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Hot-lime-mixed hemp concretes.

Zifeng Wang¹ and Sara Pavia²

^{1, 2} Dept. of Civil Engineering. Trinity College Dublin. IRELAND.

Abstract. Hemp-lime composites are sustainable, low-carbon, non-loadbearing materials with outstanding thermal properties and high vapour permeability, used in new construction or thermal upgrades. The abundant pores in the hemp shiv ($\geq 70\%$) are blamed for the low mechanical strength, and for a high water absorption that can result in long drying times and low early strength that delay building.

Hot-lime mixing involves using quicklime as a binder, so that the exothermic reaction of lime slaking takes place simultaneously to mixing. This paper investigates whether hot-lime mixing can improve the properties of hemp-lime concretes. It was hoped that the heat generated on lime slaking would reduce the water intake by the shiv, block shiv pores and promoting adhesion between the shiv and the lime matrix.

The properties of the hot-lime mixed concretes agree with former authors, with densities of 430-470 kg/m³ and strengths of 0.32 MPa (compressive) and c.0.27 MPa (flexural) at 3 months. Their vapour diffusion resistance factor of 4.62, water sorption coefficient c.4.58 kg/m²h^{1/2}, thermal conductivity 0.015-0.021W/mK and specific heat capacity of c.1135 J/kgK are also typical of hemp concretes. The improvement in properties due to hot-lime mixing is very small.

The optimum proportions and method for hot-lime mixing are investigated. The mixing process needs to avoid water competition between the shiv and the lime slaking. Mixing the hemp with quicklime prior to slaking undermines the process lowering the slaking temperature. The best mixing method involved layering the hemp over the quicklime.

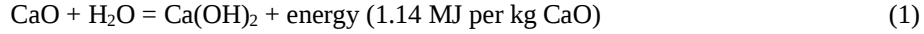
Keywords: Hot-Lime Mixing, Hemp-Lime Materials, Quicklime; Thermal Conductivity, Specific Heat Capacity, Water Vapour Permeability, Water Absorption Coefficient, Strength.

1 Introduction

In today's environment, it is important to use low-carbon, sustainable materials for construction. Hemp-lime materials are a mix of a lime-based binder and hemp. As they are made with a renewable plant (hemp), they are a carbon sink, hence hemp materials are highly sustainable and have great environmental credentials. Hemp-lime materials have been investigated by many authors in the last two decades. Previous authors have demonstrated that they are materials with outstanding thermal properties and high vapour permeability. As explained by many authors, they display ductile failure and low ultimate strengths under uniaxial compressive loads. Typical compressive strengths for 2:1 (binder: hemp by weight) range between 0.05-1.2 MPa, mainly depending on density and age, and the flexural strength is also low, ranging between 0.06 and 1.2MPa (Amziane et al 2017; Evrard 2003; Cerezo 2005; Elfordy et al. 2008; Arnaud and Gourlay 2012; Walker et al. 2014).

The abundant pores in the hemp shiv (over 70%) are blamed for the low mechanical strength (Nguyen 2010), and are responsible for the high porosity, water vapour permeability and water absorption of hemp concretes, which can result in long drying times and low early strength that delay building.

In hot-lime mixing, the exothermic reaction of lime slaking takes place either while mixing or quickly after. It was hoped that the heat released on slaking would help block hemp-shiv pores, speeding up drying and increasing strength. The slaking reaction is exothermic, and generates 1.14 MJ per kg CaO (JRC 2013). Equation 1 describes this reaction:



In hot-lime-mixed mortars, it is argued, that both the heat generated and the expansion of the quicklime on slaking produce early stiffening and fills voids which enhance the interface bond and improve microstructure (BLFI 2014 and 2016; Hunnisett 2016; Wiggins 2018; Artis 2018; Henry 2018; Copsey 2019). However, this is based on site observations has not yet been quantified. Furthermore, the composition of the quicklime and the quantity of water used for slaking control the resultant temperature, which in turn determines the surface area and particle size of the end hydrate, and this rules the properties of the resultant material (Pavia et al.2023). The speed with which the temperature rises from 20 to 60°C (on slaking under standardised conditions) indicates how reactive the lime is. The quicklime's reactivity depends on the burning temperature and time, the crystalline structure of the limestone, the impurities of the limestone and the kiln type and fuel. Soft burnt limes have high reactivity whereas hard burnt limes typically show low reactivity (JRC 2013).

This paper investigates whether hot-lime mixing with a pure quicklime of high reactivity (CL90Q) can reduce the water intake by the shiv and improve the properties of hemp concretes, promoting adhesion between the shiv and the lime matrix. It was hope that the exothermic reaction of lime slaking could seal the shiv pores and improve strength.

2 Materials and Methods

2.1 Materials

A quicklime of European designation CL90Q was used for hot-mixing. The quicklime was crushed and sieved into <1 mm powder. Some of the quicklime particles were unburnt, hence the limestone could hardly be separated from the quicklime. Therefore, the quicklime contains some unburnt limestone which lowers reactivity. Industrial hemp shiv was supplied by La Chanvrière De L'Aube in central France. The water content of the hemp was ~ 12% (at RH ~60%) prior to mixing. The hemp shiv particles vary between 5 and 30mm in length and 0.3-2mm width.



Fig. 1. Quicklime prior to crushing and sieving (left) and hemp shiv.

2.3 Mechanical Properties

The compressive strength was measured on 100×100×100mm cubes according to EN 459-2. The loading rate was 20N/s. The ultimate compressive strength was determined at the point where the behaviour departs from linear stress/strain. The flexural strength was measured with a three-point loading system, on 40×40×160mm prisms, at

10N/s loading rate, according with EN196-1. The results are the arithmetic mean of 6 tests. The flexural strength was calculated using equation 2.

$$Rf = 1.5 \times Ff \times l / b^3 \quad (2)$$

Where, Rf = Flexural strength (MPa)

Ff = Load applied to the middle of the prism (N)

b = Side of the squared cross-section of the prism (mm)

l = distance between the supports (mm)

2.4 Water vapour permeability

The water vapour permeability was measured with the method in EN 12806 using 4× Ø135mm discs, 22mm deep. The discs were sealed on top of containers with 75g calcium chloride inside as a desiccant. The containers were placed in a curing room at $18.5 \pm 3^\circ\text{C}$ temperature and $50 \pm 10\%$ RH. Weight gain was monitored over five weeks. The water vapour permeability was calculated using equation 3.

$$\delta = G \times d / A \times \Delta p \quad (3)$$

Where, δ = Water vapour permeability

G = Weight gain (kg)

d = Thickness of the specimen (m)

A = Area of the specimen (m^2)

Δp = water vapour pressure difference

The water vapour diffusion resistance factor was calculated with equation 4.

$$\mu = \delta \times \delta_{\text{air}} \quad (4)$$

Where, μ = Water vapour diffusion resistance factor

δ_{air} = Water vapour permeability of air

δ = Water vapour permeability of the material

2.5 Water absorption coefficient by capillary action

The capillary suction was measured on 100×100×100mm cubes (EN 1925). The cubes were placed on a wire grill and the weight gain measured once a day over five days. The water sorption coefficient was then calculated as:

$$A_w = m / a \sqrt{t} \quad (5)$$

Where, A_w = Water sorption coefficient ($\text{kg}/\text{m}^2 \text{ hour}^{1/2}$)

m = Weight gain (kg)

a = Surface area (m^2)

t = Time (hour)

2.6 Thermal Conductivity

The thermal conductivity was measured with the hot-box method. The hot-lime mixes (25mm thick) were applied on a hemp wall (750mm high, 930mm wide and 220mm thick). The back of the hemp wall was close to the heating panel and the side of the wall was insulated with 50mm thick PIR board, so that the external surface was covered with the hot-lime mixes. The heat flow through the wall was measured with a Hukseflux TRSYS01 system with two heat flux sensors attached to the external surface. Two thermocouples were also attached to measure the external surface temperature. The heat flux and surface temperature data were logged into TRSYS01 system every 10 minutes. The temperature of the heating panel was set as 60°C and the test was running for 48 hours until the

4

temperature in the front surface was stabilized. The thermal transmittance (U-value) was calculated with eq. 6. The test was then repeated with the hemp wall alone, and the U-value of the hot-lime mixes calculated.

$$U = \frac{1}{\frac{T_{si} - T_{se}}{Q + r_{int} + r_{ext}}} \quad (6)$$

Where, U = U-value (W/m²K)

T_{si} = Internal surface temperature (K)

T_{se} = External surface temperature (K)

Q = Heat flux (W/m²)

r_{int} = Internal surface resistance (standard 0.13 m²K/W)

r_{ext} = External surface resistance (standard 0.04 m²K/W)



Fig. 2. Hot-box assemblage for thermal conductivity measurement.

2.7 Specific heat capacity

Discs of 135 mm diameter (22 mm thick) were heated in an oven at 100°C. Once taken from the oven, their temperatures were measured, and they were then directly transferred into insulated containers with water at 15°C. The rise of the water temperature was monitored every 15 minutes over 1 hour. The results are the arithmetic mean of 4 tests. The property was calculated with equation 7.

$$\Delta T_c \times m_c \times C_c = \Delta T_w \times m_w \times C_w \quad (7)$$

Where, C_c = Specific heat capacity (J / kgK)

ΔT_c = Temperature of the heated sample minus water temperature after immersion (°C)

m_c = Mass of sample (kg)

ΔT_w = Water temperature before immersion minus temperature after immersion (°C)

m_w = Mass of water in container (kg)

C_w = Specific heat capacity of water = 4187 J / kgK

2.8 Microstructure

The microstructure and the cementing phases was investigated at 52-56 days with a Zeiss Ultra Scanning Electron Microscope -SEM- with an EDX (Energy Dispersive X-Ray analysis) attachment. SEM tests were run on both the QL mix at 56 days and the QL+GGBS mixes at age of 52 days.

3 Results

3.1 Reactivity of the quicklime used for hot-lime mixing

The reactivity of the quicklime was studied with a slaking curve (EN459-2) where the measured temperature values were recorded as a function of time. According to the results, the lime has a high reactivity: $t_{60} = 30$ s to 1min; $T'_{max} = 80-98^{\circ}\text{C}$ (100 g) and 130°C (1 Kg). It took between 30 seconds and 1 minute for the quicklime to reach 60°C depending on the size of the sample (100 g vs 1 Kg). A large amount quicklime (1.5 Kg) generated high heat and the reaction was violent. The highest temperature of the hot-mixing reaction was 130°C . The slaking reaction of the lime is assumed to be 100% complete at the time when the maximum temperature (T'_{max}) is reached which is between 5 and 7 minutes for this lime. In general, the reaction begun after 15-20 seconds, and between 20s and 1 min, the temperature raised rapidly to reach $50-60^{\circ}\text{C}$. Temperature over 60°C continued for 5-10 min with 100 g quicklime. Between 1 and 5 min a violent reaction takes place releasing steam. From 5-10 min the reaction is less violent. In all the tests, the temperature lowered after 10-15 min hence the reaction was finished.

Table 1. Reactivity of the quicklime.

Mass of quicklime	t_{60} - seconds	T'_{max} - $^{\circ}\text{C}$	t_{max} - min
100 g	30	80 - 98	5
1000 g	60	130	7

3.2 Mixing

The water content is especially important in hot-lime mixing because it strongly impacts the properties of the resultant material. The amount of water determines the slaking temperature which makes the resultant $\text{Ca}(\text{OH})_2$ crystals vary from fairly large to extremely small, and this produces materials with different properties (Pavia et al. 2023). Therefore, the slaking temperature during hot-mixing was measured with thermocouples and a thermal image camera, and the optimum proportions were investigated by trial, based on cohesion and workability. Twelve mixes were tested with varying proportions. The proportions 1.43: 4.57: 1 (quicklime: hemp: water - specimen 12 in table 2) displayed the best cohesion, workability and early strength at the lowest water content. Specimens were fabricated with this mix and their mechanical, hygric and thermal properties and microstructure investigated. This mix was blended with GGBS (at 70/30 - CL90Q/GGBS) to enhance the production of cementing hydrates (Walker et al. 2014). An amount of concrete was weighted to ensure a dry density of $c.450 \text{ kg/m}^3$.

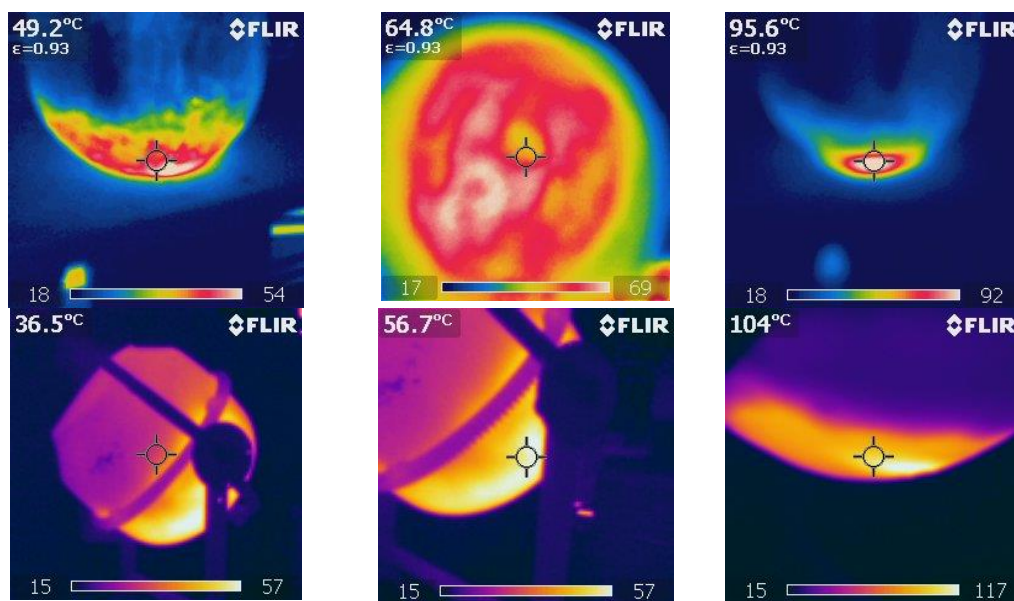
Table 2. Trial mixes and selection. L-lime. H-hemp. W-water.

Mix No	L: H: W (mass)	CS (MPa)
1	2.00: 7.00: 1	0.14
2	2.00: 5.74: 1	0.23
3	1.00: 2.00: 2	Discarded
4	0.60: 3.00: 1	Discarded
5	2.00: 5.40: 1	0.30
6	1.43: 4.43: 1	0.20
7	1.25: 3.82: 1	0.20
8	1.43: 3.63: 1	0.31
9	1.30: 3.60: 1	0.26
10	1.43: 5.43: 1	0.17
11	1.43: 5.00: 1	0.18
MIX QL = L: H: W		
12	1.43: 4.57: 1	0.33
MIX QL+GGBS = L: GGBS: H: W		
13	1: 0.43: 3.60: 1	0.30-0.36



Fig. 3. Specimen trials in table 2: specimen 1-12, from top left to bottom right.

The thermocouples and a thermal image camera allowed to record the maximum temperatures of the exothermic reaction of lime slaking and the temperature change over time. It was evidenced that mixing the hemp with quicklime prior to slaking undermines the process lowering the slaking temperature (the highest temperatures using this mixing method were under 80°C). Therefore, the hemp was layered over the quicklime so that the hemp shiv prevented heat loss during slaking. The heat generation continued over 15 minutes and high temperatures (over 50°C) lasted for over 20 minutes assisted by the insulation provided by the hemp shiv. However, once the hemp was mixed with the binder, the temperature dropped very quickly.



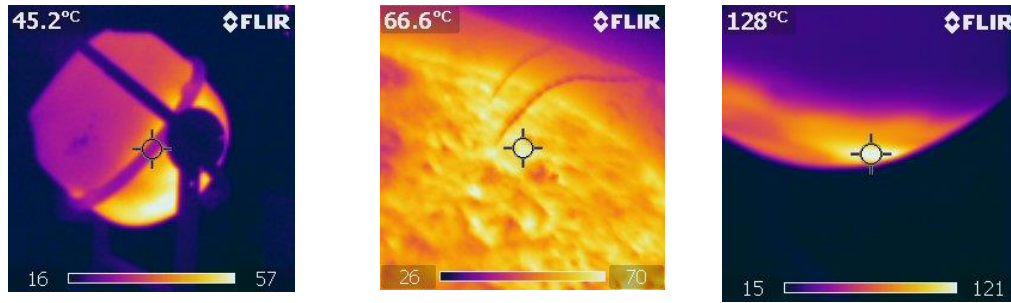


Fig. 4. Evolution of temperature during hot-lime mixing concretes 12 and 13-Table 2.

3.3 Properties of the hot-lime mixed hemp concretes

The hot-mixed hemp concretes show the typical mechanical properties of hemp-lime concrete described by former authors, and the ultimate values are slightly higher than others previously reported (Cerezo 2005; Nguyen 2010; Elfordy et al 2008; Amziane et al 2017). Walker et al. (2014), obtained compressive strength between 0.02 and 0.04MPa at 5 days and 0.29 and 0.39MPa at 1 year, when investigating lime-hemp concretes with pozzolans comparable to those in this paper.

Table 3. Properties of the hot-lime mixed hemp concretes at 3 months. Density = 430-470 kg/m³. λ = thermal conductivity. C_c = Specific heat capacity. δ = Water vapour permeability. μ = Water vapour diffusion resistance factor. A_w = Water sorption coefficient (kg/m² hour^{1/2}). COV: 0.6-9% for CS; 6.1-10.5 for FS; 18-32 for δ .

	CS	FS	μ	δ	A_w	R^2	λ	C_c J/kgK
	MPa	MPa		kg.m ⁻¹ .s ⁻¹ .Pa ⁻¹	kg/m ² hour ^{1/2}		W/(mK)	
QL	0.33	0.21	4.56	5.11×10^{-10}	4.58	0.83	0.021	1135
QL+GGBS	0.30-036	0.25	4.68	5.20×10^{-10}	4.24	0.87	0.015	1016

The hygric properties fall within the range observed by previous authors. Hemp-lime concretes typically have high water vapour permeability. The results conform with the common industry figure of water vapour diffusion resistance factor (μ) of lime-hemp concrete is 4.85 ± 0.24 measured in accordance with EN12572 for samples with a binder:hemp:water ratio of 2:1:3 and a density of c.400kg/m³ (Evrard 2008; Evrard et al. 2006).

The water absorption values also agree with former authors such as Collet (2004, 2009), Amziane et al. (2017); and Evrard (2008) reporting a water absorption coefficient of 4.42 ± 0.27 kg/m²h^{1/2} (0.0736 ± 0.0045 kg/m²s^{1/2}) for a 487kg/m³ density concrete made with a proprietary binder (DIN52617). When compared with lime-pozzolan hemp concretes (Walker and Pavia 2014), the hot-lime mixed concretes show slightly lower vapour diffusion resistance factors (4.56-4.68 vs 5.42-5.71) hence a higher permeability; and a marginally greater capillary suction, as shown by the higher water absorption coefficient values – 4.24-4.58 vs 2.65-3.37).

As lightweight materials, the thermal conductivity of hemp-lime concretes is typically low, resulting in outstanding U-values and good insulation properties. It can be very low ($0.04 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$) with typical values ranging between 0.1 and $0.3 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ (Amziane et al 2017). With respect to the specific heat capacity, hemp concrete has a high thermal mass when compared to other lightweight building materials. Previous research has identified a thermal heat capacity ranging between 1000 J/kgK for a concrete with a density of 413Kg/m³ and 1560 ± 30 J/kgK for a “wall mixture” with a density of 480 kg/m³, and values ranging from 1240 ± 172 to 1350 ± 279 (J/KgK). The specific heat capacity of the hot-mixed hemp concretes (ranging from 1016-1135 J/kgK) is slightly lower than the 1240-1350 J/kgK reported for lime pozzolan hemp concretes (Walker and Pavia 2014) and similar to the

values reached by LeTran et al. (2010) (1000 J/kgK) for hemp concretes of similar density to the ones in this paper.

3.4 Microstructure

As expected, the SEM analyses showed extensive carbonation, with abundant microcrystals of calcium carbonate resulting from the carbonation of the slaked quicklime. The calcium carbonate binder forms a continuous coating over the hemp particles that suggests a good adhesion at the interface which closely relates to strength and durability. However, the SEM analyses are qualitative, and hence no specific measurements can be made from the analyses. Both the QL and G+QL mixes show extensive carbonation, but some cementing hydrates were evidenced in the GGBS mixes. The presence of occasional hydrates does not seem to affect the physical properties of the concretes in a significant manner.

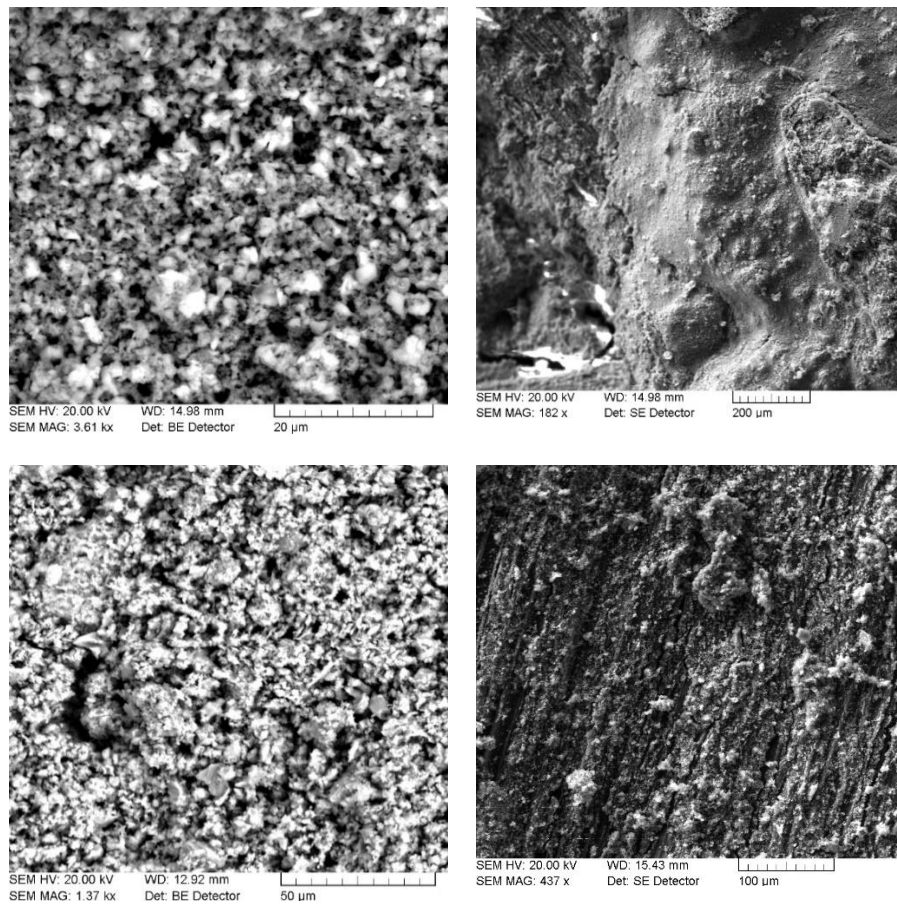


Fig. 5. Hot lime mixed hemp concrete (QL mix) showing extensive carbonation, with abundant microcrystals of calcium carbonate (left images) coating hemp particles (right images) providing a good adhesion at the interface.

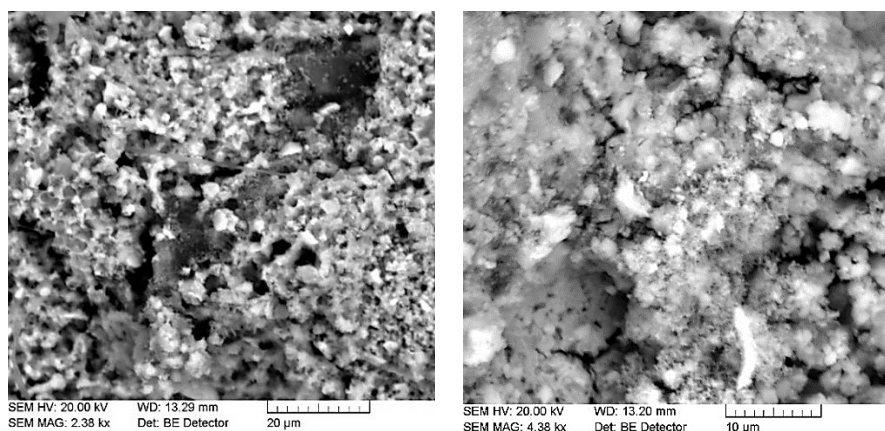


Fig. 6. Hot lime mixed hemp concrete (QL + GGBS mix) under the SEM showing extensive carbonation, with abundant microcrystals of calcium carbonate and some cementing hydrates (needles -left image) and low crystallinity gels (all over).

Conclusion

This paper investigates whether hot-lime mixing hemp concretes can improve the properties of these materials. In hot-lime mixing, the exothermic reaction of lime slaking takes place simultaneously to mixing. It was hoped that the heat generated on quicklime slaking would reduce the water intake by the shiv, block shiv pores and promoting adhesion between the shiv and the lime matrix.

It is concluded that the mixing process needs to avoid water competition between the shiv and the slaking process. Mixing the hemp with quicklime prior to slaking undermines the process lowering the slaking temperature. The best mixing method involved layering the hemp over the quicklime. The quicklime used in this research (CL90Q) is highly reactive, this results in a strong competition for water between the shiv and the slaking reaction: the shiv tends to absorb the water that the lime requires for slaking. A less reactive quicklime may render different results.

The properties of the resultant hot-lime-mixed, hemp materials slightly improved with respect to others in former literature, but they didn't improve substantially. The hot mixing method for hemp-lime composites provides a slightly superior mechanical strength than other binders at an early age (3 months), but the improvement is small.

As expected, the SEM analyses showed extensive carbonation, with abundant microcrystals of calcium carbonate resulting from the carbonation of the slaked quicklime. The calcium carbonate binder forms a continuous coating over the hemp particles that suggests a good adhesion at the interface which closely relates to strength and durability.

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