Impact of water retainers in the strength, drying and setting of lime hemp concrete

Rosanne Walker[1], Sara Pavia[1]
[1]Department of Civil, Structural and Environmental Engineering, Trinity College, Dublin 2, Ireland
email: walkerro@tcd..ie, pavias@tcd.ie

ABSTRACT: Lime hemp concrete is a sustainable, carbon negative building material that can be used in certain applications lowering the environmental impact of construction. Hemp absorbs large quantities of mixing water (325% of its own weight at 24hours), and this may not leave sufficient moisture in the binder for hydration or carbonation to take place adversely affecting curing and strength development. This paper investigates the effect of using water retainers to ensure that sufficient water is available for proper curing. Hemp concrete including a lime: pozzolan (either GGBS or metakaolin) binder and three water retainers (methyl cellulose and two commercial water retainers one of which is methyl cellulose based) were investigated. This paper studies the impact of the water retainers on strength, drying, setting and microstructure. It was found that the three water retainers delayed setting and drying.
The commercial binders did not significantly affect strength however the methyl cellulose improved the compressive strength of both lime:pozzolan pastes and hemp concrete at later ages (100 days). The increase in compressive strength is partially attributed to an enhanced binder water retention that improves hydration. This assumption is based on the increase in the amount of pozzolanic cements, evidenced with SEM at the hemp interface, in the composite with methyl cellulose.

KEY WORDS: lime hemp biocomposite; pozzolan; water retainer; methyl cellulose; setting; drying; compressive strength.

1 INTRODUCTION

Lime hemp biocomposites are sustainable, carbon negative materials that can replace high embodied energy materials in certain applications, lowering the environmental impact of construction. They are non-load bearing materials with good thermal and insulation properties which are usually site-cast, projected or prefabricated as blocks or slabs. They have been used in France since the 1990s and are gaining popularity in Europe, for example in Ireland there are now over 20 buildings constructed using lime hemp biocomposites and a further 100 that have been thermally upgraded [1].

Lime hemp composites contain a lime based binder and hemp shiv which is the woody interior of the hemp stalk. Cement is usually incorporated to produce an early hydraulic set and improve early age properties such as setting and strength. This paper is part of a wider research programme which aims at formulating a binder where cement is replaced by pozzolan, resulting in a biocomposite with a lower environmental impact. Pozzolans are materials with an amorphous siliceous or siliceous and aluminous content that react with portlandite ($Ca(OH)_2$) in the presence of water to form cementitious hydration products (calcium silicate hydrates and calcium silicate aluminate hydrates) thereby accelerating hardening of calcium limes by imparting a hydraulic set.

The purpose of this paper is to investigate the behaviour of water retainers in the lime-pozzolan hempcrete. Water retainers improve the binder ability to hold water, and were developed for use when rapid dehydration occurs either by absorption of a substrate or evaporation due to drying [2]. They are often used when the mortar is mixed with high suction brick. The mortar water retention becomes more important as the suction rate of the brick increases [3 referring to 4]. Similarly, in hemp concrete, the water retention capacity of the binder is very important due to the high suction rate of the hemp.

The hemp aggregate (shiv) is a complex woody tissue from the xylem layer of the hemp stem whose main function is conducting water therefore, as an aggregate, it absorbs large amounts of water. The lime binder counteracts this high suction ability: Lime binders typically possess a high water retention, values ranging from 94.2 to 99.5% have been consistently measured in 3:1 mortars made with hydrated and natural hydraulic limes [5], [6].

In the biocomposite, water is needed for pozzolanic reactions to take place in the lime:pozzolan binder. Water is chemically bound to hydration products calcium silica hydrate (CSH) and calcium silica alumina hydrate (CSAH) and, if water is not present, these hydration products cannot form. Nozahic and Colinart state that following mixing, the high absorption of the hemp shiv induces a competition between the hemp and binder for water [7] [8].

This study uses water retainers in an attempt to ensure that sufficient water remains in the binder to be used for carbonation and hydration. This is particularly important in lime:pozzolan binders, where water is required both initially and at later ages, due to the fact that pozzolanic reaction starts early, but it is slower than cement hydration and continues for long time periods.

Insufficient water in the binder also delays carbonation (as $Ca(OH)_2$ and CO_2 must be in solution to react) however, carbonation typically occurs over months and years and does not significantly contribute to early age properties.

The effect of the water retainers is investigated by studying the microstructure of the biocomposite as well as properties including drying, setting and strength. Setting and drying are important parameters as lime concrete requires a large amount of mixing water that leads to long setting and drying times, which are not acceptable at industrial scale [9]. Hemp concrete is a non load-bearing material therefore, compressive strength is not a critical consideration, however, compressive strength gives an indication of the integrity of the composite including degree of carbonation/hydration and cohesion at the binder/hemp interface.

2 MATERIALS AND METHODS

2.1 Materials

A hydrated commercial lime (CL90s) complying with EN 459-1 was used. Two pozzolans: Metakaolin and GGBS, were identified as having potential for use in the lime hemp biocomposite on account of their fast setting times and high reactivity [10,11]. The pozzolans' chemical composition, rate of amorphousness and surface area are included in Table 1. The chemical composition was assessed by XRF using a Quant'X EDX Spectrometer and UniQuant analysis package. The rate of amorphousness was indicated by X-Ray diffraction (XRD), using a Phillips PW1720 XRD with a PW1050/80 goniometer and a PW3313/20 Cu k-alpha anode tube at 40kV and 20mA. The specific surface area was measured using a Quantachrome Nova 4200e and the BET method, a model isotherm based on adsorption of gas on a surface.

Three water retainers were investigated; modified hydroxypropyl methyl cellulose (MC) and two commercial water retainers referred to in this paper as A (which is methyl cellulose based) and B, whose composition is unknown due to commercial considerations. Industrial hemp shiv was supplied by La Chanvrière De L'aube in central France.

Table 1. Chemical and mineral composition, rate of amorphouseness and surface area of pozzolans.

Pozzolan	GGBS	Metakaolin
SiO_2	34.14	51.37
Al_2O_3	13.85	45.26
CaO	39.27	-
Fe_2O_3	0.41	0.52
SO_3	2.43	-
MgO	8.63	0.55
Mineralogical composition	no crystalline fraction	quartz, tohdite, aluminium oxide, wollastonite and paragonite
Rate of amorphousness	Totally	Mostly
Surface area m^2/g	2.65	18.3

2.2 Preparation of samples

Due to the nature of the material, both pastes and composites needed to be investigated. For example, it is not possible to determine the setting time of the biocomposite using the Vicat test as the organic aggregate impedes needle penetration and absorbs water; hastening the drying of the paste and giving inaccurate results. Therefore, setting time was measured in pastes in which the hemp shiv was replaced by hemp water. This hemp water was prepared by soaking the hemp shiv for 45 minutes so that it releases its water soluble constituents including pectins.

2.3 Preparation of pastes

The composition of the pastes is set out in Table 2. Pastes made with water were included as control samples. The water content was fixed for all pastes to equally compare the effect of the water retainer.

Table 2. Paste composition

Sample	Pozzolan	Lime	WR	Water
MW	40g M	160g	-	W 172g
MH	40g M	160g	-	HW 172g
MH + MC	40g M	160g	4g MC	HW 172g
MH + WR (A)	40g M	160g	4g WR (A)	HW 172g
MH + WR (B)	40g M	160g	4g WR (B)	HW 172g
GW	60g G	140g	-	W 172g
GH	60g G	140g	-	HW 172g
GH + MC	60g G	140g	4g MC	HW 172g
GH + WR (A)	60g G	140g	4g WR (A)	HW 172g
GH + WR (B)	60g G	140g	4g WR (B)	HW 172g

M- metakaolin; G- GGBS; W- water; H- hemp water; MC-methyl cellulose; A,B- water retainers A and B.

Mixing was in accordance with EN 459-2 [12] except for the addition of the pozzolan (added after 1 minute). The samples were demoulded after 1 day and stored in a curing room at 20°C±3°C and relative humidity 65%±10%.

2.4 Preparation of hemp concrete

Biocomposites were made with 20% metakaolin/80% lime and 30%GGBS/70% lime; 2% water retainer (by weight of binder) was added. These pozzolan contents were considered the most suitable on the basis of reactivity, setting behaviour in the presence of hemp and environmental impact [10,11]. The binder:hemp:water ratio was fixed at 2:1:3.1 for all samples.

The binder and three quarters of the water were mixed into a paste for 1 minute and the hemp and remaining water then added. The samples were mixed in total for 5.5 minutes. After mixing, the samples were transferred into 100mm cube moulds in a single layer and lightly tamped. The samples were transferred to a curing room at temperature 20°C±3°C and relative humidity 65%±10%. The samples had a dry density of c.430kg/m^3.

2.5 Water absorption of the hemp aggregate

Hemp particles were suspended in a porous bag in a beaker of water. Overtime, the hemp absorbed water reducing the water content in the beaker. The quantity of water absorbed by the hemp was determined by weighing the beaker at regular intervals. A control sample monitored weight loss due to evaporation and absorption of the bag.

2.6 Setting time of paste

The effect of the water retainers on setting time was determined by comparing the Vicat test [EN 459-2] results of lime:pozzolan pastes with and without the water retainers.

The Vicat test determines the rate of stiffening of the paste/mortar by dropping a needle from a fixed height and measuring its penetration. Stiffness is related to drying and flocculation, the formation of hydrates and the rate of carbonation. The initial and final setting times (at 35mm and 0.5mm respectively) were recorded as standard references to provide comparative data between samples. During setting, the effect of the water retainer on drying was measured by weighing the pastes at regular intervals.

2.7 Drying

The rate of drying of the hemp concrete was monitored by weighing the biocomposite at regular intervals over 100 days. Overtime there was a net weight loss due to water loss. However, carbonation causes a small weight gain as the calcium carbonate is heavier than portlandite. This has been disregarded as carbonation weight gain is very low at early ages and should be similar in all samples.

2.8 Compressive strength of pastes and biocomposite

The unconfined compressive test of half 40*40*160 prisms was measured with a Zwick loading machine according to EN 459-2 with a loading rate of 400N/s. No standards currently apply to lime-hemp concrete and EN 459-2 was used. Typically, the composite does not break but continuously deforms, therefore, the ultimate strength was set as the stress at which the stress/strain curve departs from a linear relationship (Figure 3).

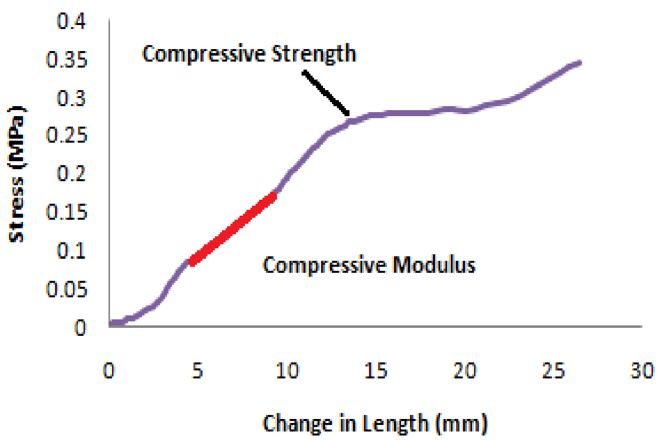

Figure 3. Representative stress/strain curve of a specimen of hemp concrete illustrating the ultimate compressive strength and compressive modulus.

2.9 Compressive modulus

The deformation of the biocomposite when load is applied is plastic [13]. The compressive modulus measures the stiffness of the material and is calculated as the slope of the linear part of the stress vs strain curve (between 5mm and 10mm deformation) (Figure 3). It was calculated by dividing the stress (force (F) / area (a)) by the strain (the distance travelled by the loading point (ΔL) and the original height of the sample (h)) according to equation 1 below:

$$E = \frac{F / a}{\Delta L / h} \qquad (1)$$

2.10 Microstructure of the hemp concrete

The effect of the water retainer on the microstructure of the binder, and the formation of pozzolanic reaction products were investigated using a Tescan MIRA Field Emission Scanning Electron Microscope. The samples were freshly fractured and covered with a gold coating in an 'Emscope SC500' plasma coating unit. The effect of the water retainer methyl cellulose was investigated in the bulk mortar and at the hemp interface at 100days. Only the methyl cellulose was considered as the two commercial water retainers did not appear to impart a beneficial effect to the biocomposite.

3 RESULTS AND DISCUSSION

3.1 Water absorption of hemp

Hemp shiv absorbs large amounts of water due to its highly porous structure [7]. Mixing water is primarily absorbed by capillary action through the tracheids of the shiv which are typically oriented along the long side of the hemp particle (Figure 4).

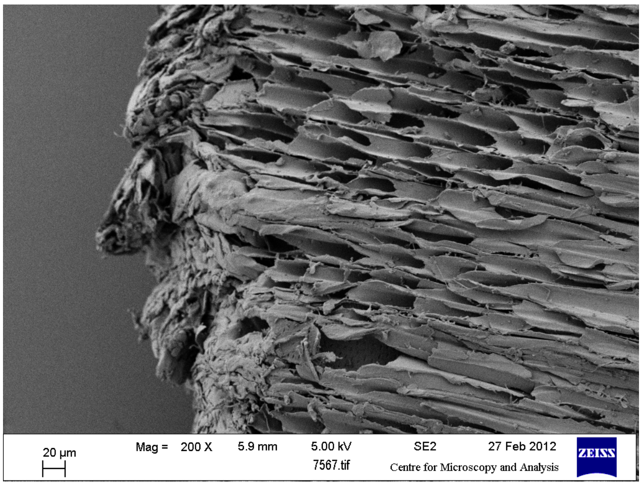

Figure 4. SEM image of the end of a hemp aggregate showing the open tracheids that absorb the mixing water.

The hemp was found to absorb approx 225% and 325% of its own weight, at 5 minutes and 24 hours respectively, as shown in figure 5. This is lower than the absorption measured by Nozahic [7] who observed a 300% percent increase at 5 minutes. The typical hemp:water ratio in hemp concrete is approximately 1:3 (by weight- in this research is 1:3.1). As the hemp absorbs 325% of its own weight at 24hours, at these proportions, the hemp has the potential to absorb all the mixing water. Therefore, the water retention capacity of the binder is of great importance. Brick masonry also has the potential to absorb large amounts of water from the mortar. Brocken [14] states that the water extraction rate from the mortar by brick is primarily determined by the sorption of the brick, but the amount of water that remains in the mortar after reaching equilibrium strongly depends on the mortar type (its water retention capacity).

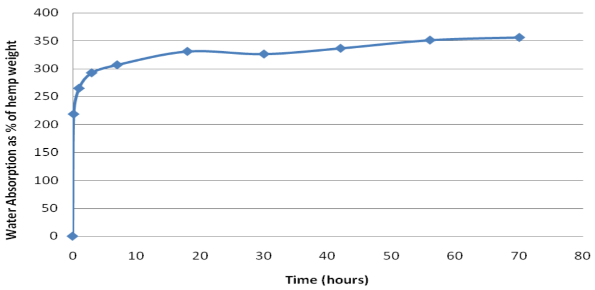

Figure 5. Water absorption of hemp aggregate.

3.2 *Setting time*

It has been demonstrated that the soluble constituents of hemp delay setting of lime:pozzolan pastes, and that the delay is greater in the lime:GGBS than in the lime:metakaolin pastes [15]. This is evidenced in the results (Figures 6 and 7), where the pastes made with hemp water (GH, MH) are delayed when compared to those made with water (GW, MW). In addition, it is well established that water retainers delay setting time [2]. This is also observed in the results in Figures 6 and 7, where all three water retainers delay setting. Commercial water retainers A and B cause a similar delay while MC delays setting the furthest.

Setting is due to drying, flocculation and the formation of hydration products (in this particular hemp concrete as a result of pozzolanic reaction). The water retainers certainly slow drying: this is demonstrated by the reduced weight loss of the pastes with water retainers in table 3.

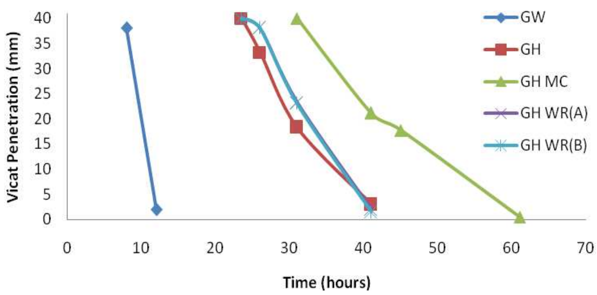

Figure 6. Effect of water retainers on the setting of lime: pozzolan (GGBS) pastes. GH WR(A) is concealed behind GHWR(B).
G-GGBS; W-water; H-hemp water; MC-methylcellulose; WR(A),WR(B)-commercial water retainers A and B.

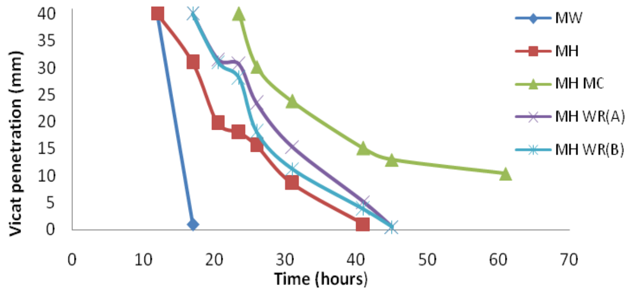

Figure 7. Effect of water retainers on the setting of lime: pozzolan (metakaolin) pastes.

Table 3. Weight loss of samples (between 0 and 60 hours) due to drying during setting

Sample	Weight Loss	Sample	Weight Loss
MW	2.8	GW	2.4
MH	2.9	GH	2.4
MH + MC	2.5	GH + MC	2.1
MH + (A)	2.7	GH +WR (A)	2.3
MH +(B)	2.4	GH +WR (B)	2.3

*M- metakaolin; W- water; H- hemp water; MC-methyl cellulose; (A) (B)-commercial water retainers A and B.

3.3 *Drying of the hemp concrete*

Drying refers to the removal of the free water so that the composite is in equilibrium with the ambient humidity. The open connected porosity of the biocomposite allows the internal transfer of fluid and facilitates the release of water during drying [13]. In the initial drying phase, liquid water will move by capillary forces to the surface and, later, due to sorption of water vapour [16]. In hemp concrete, the rate of drying drops as the moisture content of the sample decreases. According to the results, the GGBS hemp concrete dries faster than the metakaolin concrete, and the water retainers slow drying for both. The methylcellulose and commercial water retainer B delay drying to a greater extent than A (Figure 8 and 9). The drying delay caused by the water retainer has a negative impact on the concrete as long drying times are a major drawback in construction.

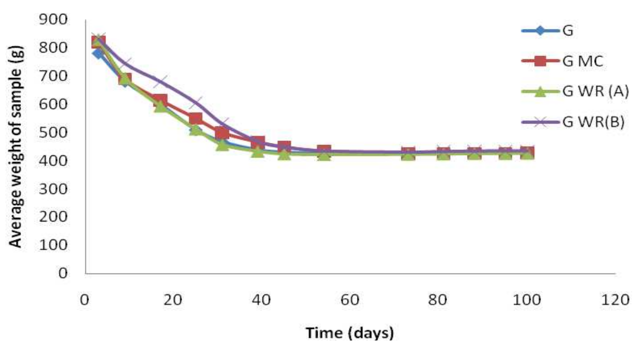

Figure 8. Effect of water retainers on the drying of the lime:pozzolan (GGBS) hemp concrete

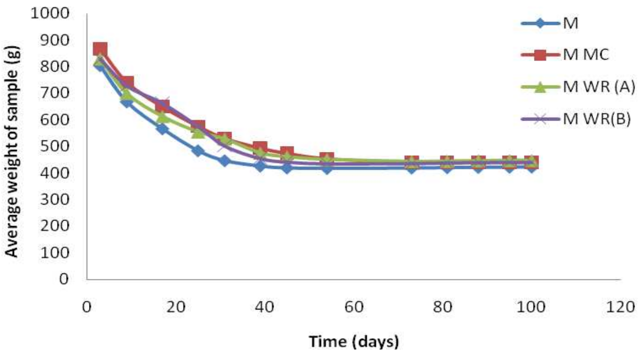

Figure 9. Effect of water retainers on the drying of the lime:pozzolan (metakaolin) hemp concrete

3.4 *Compressive strength of pastes and hemp concrete*

The trend in the compressive strength results is similar in both the pastes and the composites: methyl cellulose significantly increases compressive strength at 100 days (MMC and GMC in Figures 10 and 11), while the commercial water retainers A and B tend to slightly reduce it, but do not have a consistent significant effect (90% confidence) on compressive strength (MWR(A) (B) and GWR(A) (B) in Figures 10 and 11).

The methyl cellulose increases the strength of both the paste and the hemp concrete, this suggests that the compressive strength enhancement of the concrete is not solely due to the water retainer improving the water retention capacity of the binder in the presence of hemp.

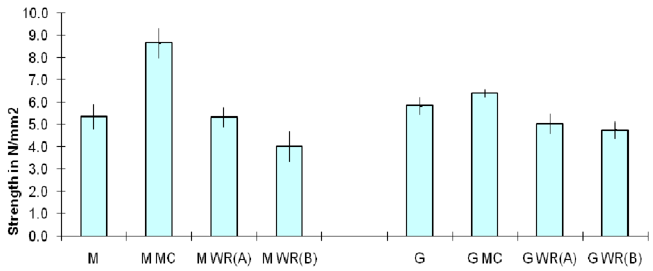

Figure 10. Compressive strength of lime:pozzolan pastes at 100days. (The error bars are ± standard deviation)

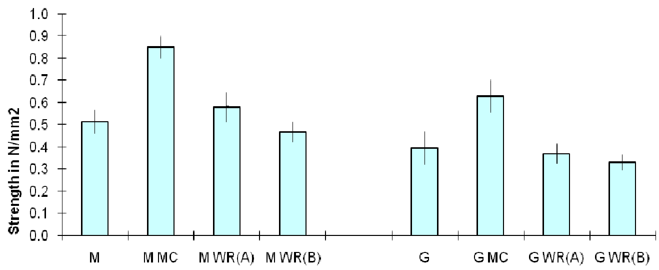

Figure 11. Compressive strength of hemp concrete at 100days.

Previous authors have reported contradictory results on the effect of different cellulose based water retainers and dosages on the compressive strength of cement mortars. The results above agree with Mischa [17] who observed increased compressive strength in portland cement mortar at 91 days for carboxymethyl-cellulose water retainer contents up to 1%. In contrast, Paiva [2] observed a reduction of the 28 day strength in a cellulose methyl–hydroxypropyl cement based render; and, similarly, Fu and Chung [18] also found a decrease in compressive strength with increasing methylcellulose content blaming this effect on the disruption of the continuity of cement phases by the presence of the methylcellulose.

3.5 *Compressive Modulus*

As it can be seen from Figure 12, the samples made with methyl cellulose are stiffer than those without water retainer or with commercial retainers A and B. This agrees with the strength results evidencing that methyl cellulose enhances compressive strength at 100days. The results also agree with Fu and Chung [18], who, investigating PC mortars, found an increase in the compressive modulus with increasing methylcellulose content.

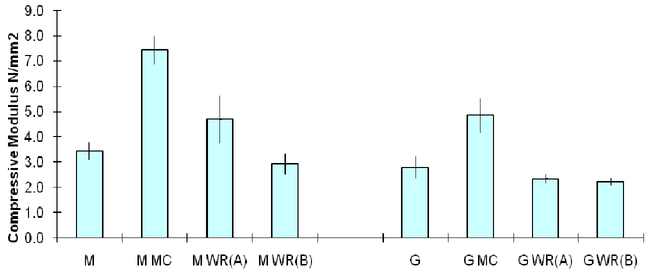

Figure 12. Compressive modulus of hemp concrete at 100 days.

3.6 *Microstructure of hemp concrete*

No significant difference was found in the paste microstructure in specimens made with and without methyl cellulose. However, differences were noted at the hemp interface. This agrees with Arizzi [19] who observed that a cellulose water retainer did not produce any mineralogical and morphological change in mortar pastes.

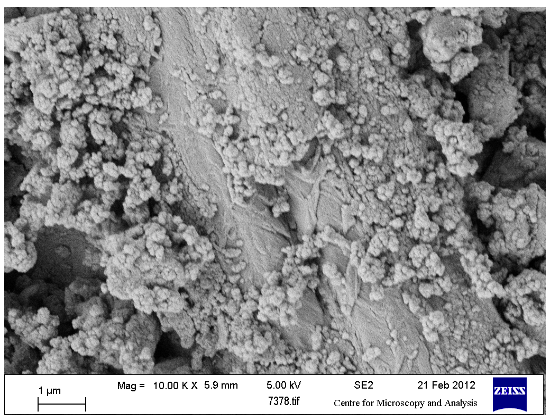

Figure 12. Hemp particle covered with calcium carbonate in a lime:pozzolan (GGBS) hemp concrete at 100 days.

As expected, the surface of the hemp particles was largely covered with small scalenohedral calcite crystals, typically smaller than 1 μm (Figure 12) [15]. These showed cracked/corroded surfaces similar to those observed by Cizer et al. [20]. Differences were noted at the hemp interface: While hydration products did not appear at the interface in the concrete without methyl cellulose (Figure 12), a few clusters of needle-shaped hydration products were evident in those containing methyl cellulose (Figures 13). This may be due to the methyl cellulose retaining water in the binder facilitating the pozzolanic reaction.

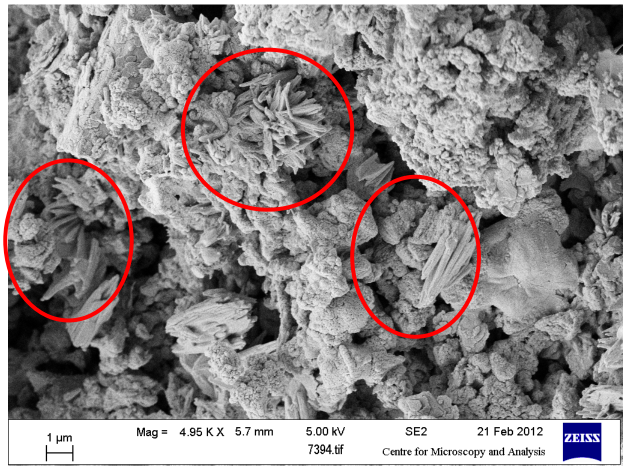

Figure 13. Hemp particle covered with calcium carbonate and clusters of hydraulic needle-like phases, in a lime:pozzolan (GGBS) hemp concrete, with methyl cellulose (at 100days).

4 CONCLUSION

All water retainers delay setting and drying of hemp concrete, however, the methyl cellulose significantly improves compressive strength at 100 days.

The methyl cellulose delays setting the furthest and (together with commercial water retainer A) also delays drying the furthest, however, it significantly improves compressive strength at 100 days. The cause of this increase has not been fully identified, however, it can be attributed to the improvement of water retention by the methyl cellulose leading to enhanced hydration (more hydrates are present at the hemp interface in the concrete including methyl cellulose).

The results also evidenced that the GGBS hemp concrete dries faster than the metakaolin concrete. In addition, it was evidenced that, at the typical hemp:water ratio of hemp concrete, the hemp aggregate has the potential to fully absorb the mixing water compromising hydration. Therefore, it is of vital importance to use a binder with high water retention.

The different behaviour of the commercial water retainers when compared to the methyl cellulose may be partly due to their lower dosage. Water retainer behaviour is strongly dosage dependent and the commercial products were diluted (in liquid form) and consequently their concentration was lower.

ACKNOWLEDGEMENTS

The authors wish to thank the Environmental Protection Agency for funding this research. The authors also thank the Traditional Lime Company, Clogrennane Lime and Ecocem for providing materials and also Dr Heath Bagshaw, Mr. Neal Leddy and Mr. Colin Reid (Centre for Microscopy and Analysis) and Dr. Robbie Goodhue (Geology Department) for their help with the SEM and XRD respectively.

REFERENCES

[1] Pronchetti, R. (2010), *Lime Hemp Workshop*, Mullingar, Co. Westmeath, Ireland.

[2] Paiva, H., Esteves, L. P., Cachim, P. B. and Ferreira, V. M. (2009), 'Rheology and hardened properties of single-coat render mortars with different types of water retaining agents', *Construction and Building Materials*, 23, 1141-1146.

[3] Tate, M. (2005), 'The most important property of cement- lime motar in masonry construction is', *Proceeding of International Building Lime Symposium,* Florida.

[4] Palmer, L. A. and Parsons, D. A. (1934), 'A study of the properties of mortars and bricks and their relation to bond', *National Bureau of Standards Journal of Research.*

[5] Pavía, S. and Hanley, R. (2010), 'Flexural bond strength of natural hydraulic lime mortar and clay brick', *Materials and Structures*, 43, 913-922.

[6] Costigan, A. (2012), 'Improvement of properties of lime mortar masonry', PhD Thesis, Trinity College, Ireland.

[7] Nozahic, V., Amziane, S., Torrent, G., Saïdi, K. and De Baynast, H. (2012), 'Design of green concrete made of plant-derived aggregates and a pumice–lime binder', *Cement and Concrete Biocomposites*, 34, 231-241

[8] Colinart, T., Glouannec, P. and Chauvelon, P. (2012), 'Influence of the setting process and the formulation on the drying of hemp concrete', *Construction and Building Materials,* 30, 372-380

[9] Elfordy, S., Lucas, F., Tancret, F., Scudeller, Y. and Goudet, L. (2008), 'Mechanical and thermal properties of lime and hemp concrete ("hempcrete") manufactured by a projection process', *Construction and Building Materials*, 22, 2116-2123.

[10] Walker, R., Pavía, S. (2010), 'Behaviour and Properties of Lime-Pozzolan Pastes'. Proceeding of 8th International Masonry Conference, Dresden, ed. by W. Jäger, B. Haseltine, A. Fried, 353-362.

[11] Walker R, Pavía S. (2011) 'Physical properties and reactivity of pozzolans, and their influence on the properties of lime–pozzolan pastes', *Journal of Materials and Structures,* 44, 1139–1150.

[12] EN 459-2 Building lime, part 2: Test Methods. (2010), *European Committee for Standardisation CEN*, Brussels.

[13] Evrard, A. (2003), 'Hemp Concrete- A synthesis of physical properties', *Construire en Chanvre.*

[14] Brocken, HJP. and Larbi JA., (1999), 'Composition of mortar as a function of distance to the brick-mortar interface: a study on the formation of cured mortar structure in masonry using NMR, PFM and XRD', *HERON*, 44, 257-270

[15] Walker, R., and Pavía, S. (2012), 'Influence of hemp's soluble components on pozzolanic reactions and on the properties of the hemp biocomposite' –in press.

[16] Holcroft, N. and Rhydwen, R., (2011), 'Effects of evaporative cooling of drying hemp-lime walls on heating load', *Proceeding of Science Symposium*, Centre for Alternative Technology, Wales.

[17] Mishra, P. C., Singh, V. K., Narang, K. K. and Singh, N. K. (2003), 'Effect of carboxymethyl-cellulose on the properties of cement', *Materials Science and Engineering*, 357, 13-19.

[18] Fu, X., and Chung, D.D.L. (1996), 'Effect of methylcellulose admixture on the mechanical properties of cement' *Cement and Concrete Research*, 26, 4, 535-538.

[19] Arizzi, A. and Cultrone, G. (2012), 'Aerial lime-based mortars blended with a pozzolanic additive and different admixtures: A mineralogical, textural and physical-mechanical study', *Construction and Building Materials*, 31, 135-143.

[20] Cizer, O., Van Balen, K., Elsen, J. and Van Gemert, D. (2008), 'Crystal Morphology of precipitated calcite crystals from accelerated carbonation of lime binders', *Proceedings of 2nd International Conference on Accelerated Carbonation for Environmental and Materials Engineering*, Italy, 149-158.