated oven to dry at  $40^\circ\text{C}$  for 48 hours, the others samples stay in the air.
- 3) Then, the mass was measured after 48 hours (drying state) and the specimens were placed in water bath for another 48 hours. The specimens are fully immersed in a room temperature of  $20^\circ\text{C}$ .
- 4) Basically, the drying and wetting masses of specimen are measured after 48 hours of the one cycle.

### 2.4.4. Carbonation

Carbonation is a progressive reaction beginning at the surface of the concrete and penetrating deeper into it with time due to carbon dioxide diffusing into the concrete. The carbonation front within the concrete can be defined as one, which advances with time.

In our case the difference between samples with accelerated carbonation and sample in the air is investigated.

Twenty days after removing samples out of the mould and curing in a temperature of  $20^\circ\text{C}$  and relative humidity of 55%, they are weight and the half is put in the oven. Two size of sample is used, 50mm cubes and 100mm cubes. The carbonation process consist in measured the carbonation stage of each sample after different times.

For 50mm cubes carbonation propagation has been measured after 3, 6 and 9 weeks using 4 samples of each mix at each time. For 100mm cubes the phenolphthalein test has been done after 6weeks in the chamber.

The chamber used had a controllable internal environment in terms of temperature, relative humidity and carbon dioxide concentration. The environment was set to a temperate of  $20^\circ\text{C}$  ( $\pm 1^\circ\text{C}$ ), relative humidity of 65% ( $\pm 1^\circ\text{C}$ ) and a concentration of  $\text{CO}_2$  of 5% ( $\pm 0.5^\circ\text{C}$ ).

After completion of carbonation period each specimen was split and the fractured face sprayed with a 1%

phenolphthalein in methylated spirit from Davidson & Hardy.

As traditionally the spraying identification is used on a freshly fractured concrete surface with phenolphthalein indicator dissolved in alcohol. It results in a colour change at a pH of approximately 9 ( $\pm 0.5$ ). The phenolphthalein halfway stage occurs for a pH about 9 ( $\pm 0.5$ ). The region appears unstained when pH is under 9 ( $\pm 0.5$ ) whereas it is stained in pink when pH is beyond 9 ( $\pm 0.5$ ).

### 3 RESULTS AND DISCUSSION

#### 3.1 Characterization

##### 3.1.1. Water absorption of particles

In this part the water absorption was measured on normal hemp shiv (NH), treated hemp shiv with water repellent (WR). Fig. 3 shows the variation of water absorption

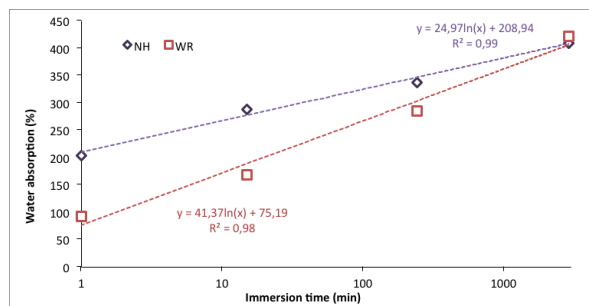


Fig. 3 : Water absorption of particles in function of time

This graph (Fig. 3) is very interested because it reveals a defect of WR treatment. Actually the treatment has good effects at first but with the time it enables water to enter in particles:

- After 1min of immersion the WR is the less absorbent with less than 100% of absorption (92.9%), compare to 204% for NH.
- Finally after 48 hours WR is the same as NH. The WR treatment is not effective with time. After 48 hours WR had absorbed 255% and NH/WR 410% almost the double.

According to the literature review, water absorption of bio-aggregates is a recurrent problem. The water absorbed by the particles is not available for the binder hydration this is the reason why this investigation is based on aggregates treatments. These first results can conduct to primary conclusions and hypothesis. The coating is just a thin layer, which can be affect, and worn with time therefore the water enter in particles, especially during this test because of the spin salad. When aggregates are spin they lost little by little the water repellent coating, this is the reason why at the end of the test water absorption rate is the same for NH and WR.

##### 3.1.2. Bulk density

The bulk density on treated hemp shiv and normal hemp shiv were measured to compare the weight added or not on our samples. Following the process described on previous chapter we have measured the bulk density of three samples for each type of hemp. An average can enable us at the end to obtain the bulk density.

The bulk density obtained for normal hemp was around  $100.9 \text{ kg/m}^3$ , with a standard deviation of  $4.1 \text{ kg/m}^3$ . Our results are lower than literature results, the usual value is around  $110 \text{ kg/m}^3$ , these results can be explain by the important quantity of fibres contained in the hemp shiv of this study as we saw previously in the particles size analysis.

In the case of WR the bulk density obtained was  $104.2 \text{ kg/m}^3$ , with a standard deviation of  $1.5 \text{ kg/m}^3$ . This result is very close to the normal hemp shiv density, the WR treatment do not add a significant mass to the particles. If the standard deviation is taken into account there is no convincing difference.

Results reveal that there is no mass added with WR repellent as it is just a coating. The density of specimens has been link to hemp shiv properties on every stage of the project.

#### 3.2 Treatment effects

##### 3.2.1. Compression strength

###### • Density

Before every test all the samples are weight and the density is measured. Fig. 4 shows the density value for each mix at each test time, respectively 7 days (7d), 14 days (14d) and 28 days (28d). In every mix of each binder the density has been reduced with time, the most significant decrease is for the WRC :  $421 \text{ kg/m}^3$  at 7 days and  $248 \text{ kg/m}^3$  at 28 days (namely  $174 \text{ kg/m}^3$  of loss). This loss of mass is important for the rest of the results and might affect compressive strength. The less consequential reduction is observed on NHC mix with:  $374 \text{ kg/m}^3$  at 7d and  $335 \text{ kg/m}^3$  at 28d (namely  $39 \text{ kg/m}^3$  of loss).

Fig. 4 highlights the fact that all mixes with Vicat have a largest density than mix with calcic lime. For example at 28d the difference in percentage represents 9% for NH and 32% for WR between Calcic mix and Vicat mix. Therefore WR treatment gets again the largest values of mass lost. All these observations have to be taken into account for compression strength comparison.

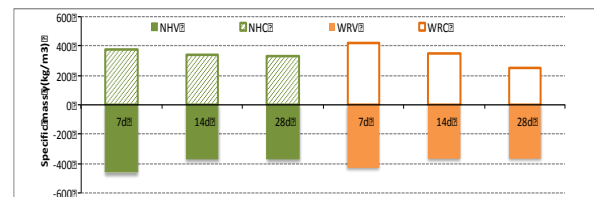


Fig. 4 : Samples density before compressive test

###### • Compression strength at 5% of deformation

Fig. 5 shows the strength in compression of every mix at three different times: 7d, 14d and 28d. In every graph curves of samples with mixed lime are represented in dotted line and Vicat ones with continue line. The first one represents the compression strength at 5% of deformation (2.5mm in case of 50mm cubes) with one curve per mix and the second graph is a columns representation of the same results. Compressive solicitation on hemp concrete can increase as infinity way but the mechanical solicitation range is between 0% and 5% of deformation, it represents the limit of service [Nozahic 2012].

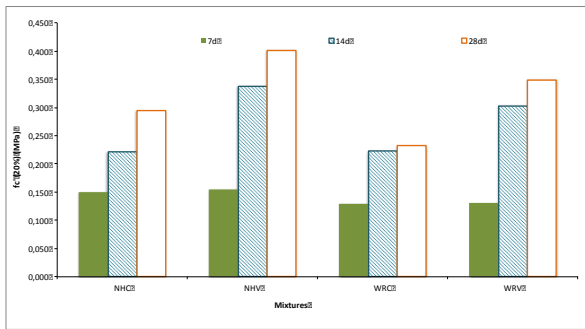


Fig. 5 : Compressive strength at 5% of deformation

At 7 days, the young age, NH compressive strength is higher than WR. At 28 days it is NHV mix which get the highest strength in compression about 0.210 MPa. NHC follows with 0.140 MPa and after WR mixes get the lower strength in compression 0.133 MPa for WRV and 0.130 MPa for WRC. The second graph highlights the fact that at 5% of deformation NHV have the highest strength in compression. WR mixes are the lowest values with NHC, so the WR treatment appears not effective for the strength in compression at 5% of deformation.

Fig. 5 shows also that the strength at fourteen days is not very necessary at 5% of deformation because it is close to 28d results.

• **Development of compressive strength**

If the strength at 28d is considerate as 100% in our case, the graph as follow highlights the percentage at 7d and 14d. At 7d the strength for Vicat mix is around 40% and for Calcic lime it is more about 50%. In the same case at 14d for every mix the strength is between 80% and 90% except for NHC mix (75%). This part concluded on the fact that the compression strength at 14 days is not essential, the most important values stay at 7d and 28d.

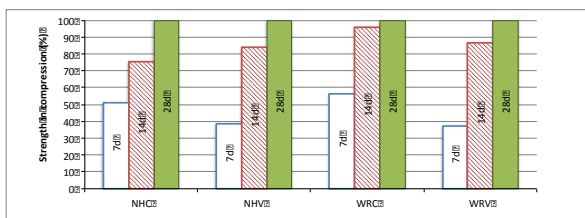


Fig. 6 : Compressive strength evolution with time

**3.2.2. Capillary absorption**

After the water absorption test on particles, capillarity of samples has been measured at three different ages: 14 d, 28 d and 60 d. Like in every test every mixes was tested to see the effect of treatment on this property and compare with a non-treated sample. The process used has been presented previously.

Results have been presented in two ways. Firstly the evolution with time of water absorption in mass percentage has been plotted (Fig. 7). Secondly as the test enables the water to enter only by on face, the water absorption in kilograms per square meter has been calculated. Then it is plotted in function of square time and the trend curves can be traced (Fig. 8). The sorption coefficient has been obtained after 5 hours of immersion thanks to this last graph.

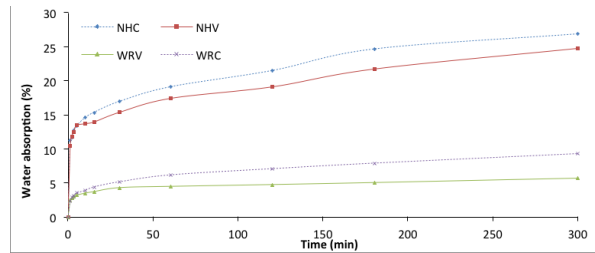


Fig. 7 : Water absorption during capillarity test at 28 days

The water absorption increase directly after the first immersion but this time the value is more about 16.80% for NHV while NHC stay stable with 11.46%. After 28 days results of first immersion are quite the same of 14d, the two treatments have been effective. The WR is with 1.90% for WRC and 1.66% for WRV (Fig. 7).

After 300 minutes, at the end of the test the rate keep increase for NH mixes with 27.28% for NHC and 31.68% for NHV. It stays quite stable for treated ones between 3% and 8%. WRV get the lower absorption rate with 5.99%. On another hand WRC rate value is 7.90%, it stays four times bellow NH one.

Fig. 8 highlights the sorption coefficient. Values have been plot with the water absorption in kilograms per square meter in function of square time. The sorption coefficient has been calculated with the trend curves.

NH mixes sorption coefficient varied between 3.62 for NHV to 3.70 for NHC. It is still almost the double compare to treated samples which varied between 0.54 and 1.75. This time WR mixes are quite close to each other's with 0.74 for WRV and 0.90 for WRC. Therefore WR mixes absorb 4.5 times less water than NH ones.

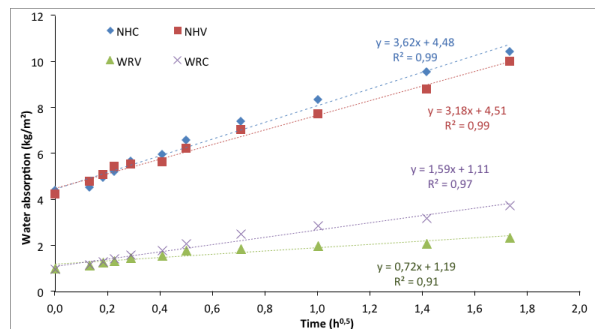


Fig. 8 : Variation of water absorbed per square meter in function of square time.

With the water absorption capacity in percentage plotted in function of samples density the results observed are quite the same as previously in 14d test. This time NHV is the more absorbent mix with 16.8%, the NHC with a density also around 450kg/m<sup>3</sup> is less absorbent with a rate of 11.46%. These results according to the literature are not expected, normally when the density increase the water absorb decrease, because the porosity is supposed to decrease. WR samples density is around 350kg/m<sup>3</sup> and 400 kg/m<sup>3</sup> a little bit less than previously and the water absorption is six time lower than the one of NH samples with 1.90% for WRV and 1.66% for WRC.

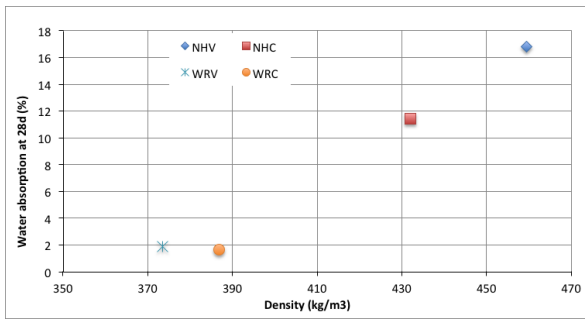


Fig. 9 : Water absorption percentage in function of samples density

**3.2.3. Weathering test**

Weathering tests have been conducted with 48 hours cycles of wetting and drying in the oven but sometimes cycles has lasted 72 hours because of the laboratory availability (on weekend).

**3.2.3.1 Full immersion test**

The full immersion test is the result of 12 to 15 cycles depending on mixes. The following graph (Fig. 10) shows that, NHC is the more absorbent mix all along the test and in the opposite WRV is the less absorbent mix over the test duration. This graph highlights also the fact that for WR and NH mixes mixed lime binder is more absorbent than Vicat cement. WR are 25% less absorbent than NH mixes.

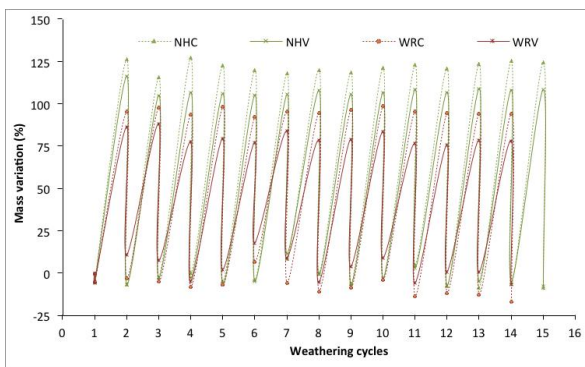


Fig. 10 : Mass evolution during drying/wetting cycles of weathering test with full immersion

The graph in Fig. 11 as follow highlights the fact that the mass variation was quite stable after 4 cycles during the test.

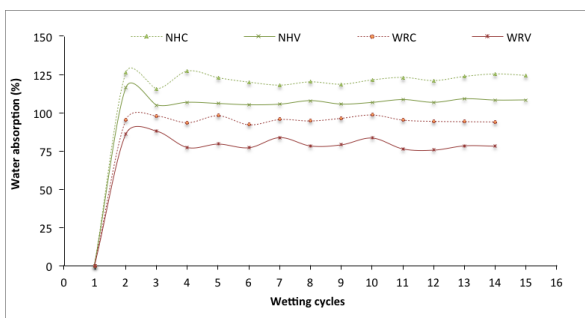


Fig. 11 : Mass evolution during wetting cycles of weathering test with full immersion

Another graph on Fig. 12 enables to compare the percentages of water absorption after 12 cycles of wetting/drying. It highlights the fact that NHC is the

more absorbent mix with 121% following by NHV with 107%, WRC with 95% and WRV with 76%.

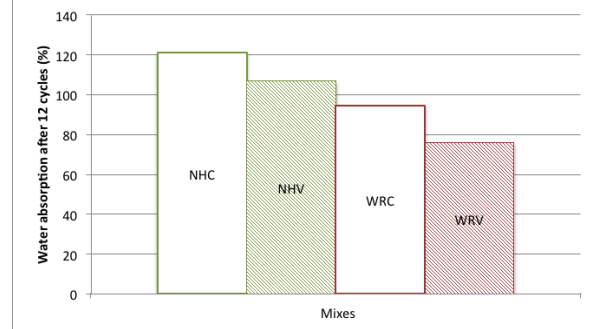


Fig. 12 : Water absorption rate after 12 cycles

It can also be noticed that the rate obtained after 12 cycle for the NHV is quite the same as the one obtained by Castel on is project with the same mix (Mix A') around 110% [Castel 2016].

Even if it can represent the floating over the long term this test does not reflect the reality. The samples are fully immersed compare to a floating when walls are just partially immersed at the bottom.

**3.2.3.2. Compressive strength of weathering and no-weathering samples**

As the two different immersions have not been conducted in the same time, the duration is not the same at the end and the results for the strength in compression cannot be compared. So the following parts will present in the first time the strength in compression for full immersion. On each part weathering samples have been compared to no weathering samples.

- Full immersion test in compression after 14 and 15 cycles

Compressive strength in this part has been conducted after 14 cycles for WR and 15 cycles for NH mixes. The following graphs in Figs. 13 and 14 highlight the difference of compressive strength between NW and FW samples. As expected FW samples get a lower strength than NW ones, the first graph presents compressive strength results and the second represents the difference in percentage between NW and FW samples.

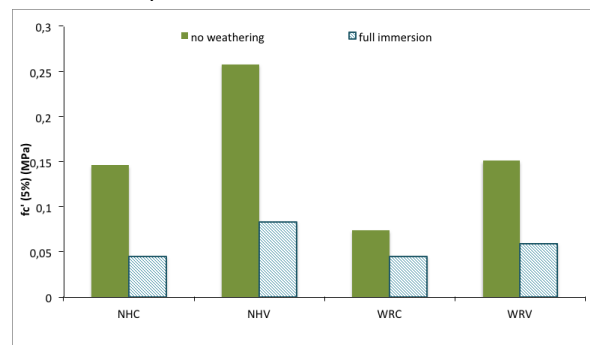


Fig. 13: Compressive strength evolution of NW and FW samples in every condition at 5% of deformation

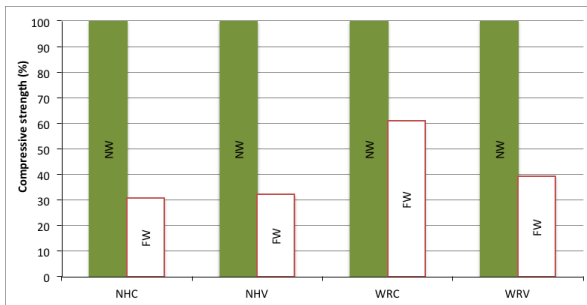


Fig. 14 : Compressive strength percentage comparison between NW and FW samples at 5% of deformation

The higher difference is for the NH mixes with 70%, indeed NHV-NW samples get the highest strength in compression but also the highest difference between the two conditions. In this case it can be considered as the most fragile sample or the most affected by weathering test as it has been seen in the previous part NHV mix is also one of the most absorbent mix.

WR mixes depend on binder: mixed lime is around 35% difference and Vicat cement around 60%. Therefore the treatment might have effect on compressive strength of weathering samples for full immersion.

According to Castel et al results [Castel 2016] the strength in compression for NHV mix (Mix A' in his case) the compressive strength for NW is around 0.22MPa and 0.12MPa for FW. In our case we get a slightly higher compressive strength for NW samples with 0.26MPa and in the opposite we get a smaller value for FW samples with 0.083 MPa. This 20-30% is not negligible, therefore even if we have close results for water absorption rate the comparison can change in function of the test conducted.

### 3.2.4. Carbonation test

- Phenolphthalein spraying

Before spraying samples with phenolphthalein lotion, samples were previously cut in the middle.

Phenolphthalein test has been done after six and nine weeks of carbonation cure, results are presented in the Figs. 15 and 16. The thickness of carbonation stage cannot be measured in this case; just visual observation can be made.

All mixes with Vicat, which have stayed in the carbonation chamber, look totally carbonated. The difference can be observed for mixes, which stay in the air, they are not totally carbonated. This observation highlights the fact that chamber cure has increased the carbonation phenomenon. Another point is the little difference between WRV and NHV mixes, WRV mix is less carbonated than NHV but this difference has to be taken carefully because this test is just visual, a TGA analysis will confirm that.

On the other hand for mixed lime the section is almost entirely pink, in case of NH and WR mixes, therefore mixed lime has not been carbonated at this stage.

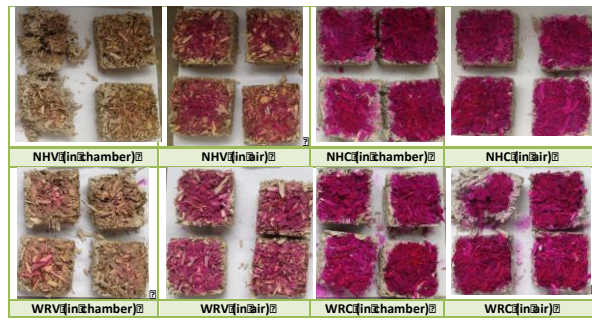


Fig. 15: 50mm cubes samples after phenolphthalein spray at 6 weeks of curing

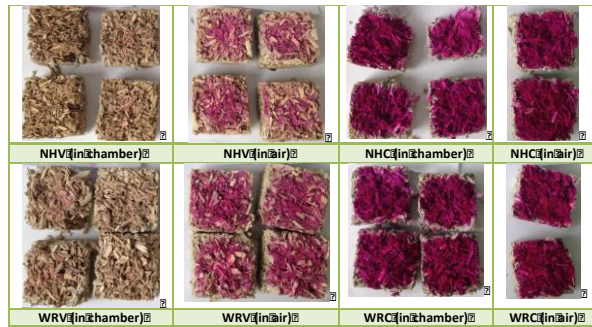


Fig. 16: 50mm cubes samples after phenolphthalein spray at 9 weeks of curing

These tests at two different times highlight the fact that carbonation phenomenon is very slow. Even after nine weeks of cure some samples stay totally uncarbonated. The carbonation chamber enables the binder to carbonate faster but it still slow for NHC and WRC mixes. These two mixes are very porous and very crumble. As the aggregates have absorb a large quantity of water there is not enough water at the end to react with binder. Therefore a powder appears when the samples are cut.

These tests highlight also the fact that 50mm cubes are not the best size to measure carbonation on samples because the depth cannot be measured. Samples are totally carbonated or not carbonated at all there is no middle stage of reaction or at least it is not visible with naked eye.

Therefore another phenolphthalein test at 6 weeks has been done on 100mm cubes pictures after reaction are resumed in Fig. 17.

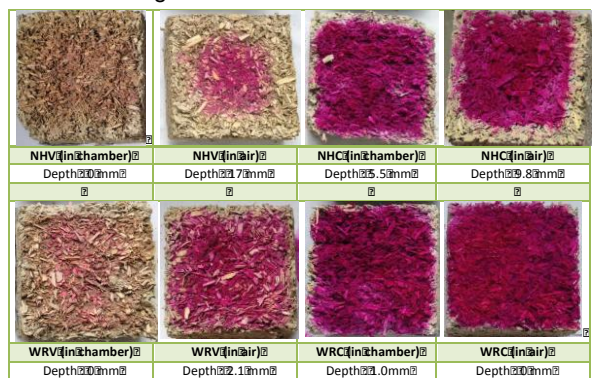


Fig. 17: 100mm cubes samples after phenolphthalein spray at 6 weeks of curing

As expected and noted previously, chamber environment enabled the acceleration of carbonation phenomenon. Otherwise NHC look opposite of this conclusion indeed, the depth of NHC samples which have stayed in the air is higher than the depth of



samples which have stayed in the chamber. This difference has to be verifying with TGA test but it might be because of the samples density. NHC sample which have stayed in the air looks denser than the one who stay in the chamber.

These results confirm observations made previously apart for WRC mix which is still no carbonated, on others samples the difference between air and chamber environment is visible. It can be notice that Vicat mixes are more carbonated than mixed lime mixes.

WR treatments have reduced the phenomenon it can be noticed that for the same binder, NH mixes are more carbonated than WR mixes.

- Compressive test after 9 weeks

Two representations resume the results of compressive test at 5 % of deformation for 9 weeks samples. Apart for WRV and NHC mixes, the carbonation chamber environment has increased the strength of samples as it can be seen on Fig. 18.

The second graph represents the relation between air and chamber samples (Fig. 19). NHV the chamber multiplies per 1.5 the strength in compression, regarding WRC mix, the compressive strength stay stable.

WRC and NHC get the lowest strength in compression but according to the previous part they have also got the lowest carbonation stage. More a sample is carbonated more it is strong. NHV and WRV obtained a slightly higher strength, which is link to the carbonation stage also because these two mixes are a little bit more carbonated than the previous two.

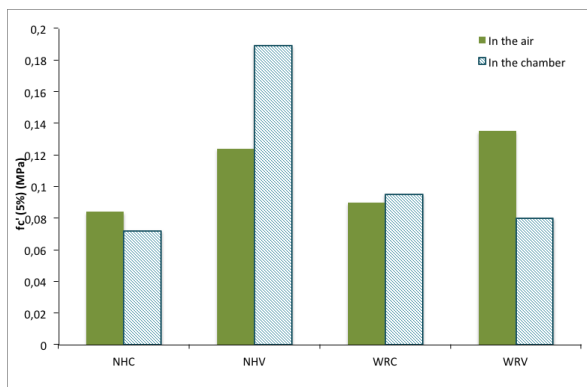


Fig. 18 : Compressive strength comparison between samples stayed in the air and in the chamber after 5% of deformation

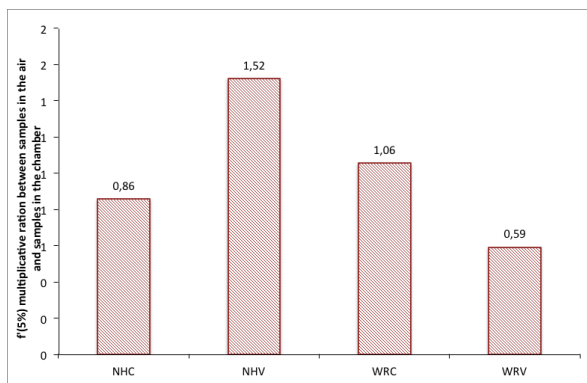


Fig. 19 : Coefficient of the relation between air and chamber samples after compressive test at 9 weeks

## 4 CONCLUSION

Different kinds of test have been carried out during this investigation on four mixes composed of two binders and water repellent (WR) treatment. These experiences have enabled to better understand the effects of aggregates treatment on different properties.

- Firstly the characterisation of hemp shiv particles have highlighted the fact that the density of the WR aggregates remained the same as no-treated hemp shiv particles. The WR treatment resulted in no reduction of the water absorption of particles.

- Subsequently, the lowest compressive strength were obtained with mixes made with WR and it was even worse than no-treated hemp concrete.

- The results of the capillary absorption showed that there is no obvious link between the density of the specimens and the water absorption capacity particularly for samples treated with WR. This is a different to the results reported in literature. In fact, WR samples are the lighter ones and having the lowest values of capillary absorption. In general, the sorption coefficient of hemp concrete has been divided by two with treatments.

- The weathering has been carried out in full immersion in water. Full immersion has shown that treatments significantly decreased the water absorption during cycles. Treatments led to a smaller difference in compressive strength between weathering and no weathering samples.

- Finally the carbonation test was conducted on small (50mm cubes) and large specimens (100mm cubes). Phenolphthalein results after 6 and 9 weeks show that Vicat binder was totally carbonated compared to mixed lime. To remain, the fact that calcic lime was mixed (with fly ash and Portland cement) has definitely increased the phenomenon compared to what was reported in literature.

## 5 REFERENCES

- [Amziane 2016] Amziane, S., Sonebi, M., *Overview on Biobased Building Material made with plant Aggregate*, RILEM Technical Letters, 1, 2016, 31-38.
- [Amziane 2015] Amziane, S., Sonebi, M., *Proceedings of the First International Conference on "Bio-Based Building Materials"*, ICBBM-2-15, Proceedings PRO 99, Ed. Amziane, S., Sonebi, M., 2015, RILEM Publications s.a.r.l., 746p.
- [Amziane 2017] Amziane S., Collet F., *Round Robin Test for hemp shiv characterisation: Part 2, bulk density and particle size distribution*, Springer Edition, Edited by S. Amziane, 2017.
- [Amziane 2003] Amziane, S., Arnaud L., *Bio-aggregate-based Building Materials – Applications to Hemp Concretes*. - Ed. Wiley, 2013, 314p.
- [Castel 2016] Castel, Y., Amziane, S., Sonebi, M *Durabilité du béton de chanvre : résistance aux cycles d'immersion hydrique et séchage - 1<sup>ère</sup> conférence EuroMaghrébine des Biocomposites*, Marrakech, 2016, 7 p.
- [Chabannes 2015] Chabannes M., E. Garcia-Diaz E., Clerc L., Bénézet J.-C., *Studying the hardening and mechanical performances of rice husk and hemp-based building materials cured under natural and*

accelerated carbonation, *Constr. Build. Mater.* 94 (2015) 105–115

[Cizer 2012] Cizer O., *Real-time investigation of reaction rate and mineral phase modifications of lime carbonation* - Construction and Buildings Materials, 2012, 741-751.

[Construire 2012] Construire en Chanvre, Règles professionnelles d'exécution d'ouvrages en bétons et mortiers de chanvre, SEBTP, Paris, France, 2012.

[Falchi 2015] Falchi L. et al. *The influence of water-repellent admixtures on the behaviour and the effectiveness of Portland limestone cement mortars* - Cement & Concrete Composites, 2015, 107-118.

[Kashtanjeva 2015] Kashtanjeva, A., Sonebi, M., Amziane, S., *Investigation of the Mechanical Performance and Drying Shrinkage of Hemp Concrete, Proceedings of the First International Conference on Bio-Based Building Materials, Ed. Amziane, S., Sonebi, M., Clermont-Ferrand (France), June 2015, RILEM Publications s.a.r.l., 309-315.*

[Khazma 2011] Khazma M., et al. *Optimisation of flax shiv-cementitious composites : Impact of different aggregate treatments using linseed oil* - Construction and Buildings Materials (61), 2014, 442-452.

[Izaguirre 2015] Izaguirre et al. *Effect of water-repellent admixtures on the behaviour of aerial lime-based mortars*, Cement and Concrete Research, 2008, 1095-1104.

[Lawrence 2007] Lawrence et al. *Effects of carbonation on the pore structure of non-hydraulic lime mortars*, Cement and Concrete Research, 37, 2007, 1059-1069.

[Magniont 2010] Magniont, C., *Contribution à la formulation et à la caractérisation d'un écomatériau de construction à base d'agroressources* - PhD Thesis, 2010, 343 p.

[Nozahic 2012] Nozahic, V.; Amziane, S.; Torrent, G.; Saïdi, K. et al.; Design of green concrete made of plant-derived aggregates and a pumice–lime binder. *Cement & Concrete Comp.* 34(2) 2015, 231-241.

[Nozahic 2012] Nozahic V., Amziane, S. Influence of sunflower aggregates surface treatments on physical properties and adhesion with a mineral binder, *Composites Part A*, 2012, 43: 1837-1849.

[Nguyen 2010] Nguyen, T.T.; Picandet, V.; Amziane, S.; Baley, C.; Influence of compactness and hemp hurd characteristics on the mechanical properties of lime and hemp concrete. *European Journal of Environmental and Civil Engineering*, November 2009, Volume 13, Issue 9, p. 1039-1050.

[Page 2015] Page, J., Sonebi, M., Amziane, S. *Experimental Investigation on the Performance of Hybrid Hemp-Flax Composite Materials*, Proceedings of the First International Conference on Bio-Based Building Materials, Ed. Amziane, S., Sonebi, M., Clermont-Ferrand (France), 21-24 June 2015, RILEM Publications s.a.r.l., p. 243-250.

[Sonebi 2013] Sonebi M., Robinson, A., Amziane, S., Influence of Presaturation Regime, Percentage of Lime and Compaction on the Performance of Hemp Concrete, Proceedings of First Int. Conference on Rheology and Processing of Construction Materials and 7th Int. RILEM Conf. on SCC, 2013, CD 8p.

[Sonebi 2015] Sonebi, M., Wana, M., Amziane, S. Investigation of the Mechanical Performance and Weathering of Hemp Concrete, Proceedings of the First International Conference on Bio-Based Building Materials, 2015, RILEM Publications s.a.r.l pp. 416-421.

[Walker 2014] Walker R., Pavia S., Mitchell R., Mechanical properties and durability of hemp-lime concretes, *Constr. Build. Mater.* 61 (2014) 340–348.