Performance of Lightweight Hemp Concrete with Alkali-Activated Cenosphere Binders Exposed to Elevated Temperature

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Abstract

This study investigates the performance of three different types of cenosphere as a lightweight supplementary cementitious material for alkali-activated binder for lightweight carbon-negative hemp-concrete for non-load bearing applications. Mechanical performance of hemp concrete exposed to three temperatures, room temperature (RT), 300 °C and 600 °C are studied using mechanical testing, thermogravimetric analysis (TGA) and Fourier-transform Infrared Spectroscopy (FTIR). Hemp concrete with cenosphere binder remained its integrity and showed a lower load carrying capacity even after exposure to elevated temperatures. Compressive strength capacity and elastic modulus of the samples reduced with the increase of temperature and shows the composite material is more suitable for non-load bearing application considering its mechanical behavior and fire requirements. The density, age, and type of cenosphere showed effects on mechanical properties at room temperature and elevated

temperatures. The study shows that alkali activated cenosphere binders can potentially be a sustainable alternative to the lime binder.

Keywords: Hemp concrete; Elevated Temperature; Low-Carbon building material; Fire Resistance; Cenosphere; Alkali-activated Binder; Stress-strain behavior; Elastic modulus; Compressive strength; Failure mechanisms; Lightweight

1 Introduction

Shiv made of stalk of the hemp plant, and in some cases, fibres which are by-products of hemp farming are mixed with lime binder to produce lightweight hemp concrete [1]. Due to porous cellular structure, the hemp shivs have light-weight, insulation properties, acoustic and good hygrothermal properties and it is used as a bio-lightweight aggregate [2–4]. Hemp concrete has industrial application such as non-load bearing wall, slab and roof panels. However, due to porous nature and lesser strength of hemp shivs, hemp-lime concrete has a lower compressive strength relative to its density compare to other types of lightweight concrete such as aerated concrete [5]. Therefore, hemp concrete is more suitable for non-load bearing applications where load bearing capacity is not significant but thermal and acoustic properties are more important [6–8]. Moreover, carbon emission analysis for hemp lime concrete shows that it has the ability to sequester CO_2 –around 1.5-2.1 kgCO_2/kg (kilogram of carbon dioxide per kilogram of hemp concrete) [8–10] both the growing phase of hemp plant and in the curing process of lime, which absorbs CO_2 from the atmosphere in the carbonization process. Even though production of lime involves two CO_2 emission processes which are due to heating and calcination of limestone, it has a net negative carbon emission due to it is higher carbon sequestration rate within its life cycle. So that it considers as a carbon negative material [11,12]. In addition, by increasing the percentage of hemp shiv by 20%, the thermal conductivity of the hemp concrete can be reduced by 67% [7] which will contribute to the reduction of energy

consumption and carbon dioxide emission of buildings. Therefore, having a lower density with higher amount of hemp shiv and lower amount of binder results a higher thermal performance and decrease the operational energy and emissions.

In the experimental programs, binders such as starch [13], cement [13,14], clay [13,15] metakaolin, and slag [15] are investigated for hemp concrete. In addition, mix of different binders consisted of organic and/or inorganic cementitious materials such as lime and cement, cement and magnesium oxide, lime and metakaolin, starch and magnesium oxide are studied [13,15–17]. In general, research showed that mechanical properties varied with the type of the binders and binders made of starch and cement showed relatively higher compressive strengths than the lime binder.

Even though hemp-lime concrete is a carbon negative material, lime, the binder has a higher embodied energy than cement, results in a range of 5.3-5.63 MJ/kg [18,19], and its heating and calcination emit CO₂ in a range of 0.42-0.8 kgCO₂/kg of lime [11,20] and 0.7 kgCO₂/kg [20], respectively. Therefore, using a binder with a lower carbon footprint and an embodied energy while sustaining the performance of hemp concrete can further increase the sustainability and reduce the environment impacts.

Cenosphere is a by-product of coal power plant and known as top ash, and it is considered as a lightweight environmentally-friendly raw material and filler with a good fire performance and thermal properties due to its gas-filled hollow nature [21,22]. Moreover, due to its cementitious behaviour it can use as a supplementary cementitious material (SCM) which can further enhance the mechanical properties of hemp concrete while keeping lightweight characteristics [22]. SCM such as cenosphere (top-fly ash), fly ash and slag are by-products and do not emit large amount of CO₂ or do not have high embodied energy like cement or lime [23–25]. Alkali activated binders made from fly ash and slag have lower carbon footprint and

embodied energy compared to cement-based binders [26,27]. Therefore, alkali-activated binders consist of lightweight cenosphere, fly ash and slag could be an attractive alternative binder for hemp concrete for developing building elements with lower embodied energy and carbon footprint.

Fire resistance level (FRL) of non-load bearing walls is an important and panels must satisfy the required FRL [28]. The fire resistance of walls is assessed based on the time that the elements can maintain its structural adequacy/integrity/insulation when exposed to fire. For instant, internal partition wall of a residential building smaller than 300 m² must have a fire rating level of 60/60/60 and -/60/60 for non-load bearing and non-load bearing walls according to AS 1530.4 [29], respectively. Since non-load bearing walls does not intend to carry any loads, structural adequacy is not tested for non-load bearing elements. Where the grading period is given in minutes for three criteria: structural adequacy, integrity, and insulation, respectively. However, fire performance of hemp concrete which contains organic hemp shiv and fibre is a major constraint to use hemp concrete in the construction industry. Lubos and Gregor [30] evaluated the fire resistance of hemp panels when exposed to standard time-temperature curves as given AS 1530.4 [29] and found that its performance is dependent on density and thickness of the panels. According to the experimental results [30], hempcrete panel requires a thickness of 400 mm to achieve FRL of -/60/60. In order to increase the FRL of hemp concretes, research [30] suggested increasing the thickness, and density of hemp concrete panels and providing an external protective layer such as MgO board or a rendering. However, increasing the density increase the weight of the panels, and providing thicker panels increases the wall thickness and reduces the floor area. Experimental program [16] to evaluate the fire reaction of low density (LD) and medium density (MD) hemp concrete panels according to EN 13823 [31] carried out. The study found that LD and MD panels have a quite good resistant to the fire with a class “C-s2-d0” and “B-s2-d0”, respectively. Class C is

prescribed as limited contribution to fire with similar performance as phenolic foam while class B contributed less to fire than class C and performs similarly to plaster boards with thin combustible cover. Researchers [32,33] reported that two fire tests on hempcrete walls subject to 135 kN of vertical load according to BS EN 1365-1 [34] resisted for 73 minutes in respect to integrity, insulation, and load-bearing capacity. As concluded by researchers [30,32], data related to performance of hemp concrete exposed to fire or elevated temperature is limited though fire performance is critical for load bearing and non-load bearing elements.

As discussed, the fire performance of hemp concrete can be increased by increasing the amount of binder, thickness of wall, and type of binder so that it can act as a shield around the hemp shiv. The density can be increased through the compaction or increasing the amount of binder. However, adding more binder increases the density of hemp concrete and increase the weight. Therefore, using a lightweight cenosphere binder (around 1400-1600 kgm⁻³) which is lighter than lime (around 1750-1900 kgm⁻³) can produce lighter panels with higher binder content. Experimental results from research [35] showed that increase of the amount of binder increases the mechanical properties such as compressive strength and modulus of elasticity. Thus, using a lighter binder can provide a higher binder content and can potentially improves mechanical properties and fire performance while having a lower density. Therefore, performance of hemp concrete with cenosphere-based binders exposed to high temperature is evaluated in this study.

2 Experimental Program

2.1 Materials

2.1.1 Cenosphere

Three types of cenosphere available in the industry were used in the experimental program. In this study, they are named as CEN, ESP, and POZ. The physical appearance of cenosphere

119 sourced from three different sources are shown in Figure 1. Figure 2 shows the images of
120 scanning electron microscopy analysis. The cenosphere are thin-walled microsphere with a
121 shell thickness of 5 to 20 μm and diameters in a range of 68-232 μm . Figure 3 shows the particle
122 size distribution of the cenospheres.

Figure 1: Three types of cenosphere

Figure 2: SEM images

124 Bulk density and true density of CEN, ESP, and POZ are given in Table 1. Chemical
 125 composition and phase identification of the cenosphere as given by XRD and XRF respectively
 126 are given in Table 2.

Figure 3: Particle size distribution of the cenospheres

127

128

Table 1: Physical Properties of cenosphere

	Unit	ESP	CEN	POZ
Bulk Density	kgm ⁻³	350	480	150

True Density	kgm ⁻³	630	830	330
Diameter Range				
Range	µm	91-232	70-192	68-231
Average	µm	147	117	129
Shell thickness	µm	12-20	3-5	8-10
Strength	MPa	40	30	40

Table 2: Chemical composition and phase identification of the cenospheres

Chemical Compound (mass %)	CEN	ESP	POZ
Na ₂ O	0.6	0.4	0.6
MgO	0.8	1.0	0.9
Al ₂ O ₃	29.4	29.1	26.8
SiO ₂	62.1	61.0	64.5
P ₂ O ₅	-	0.1	0.2
SO ₃	-	-	-
K ₂ O	1.6	2.2	2.5
CaO	0.7	0.7	0.5
TiO ₂	1.1	1.0	0.9
MnO	-	-	-
Fe ₂ O ₃	3.45	4.2	3.1
Mineral composition (wt%)			
Mullite	8	12	8
Quartz	3	6	7
Amorphous phase	88	82	85

All three cenosphere seems to have a similar range of size between 40 µm and 300 µm. However, according to figure 3, CEN presents slightly smaller particles than POZ or EPS. this difference in size could influence the reactivity of the binder by increasing nucleation sites and hence increase the reactivity of the binder. Further, POZ have a small population of particle in between 0.1 and 0.5 µm, such small population can hardly influence the behaviour of the binder. Table 1 shows some properties of cenosphere using SEM images and nanoindentation, respectively.

X-ray diffraction spectra of the cenosphere materials are shown in Figure 4. The cenospheres contained mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$) and quartz (SiO_2), and amorphous phase shown as “humps” in the spectra. As shown in Table 2, CEN seems to present a higher content of amorphous compounds which might increase its reactivity in cementitious system. High amounts of the amorphous phase with SiO_2 in the cenosphere particles raise the question of long-term stability of the cenosphere in alkaline environment in cement composites.

Figure 4 X-ray diffraction spectra of the cenosphere

2.1.2 Hemp Shiv

The hemp shiv sourced from a local supplier with a bulk density of 188 kgm^{-3} were used as the lightweight bio-aggregate. The studies by researchers [35,36] showed that using fine hemp particles improves the mechanical properties. Therefore, in this study smaller hemp particle in a range of 1-5 mm are used, and larger particles are removed using 5 mm sieve. The moisture content at Saturated Surface Dry (SSD) condition was 18%: according to AS 1141.6 [37]

2.1.3 Cementitious Material

A Class F fly ash complying with AS 3582.1 [38] and a slag cement satisfying the specifications of AS 3582.2 [39] were used in the experimental program. Silica fume which

satisfied the requirements as specified in AS 3582.3 [40] was used. A natural river sand with a fineness modulus of 2.6 and apparent particle density of 2610 kg/m³ was used as the fine aggregate. Potassium hydroxide and potassium silicate (KOH/KSiO₃) solution is used as an activator. The KOH/KSiO₃ solution had a concentration of 720 g/L. The solution consisted of 40% KOH and 60% of KSiO₃ by weight.

2.2 Mix Design

Three different alkali-activated binders using three different cenospheres-CEN, ESP, and POZ were used to prepare the samples. Considering the cost of cenosphere, cenosphere is mixed with fly ash, slag, silica fume, and sand. Hemp samples with a wet density around 1100 kgm⁻³ and dry density of 650 kgm⁻³ were made using the three binders. Two different wet densities are studied for ESP binder in order to investigate the effect of binder content. The study is part of developing a prefabricated composite lightweight-exterior cladding system as an alternative to brick veneer for residential building. In that case, panels must resist some impacts as well as fire. Considering the thickness of panels and ability to resist impact loadings, a higher-densities such as 900 kgm⁻³ and 1200 kgm⁻³ were considered. The composite panel consists of two layers of higher density hemp and lower-density hemp. Therefore, reduction in thermal performance due to higher densities will be compensated by the lower-dense hemp layer of the composite panel.

In order to get the lower density mix, ESP-900 half of the binder of Mix 01 is used for Mix 02. With the compaction of 0.7, final dry density of 1200 kgm⁻³ and 900 kgm⁻³ were expected for higher and lower density samples, respectively. The mix design proportions for CEN-1200, ESP-1200, POZ-1200, and ESP-900 are given in Table 3: first term is representing the type of cenosphere in the binder, and second term represents the dry density at 14 days.

Table 3: Mix design

	Mix No		Mix 01	Mix 02	Mix 03	Mix 04
Materials	True Density (kg/m ³)	Unit	ESP-900	ESP-1200	CEN-1200	POZ-1200
Hemp shiv	200	m ³	1	1	1	1
Slag	2860	kgm ⁻³	95.7	191.3	191.3	191.3
Fly ash	2290	kgm ⁻³	9.6	19.1	19.1	19.1
ESP	630	kgm ⁻³	49.9	99.8		
CEN	830	kgm ⁻³			99.8	
POZ	330	kgm ⁻³				99.8
Silica fume	2180	kgm ⁻³	20.4	40.8	40.8	40.8
Activator	1120	kgm ⁻³	44.6	89.3	89.3	89.3
Sand	2610	kgm ⁻³	138.4	276.8	276.8	276.8
Water	1000	kgm ⁻³	79	158	158	158
Total water	1000	kg	96.9	193.7	193.7	193.7
Water/binder			0.55	0.55	0.55	0.55
Hemp/binder			1.14	0.57	0.57	0.57
Total weight		kg	659	1097	1097	1097
Wet Density		kgm ⁻³	659	1097	1097	1097
Wet Density after Compaction		kgm ⁻³	1013	1567	1567	1567

2.3 Specimen Preparation

Nine hemp cylindrical samples were prepared for each mix and in total thirty-six samples with a diameter of 50 mm and height of 100 mm are prepared. Cylindrical samples were prepared to test the compressive strength at room temperature (around 15-20 °C), 300 °C, and 600 °C. Samples names were given in the following format: CEN-1200-14d-300T-s1; where CEN, 1200, 14d and s1 stand for cenosphere type, dry density at 14 days, age of testing, heated temperature and sample number, respectively. In sample naming, RT refers to room temperature. Samples were air cured in the room temperature.

2.3.1 Pre-soaking

Due to the excess water absorption of hemp shiv binder was less workable and binder didn't have enough water for the alkali-reaction. Considering that, the hemp shiv was soaked for 24 hours by a KOH/KSiO₃ solution at temperature around 20 °C. KOH/ KSiO₃ solution with a concentration of 228 g/L is selected for pre-soaking so that it is similar to the concentration of the final mix where water and activator solution added together. The soaked hemp was strained

and dried until it became to SSD condition. A similar method was used by Huiskes et al [5] for porous lightweight aggregates with higher absorption and Sedan et al. [41] presented that alkaline treatment on hemp increased the mechanical properties, indicating that interfacial bonding is improved.

2.3.2 Mixing and Compaction

Dry mix of slag, fly ash, silica fume and sand were poured into the Hobart mortar mixer and mixed for three minutes at a low-speed of 25 rpm in order to get a uniform dry mix. The activator solution was poured into the mixer and mixed for another one minute at low speed. Half of soaked hemp shiv and water was added and mixed for another three minutes at 50 rpm. Finally, the remaining amount of hemp was added and mixed for three minutes at 50 rpm. Cylindrical moulds (50x100 mm) were filled with hemp concrete mix and compacted. The compaction is done in three layers using a rod with a diameter, height and mass of 25 mm, 75 mm, and 280g, respectively. Ten drops from 25 mm height were given for each layer for the compaction. According to the trials, the compaction ratio in a range of 0.65-0.7 are achieved for higher and lower density samples, respectively. Wet densities of samples were measured after casting. After 7 days, the samples were demoulded and cured in a temperature around 20 °C in an oven for 14 days then samples were kept in room temperature. After 14 and 56 days, dry densities of samples were calculated prior to testing.

2.3.3 Heating

Some of the samples were heated up to 300 °C and 600 °C using an oven for 90 minutes. The temperature curve for heating process is given in the Figure 5. In terms of heating samples up to 300 °C, the temperature in oven took 30 minutes to reach 300 °C at a rate of 10 °C/min and remained 300 °C for 60 minutes before natural cooling in the oven TO room temperature. Temperature of 300 °C was selected because TGA results showed that evaporation of excess water and burning of hems shiv ends around 300 °C. The heating rate and the maximum

217 temperature were selected to be lower than that of the heating curve defined by AS 1530.4 [29]
 218 to prevent spalling [42]. To assess the performance of material after 90 min, targeting -/90/90
 219 FRL, heating time was selected as 90 minutes. Samples were weighted before and after heat
 220 treatment in order to measure the mass loss. Figure 6 shows the nature of the samples before
 221 and after heating.

Figure 5: Heating curve

222

Figure 6: Samples before and after heating (CEN)

223 2.4 Test Setup, Instrumentation, and Testing

224 The compressive strengths of samples are measured using a universal testing machine, and load
 225 versus displacement behaviour is captured. Displacement control loading method with a

displacement rate of a 4 mm/min was used. The test set up for the compressive strength testing is shown in Figure 7. Microscopic images were taken from the cross-section of the samples at room temperature by a Leica M205FA automated microscope to compare the interfacial bonding characteristics in three groups of samples.

2.4.1 Compressive Strength and Elastic Modulus

The samples were tested at 14 days and 56 days so that time-dependent behaviour can be observed. Compressive strength at 14 days selected in order to evaluate early strength with excess moisture content and 56 days selected in order to evaluate the properties where excess moisture is evaporated. Also, compressive strength of cenosphere binders at 14 days and 56 are evaluated to observe the variation of properties with the time and compare results with properties of hemp concrete. Proof stress of 0.1 % is used to calculate the compressive strength and elastic modulus of hemp concrete is evaluated using stress-strain curve.

Figure 7: Test set-up

2.5 Fourier transform infrared spectroscopy

Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR) is used to analyse the chemical bonding structure of the samples at room temperature and after exposure to 600°C using a Varian FTS 7000 FT-IR spectrometer, with a Specac MKII Golden Gate single reflectance diamond ATR attachment with KRS-5 lenses. Absorbance spectra are collected with a 4000-400 cm⁻¹ wavenumber at a resolution of 2 cm⁻¹ and a scanning speed of 5 kHz with 64 scans.

2.5.1 Thermogravimetric analysis

Thermo-gravimetric analysis was conducted in a thermogravimetric analyser (Perkin-Elmer Pyris). Blank experiments were performed to achieve the baselines and then calibrated with samples after testing. For each test, sample of 35 – 40 mg was loaded to the ceramic crucible of the analyser and heated from 30°C to 1000°C with the heating rate of 10°C/min. For this analysis, the heating chamber was ventilated with Air at the flow rate of 80 mL/min.

3 Results, Analysis and Discussion

3.1 Microscopic analysis of hemp concrete

Figure 8 shows the microscopic images of the hemp concrete at the room temperature. The cross section of the samples shows the interfacial bonding between the binders and hemp particles. Comparing the gap between the binder and hemp particles shows some differences between the interfacial bonding with hemp. POZ binder shows the biggest gap at the interface followed by the EPS which also shows an air gap at the interface. CEN samples show the best interfacial bonding with minimum gap between the hemp particles and the binder.

Figure 8. Microscopic images of the cross-section of samples at room temperature

3.2 Mechanical Properties of Hemp Concrete

3.2.1 Variation of Density with Age and Temperature

Table 4 shows the density of binders and hemp concrete at different stages. Wet density of hemp concrete is approximately similar for Mix 1, 3, and 4 and lower for Mix 2 which has a

lower-binder content. The dry densities of samples decreased with the time due to evaporation of excess moisture. Therefore, mix design of hemp concrete for different densities shall account the density variation with the time due to evaporation of excess moisture. At 14 days, there wasn't a significant difference in density for the mixes. However, at 56 days, CEN showed a lower density relative to ESP and POZ Mixes. After heating, the density is further reduced due to evaporation of trapped moisture and burning of hemp samples. The density decreases with the increase of temperature.

Table 4: Density of mixes

Sample Id	Wet Density	Hemp concrete				Binder Only	
		Dry Density		After Heating		Dry Density (56d)	After Heating
		14 days	56 days	14 days	56 days		
		(kgm ⁻³)				(kgm ⁻³)	
ESP-900d-RT	1056	965	867	-	-	1554	-
ESP-900d-300				696	709	1578	1268
ESP-900d-600				588	550	1581	1185
ESP-1200d-RT	1576	1172	1070	-	-	1554	-
ESP-1200d-300				922	929	1578	1268
ESP-1200d-600				838	885	1581	1185
CEN-1200d-RT	1514	1128	988	-	-	1516	-
CEN-1200d-300				840	905	1546	1320
CEN-1200d-600				773	754	1579	1210
POZ-1200d-RT	1554	1145	1083	-	-	1565	-
POZ-1200d-300				898	926	1590	1364
POZ-1200d-600				839	854	1604	1300

3.2.2 Failure Mechanism and Compressive Strength

Figure 9 shows the typical failure modes of hemp concrete for heated and non-heated samples. The samples were crushed when load was applied and bulged-out with the increase of deformation. For the lower density, samples were able to squeeze more relative to the samples with higher density due to higher void ratio and hemp content. The failure mechanism is different to the concrete samples because of crushing mechanism and different to typical cone type failure for mortar and concrete. For sample exposed to elevated temperature couldn't go through large deformation and the failure was brittle than non-heated samples. As shown in

Figure 9, non-heated sample did not spall or break into pieces when the test was carried out and able to keep its integrity while undergoing large deformation. However, for heated sample broke into pieces and spalled while the testing.

Figure 10 shows compressive strength of hemp concrete and binders for the cenosphere at 14 and 56 days. Table 4 concluded that the density of sample is changing with the time. The density of light-weight concrete is correlated to the compressive strength [5,43]. In order to give an accurate demonstration of compressive strength relative to the dry density, compressive strength plotted along with density using two Y-axes and a shared X-axis. For 14 days results, the samples with 1200 density had similar compressive strength. ESP-900 had the lowest strength which has the lower density or the binder content. At 56 days, higher compressive strengths were given by POZ and CEN mixtures, and it is aligned with the binder results because compressive strength of ESP binder reduces at 56 days and respective hemp mix gave a lower compressive strength. As shown in Figure 10, compressive strength of mixes decreased with the time. The compressive strength of ESP-1200 was significantly reduced at 56 days relative to other mixes. Similarly, compressive strength of ESP binder reduced at 56 days relative to 14 days results. The decrease of the compressive strength in cement-based sample including cenosphere has been observed before in the literature [44–46] and confirmed here. It can be explained as follows: Adding the cenosphere in a binder can develop a high strength for a lower density. It can behave as a filler, hence increasing the density as well as a pozzolanic addition due to their chemical composition and amorphous nature. However, as the cenospheres are hollow, after a certain time, the surface of the shell is consumed, causing the structure to become disintegrated and the creation of microstructural cracks and shell less voids which in terms affects the strength and performance of the material. It has been observed in the literature [44–46] that a higher replacement of cenospheres have tortuous and cracked patterns,

303 with the cracks going through the cenospheres as the weakest regions which shows the energy-
304 dissipating mechanism.

305 The compressive strength results showed that hemp concrete with CEN and POZ had
306 approximately similar results at 14 and 56 days for non-heated samples. The results concluded
307 that density and compressive strength of hemp concrete influenced by the type of cenosphere.
308 Also, compressive strength of hemp concrete reduced with the time and the percentage of
309 reduction was influenced by the types of the cenosphere though the compressive strength
310 increases in the binder for CEN and POZ mixes. Therefore, it can be assumed that binder may
311 not be the reason behind the decrease in compressive strength of hemp concrete. Density of
312 samples was reduced with the time due to evaporation of water which causes reduction in
313 density. Further reduction of density results from removal of pore water within the hemp shiv
314 can be a reason behind reduction in strength.

315

ESP-900d-56d-RT-s1

ESP-1200d-56d-RT-s1

ESP-900d-56d-300-s1

ESP-1200d-56d-300-s1

ESP-900d-56d-600-s1

ESP-1200d-56d-600-s1

Figure 9 Failure mode of samples

a). Hemp concrete

b). Binder -standard deviation

Figure 10: Compressive strength of hemp concrete and binders

317 Figure 11 shows the variation of compressive strength with the temperature after exposed to
 318 elevated temperature. The compressive strength of samples exponentially decayed with the
 319 increase of temperature. At 14 days, CEN had the highest compressive strength for heated
 320 samples. In contrast, ESP showed highest compressive strength for non-heated samples.

However, at 56 days, POZ showed the highest compressive strength for heated and non-heated samples. ESP-900 showed the lowest compressive strength for heated samples. This concluded that compressive strength after exposure to elevated temperature is dependent on density, types of cenosphere and the age of the hemp concrete. Different lime binders are used in the literature with densities in a range of 1733-1989 kgm⁻³ and compressive strength in a range of 1.2-3.0 MPa [47–49]. Table 4 and Figure 10 showed that cenosphere binders have a lower density and similar strength range than the lime binders in the literature.

Figure 11: Compressive strength after exposure to elevated temperature

3.3 Stress-Strain Behaviour and Toughness

Stress-strain behaviour is an important mechanical property in order to evaluate structural behaviour, capacity and evaluate the suitability for load-bearing applications. As well as, it is used to evaluate the ductility and toughness which is an important characteristic for load bearing and non-load bearing. The toughness represents the ability to undergo large deformation and stored plastic energy until the fracture. This is important for both load bearing and non-load bearing structural element to evaluate its resilience in case of excessive deformation due to earthquakes, low-velocity impacts, and differential settlements. It must be noted that the failure of the sample happened due to crushing and failure was highly localized around the top or the bottom where load was applied. Therefore, it should be noted that

calculating strain using displacement and sample height may not represent the accurate strain of the sample. However, in this study for the comparison purposes, strain was calculated using full sample height. Figure 12 shows the general trend of stress-strain behaviour of mixes. The stress-strain behaviour shows that the sample with lower density, ESP-900 is more ductile than sample with higher density. Figure 12 shows that stress increases with the increase of displacement after yielding for ESP-900. Thus, stress hardening behaviour is shown by ESP-900 at 14 days. However, at 56 days, ESP-900 showed a plastic behaviour with a plateau after the peak and didn't show a stress hardening. The stress-strain behaviour of ESP-900 was different to the typical stress-strain behaviour of concrete or mortar, and it didn't show a brittle behaviour with a descending branch after reaching to the peak. The ductile behaviour is suitable because plaster boards for exterior cladding elements such as cement, calcium silicate and gypsum boards are brittle and vulnerable to cracking. Lower density samples which has a lower binder content and a higher hemp shiv content had a higher ductility and can undergo large deformation without reduction in capacity. For higher density, post-peak branch of stress-displacement curve was descending without an abrupt reduction in the stress. The result shows that steepness of the descending branch increases with the time and samples are more brittle at 56 days than 14 days. Stress-strain behaviour at 300 and 600 °C shows that strengths of samples reduce significantly with the increase of temperature. Table 5 shows the modulus of elasticity and toughness of the samples at 56 days. Toughness is calculated using I_{10} and I_{20} toughness indices as given in ASTM C 1018 [50]. Higher toughness index implies that material can absorb energy and can plastically deform without fracturing. Stiffness and toughness of the samples decreased significantly with the increase of temperature. POZ-1200 had a higher toughness value than the other samples. Also, ESP-900 had a higher toughness values than ESP-1200 and CEN-1200 due to its higher deformability contrast to the lower strength.

Figure 12: Stress-strain behaviour at 14 and 56 days

Table 5: Properties after exposure to elevated temperature

Sample name	Temperature (°C)	Modulus of Elasticity (56d) (MPa)	Toughness (56d) (J/J)	
			I ₁₀	I ₂₀
ESP-900	RT	105	0.099	0.141
	300	52	0.006	0.007
	600	26	0.005	0.005
ESP-1200	RT	94	0.084	0.084
	300	54	0.012	0.012
	600	21	0.007	0.007
CEN-1200	RT	160	0.066	0.135
	300	68	0.014	0.020
	600	19	0.002	0.004
POZ-1200	RT	158	0.112	0.170
	300	95	0.012	0.015
	600	43	0.006	0.007

365 3.4 Modulus of Elasticity

366 Figure 13 shows the modulus of elasticity of hemp concrete for the mixes with different
 367 cenospheres and two densities. Figure 13 shows the modulus of elasticity versus temperature
 368 after exposure to the elevated temperature.

Figure 13: Elastic modulus hemp concrete and binders

369 The modulus of elasticity decreased with the increase of temperature. It appeared that modulus
 370 of elasticity decreased with the time. Similar to the trend of compressive strength versus

temperature, CEN and POZ mixes showed the highest modulus of elasticity at RT. POZ showed highest modulus of elasticity when exposed to elevated temperature at 56 days. ESP-1200 mix showed lower modulus of elasticity versus temperature relative to other mixes at 56 days. The results concluded that modulus of elasticity for heated samples are dependent on age and the type cenosphere.

Figure 14: Modulus of Elasticity after exposure to elevated temperature

3.5 Comparison of Compressive Strength of Different Binders

In order to compare the compressive strength of hemp concrete with different types of binders in the literature and the results of the present study, compressive strength versus density data are collected. Figure 15 shows the density versus compressive strength behaviour of different binders as given by researchers [6,9,13,14,17,35,47,48,51,52]. The regression lines for the lime binder in the literature and the present study are shown in Figure 15.

Figure 15: Comparison of Binders

Figure 15 concludes that starch binder gave higher compressive strengths relative to other inorganic binders such as cement, lime, and cenosphere binder. The compressive strength of cement binder is over the regression line of the lime binder. Therefore, higher strengths can be achieved for particular density using a cement binder and starch. Therefore, using starch and cement as alternative binders can produce hemp concrete with higher compressive strength relative to the traditional lime binder. The trend lines of lime binder and present study showed that performance of cenosphere binder can have similar compressive strengths relative to the lime binder: data of ESP at 56 days is omitted in the regression analysis considering its reduction of strength with the time.

Cement and lime have average embodied carbon footprint of 0.83 KgCO₂/Kg and 0.7 KgCO₂/Kg, respectively [18]; and have average embodied energy of 4.6 MJ/Kg and 5.3 MJ/Kg, respectively. However, starch is an organic material which fire performance is not good as other inorganic binders such as lime, cement, slag etc. In other hand, even though cement is an alternative inorganic binder, it has a higher CO₂ emission than lime and reduces the

sustainability of hemp concrete. The cenosphere binders used in the present study is made of supplementary cementitious materials such as cenosphere (top fly ash), fly ash, silica fume and slag which are by-products and doesn't emit large amount of CO₂ or have higher embodied energy as cement or lime [23–25]. In addition, fly ash and slag binders with alkali activation have lower CO₂ emission and embodied energy relative to cement-based binders [26,27]. Thus, the study shows that binder with suitable cenosphere can have similar compressive strengths to the lime binder and can be a more sustainable alternative to the lime binder.

3.6 FTIR analysis of the binders

Figure 16. FTIR results of binders

The band around 720 cm⁻¹ is known to be related to aluminosilicate ring structures [53] and can be used to compare the zeolite phases formed in samples [54,55] and the bands at about 780 cm⁻¹ are related to Al-O vibrations in Al₂O₃ structures [56,57]. The position of the band around 950 cm⁻¹ and 1080 cm⁻¹ gives an indication of Si–O absorption band in C-S-H and N-

A-S-H gels (at 960 cm^{-1} mainly related to C-S-H and at 1080 cm^{-1} mainly associated with for N-A-S-H) [58]. The peaks at 1400 cm^{-1} have been attributed to asymmetric stretching of Al–O/Si–O bonds [59]. The band at about 1500 cm^{-1} is related to the presence of sodium carbonate [60], and the band around 1660 cm^{-1} is related to bending vibration of water.

The FTIR spectra of all samples are similar at room temperature. However, there are differences between the FTIR results after heating samples at 600°C . The intensity of the N-A-S-H band at 1080 cm^{-1} increases for POZ at 600°C and it remain unchanged for CEN and ESP. The intensity of the C-S-H gel at 960 cm^{-1} drops in all samples. That is because geopolymer gels (N-A-S-H) are more resistant to the elevated temperature than C-S-H gels [61]. The intensity drops at 960 cm^{-1} is larger in POZ and EPS samples, but it is very small in the CEN sample. In overall, CEN binder showed more resistant to elevated temperature relative to ESP, POZ in term of C-S-H.

3.7 TGA Analysis

The thermal stability of hemp concrete is characterised by exposing the ground sample to elevated temperature of 1000°C . Thermal-gravimetric (TG) and reaction rate curves of hemp concrete samples are shown in Figure 17. In general, the mass loss percentage of all samples remains around 20 %wt with a marginally higher mass loss of ESP-900 owing to its lower density. There are three major changes during TGA analysis which correspond to physical water evaporation ($100\text{--}200^{\circ}\text{C}$), thermal degradation of hemp (between $400\text{--}600^{\circ}\text{C}$) [62] and the third degradation at high temperature (over 800°C). Similar trends were found with ESP-900, ESP-1200 and POZ-1200, but the second degradation of CEN-1200 was milder with the reaction rate half of those of ESP-1200 and POZ-1200. The lower degradation of hemp in CEN-1200 may explain its high compressive strength compared to ESP-1200 and POZ-1200 after fire exposure (Figure 11). CEN-1200 also possesses a high reaction rate (as high as $0.06\text{ \%}/^{\circ}\text{C}$)

432 at high temperature (over 800°C) which was not captured from the fire exposure conducted in
433 this paper.

a.) ESP-900-56d

b.) ESP-1200-56d

c.) CEN-1200-56d

d.) POZ-1200-56d

Figure 17: Thermal-gravimetric (TG) and reaction rate curves of hemp concrete

The difference of mechanical performance at elevated temperature couldn't be explained using TGA and FTIR results since CEN showed relative good performance in TGA and FTIR though POZ mix showed higher elastic modulus, a similar compressive strength of CEN after exposed to higher temperature at 56 days. One reason behind better performance of POZ can be due to formation of geopolymer N-A-S-H gel which can enhance the strength and elastic modulus as well as resistance to the elevated temperature. Also, as shown in **Error! Reference source not found.**, POZ has the lowest true density and similar mass of 99.8 kg is used for the mixes. Therefore, the volume of POZ is higher than of other mixes which is 0.3 m³ per 1 m³ of hemp

concrete though it is 0.16 m^3 and 0.12 m^3 for ESP and CEN, respectively. Therefore, POZ mix has a higher binder content relative to other mixes and authors suggested that can be a reason for better performance of POZ for elevated temperature and RT at 56 days even though CEN shows better characteristics in FTIR and TGA analysis.

4 Conclusions

The results on compressive strength concluded that compressive strength of heated and non-heated samples is dependent on type of cenosphere, density and the age of hemp concrete. Modulus of elasticity results followed a similar trend of compressive strength results: Modulus of elasticity reduced exponentially with the increase of temperature and age, type of cenosphere and density. POZ-1200 showed a higher elastic modulus at different temperatures at 56 days. For ESP binder, compressive strength decreases with the time though compressive strength of CEN and POZ binders increases with the time. As stated in literature, failure in outer-shell structure of cenosphere due to alkali reaction and creation of shell-less voids and cracks can be the reason behind reduction in mechanical properties of ESP binder. However, the same phenomena didn't observe in other mixes. Even though compressive strength of binders was increased with the time for CEN and POZ mixes, compressive strength of hemp concrete was decreased with the time. Therefore, it is important to observe the long-term behaviour of cenosphere based binders as well as hemp concrete considering the long-term performance of the materials.

Stress-strain relationship of samples concluded that behaviour was not brittle, and samples were able to undergo large deformation at room temperature. The stress-strain behaviour vary dependant on density and age of samples. The samples with a lower density can undergo large deformation which is suitable for internal and external claddings.

Regarding thermal stability, all samples showed a consistency with the mass loss of 20% with three stages of degradation. That is due to the similar quantity of hemp in the mixes. Thermogravimetric analysis of samples at 56 days showed the correlation between the lower degradation rate at 400-600°C of CEN-1200 and its higher compressive strength after exposure to 600°C. FTIR analysis showed that at high-temperature C-S-H get degraded notably for all sample except CEN mix. Also, geopolymer N-A-S-H gel formation was observed for POZ at 600 °C. In overall, FTIR analysis showed that CEN has a higher resistance than other mixes. The better performance of POZ may due to formation of N-A-S-H gel and higher binder content relative to other mixes due to its lower particle density.

The study showed cenosphere binder can achieve compressive strength similar to the lime binder while having a lower embodied energy and carbon emission relative to cement or lime binder. Thus, the study showed that an alkali-activated cenosphere binder has the potential to be an alternative sustainable binder to the traditional lime binder while satisfying the mechanical properties. Authors suggest more experimental program using different mix proportions for alkali-activated binders and geopolymer binders while considering cost of cenosphere in order established an economical and more sustainable binder for hemp concrete for future studies.

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491 6 Reference

- 492 [1] L. Kidalova, N. Stevulova, E. Terpakova, A. Sicakova, Utilization of alternative materials
493 in lightweight composites, *J. Clean. Prod.* 34 (2012) 116–119.
494 doi:<https://doi.org/10.1016/j.jclepro.2012.01.031>.
- 495 [2] M. Bell, The Potential of Hemp: A new approach to the material and its use within the
496 construction industry, 2012.
- 497 [3] U. Dhakal, U. Berardi, M. Gorgolewski, R. Richman, Hygrothermal performance of
498 hempcrete for Ontario (Canada) buildings, *J. Clean. Prod.* 142 (2017) 3655–3664.
499 doi:<https://doi.org/10.1016/j.jclepro.2016.10.102>.
- 500 [4] T.S. Kiessé, A. Ventura, H.M.G. van der Werf, B. Cazaciu, R. Idir, Andrianandraina,
501 Introducing economic actors and their possibilities for action in LCA using sensitivity
502 analysis: Application to hemp-based insulation products for building applications, *J.*
503 *Clean. Prod.* 142 (2017) 3905–3916. doi:<https://doi.org/10.1016/j.jclepro.2016.10.069>.
- 504 [5] D.M.A. Huiskes, A. Keulen, Q.L. Yu, H.J.H. Brouwers, Design and performance
505 evaluation of ultra-lightweight geopolymer concrete, *Mater. Des.* 89 (2016) 516–526.
506 doi:[10.1016/j.matdes.2015.09.167](https://doi.org/10.1016/j.matdes.2015.09.167).
- 507 [6] S. Benfratello, C. Capitano, G. Peri, G. Rizzo, G. Scaccianoce, G. Sorrentino, Thermal
508 and structural properties of a hemp–lime biocomposite, *Constr. Build. Mater.* 48 (2013)
509 745–754. doi:[10.1016/j.conbuildmat.2013.07.096](https://doi.org/10.1016/j.conbuildmat.2013.07.096).
- 510 [7] F. Collet, S. Prétot, Thermal conductivity of hemp concretes: Variation with formulation,
511 density and water content, *Constr. Build. Mater.* 65 (2014) 612–619.
- 512 [8] Y. Florentin, D. Pearlmutter, B. Givoni, E. Gal, A life-cycle energy and carbon analysis
513 of hemp-lime bio-composite building materials, *Energy Build.* 156 (2017) 293–305.
514 doi:[10.1016/j.enbuild.2017.09.097](https://doi.org/10.1016/j.enbuild.2017.09.097).
- 515 [9] A. Shea, M. Lawrence, P. Walker, Hygrothermal performance of an experimental hemp–
516 lime building, *Constr. Build. Mater.* 36 (2012) 270–275.
517 doi:[10.1016/j.conbuildmat.2012.04.123](https://doi.org/10.1016/j.conbuildmat.2012.04.123).
- 518 [10] C. Maalouf, C. Ingrao, F. Scrucca, T. Moussa, A. Bourdot, C. Tricase, A. Presciutti, F.
519 Asdrubali, An energy and carbon footprint assessment upon the usage of hemp-lime
520 concrete and recycled-PET façades for office facilities in France and Italy, *J. Clean. Prod.*
521 170 (2018) 1640–1653. doi:<https://doi.org/10.1016/j.jclepro.2016.10.111>.
- 522 [11] R. Busbridge, R. Rhydwen, An investigation of the thermal properties of hemp and clay
523 monolithic walls, (2010).
- 524 [12] A. Arrigoni, R. Pelosato, P. Melià, G. Ruggieri, S. Sabbadini, G. Dotelli, Life cycle
525 assessment of natural building materials: the role of carbonation, mixture components and
526 transport in the environmental impacts of hempcrete blocks, *J. Clean. Prod.* 149 (2017)
527 1051–1061. doi:<https://doi.org/10.1016/j.jclepro.2017.02.161>.
- 528 [13] G. Balčiūnas, S. Vėjelis, S. Vaitkus, A. Kairyte, Physical Properties and Structure of
529 Composite Made by Using Hemp Hurds and Different Binding Materials, *Procedia Eng.*
530 57 (2013) 159–166. doi:[10.1016/j.proeng.2013.04.023](https://doi.org/10.1016/j.proeng.2013.04.023).
- 531 [14] C. Niyigena, S. Amziane, A. Chateaneuf, L. Arnaud, L. Bessette, F. Collet, C. Lanos, G.
532 Escadeillas, M. Lawrence, C. Magniont, S. Marceau, S. Pavia, U. Peter, V. Picandet, M.

- Sonebi, P. Walker, Variability of the mechanical properties of hemp concrete, *Mater. Today Commun.* 7 (2016) 122–133. doi:10.1016/j.mtcomm.2016.03.003.
- [15] R. Walker, S. Pavia, R. Mitchell, Mechanical properties and durability of hemp-lime concretes, *Constr. Build. Mater.* 61 (2014) 340–348. doi:10.1016/j.conbuildmat.2014.02.065.
- [16] E. Sassoni, S. Manzi, A. Motori, M. Montecchi, M. Canti, Novel sustainable hemp-based composites for application in the building industry: Physical, thermal and mechanical characterization, *Energy Build.* 77 (2014) 219–226. doi:10.1016/j.enbuild.2014.03.033.
- [17] N. Stevulova, L. Kidalova, J. Cigasova, J. Junak, A. Sicakova, E. Terpakova, Lightweight Composites Containing Hemp Hurds, *Procedia Eng.* 65 (2013) 69–74. doi:10.1016/j.proeng.2013.09.013.
- [18] G. Hammond, C. Jones, F. Lowrie, P. Tse, Inventory of carbon & energy: ICE, Sustainable Energy Research Team, Department of Mechanical Engineering, University of Bath Bath, 2008.
- [19] B.V. Reddy, K.S. Jagadish, Embodied energy of common and alternative building materials and technologies, *Energy Build.* 35 (2003) 129–137.
- [20] K. Ip, A. Miller, Life cycle greenhouse gas emissions of hemp–lime wall constructions in the UK, *Resour. Conserv. Recycl.* 69 (2012) 1–9. doi:10.1016/j.resconrec.2012.09.001.
- [21] A. Arizmendi-Morquecho, A. Chávez-Valdez, J. Alvarez-Quintana, High temperature thermal barrier coatings from recycled fly ash cenospheres, *Appl. Therm. Eng.* 48 (2012) 117–121. doi:10.1016/j.applthermaleng.2012.05.004.
- [22] A. Hanif, S. Diao, Z. Lu, T. Fan, Z. Li, Green lightweight cementitious composite incorporating aerogels and fly ash cenospheres – Mechanical and thermal insulating properties, *Constr. Build. Mater.* 116 (2016) 422–430. doi:10.1016/j.conbuildmat.2016.04.134.
- [23] C. (Catherine E.L. De Wolf, Material quantities in building structures and their environmental impact, Thesis, Massachusetts Institute of Technology, 2014. <http://dspace.mit.edu.ezp.lib.unimelb.edu.au/handle/1721.1/91298> (accessed February 23, 2018).
- [24] C.C.E.L. De Wolf, Low carbon pathways for structural design: embodied life cycle impacts of building structures, Massachusetts Institute of Technology, 2017.
- [25] K. Kupwade-Patil, C. De Wolf, S. Chin, J. Ochsendorf, A.E. Hajiah, A. Al-Mumin, O. Büyüköztürk, Impact of Embodied Energy on materials/buildings with partial replacement of ordinary Portland Cement (OPC) by natural Pozzolanitic Volcanic Ash, *J. Clean. Prod.* 177 (2018) 547–554. doi:10.1016/j.jclepro.2017.12.234.
- [26] E. Jamieson, B. McLellan, A. van Riessen, H. Nikraz, Comparison of embodied energies of Ordinary Portland Cement with Bayer-derived geopolymer products, *J. Clean. Prod.* 99 (2015) 112–118. doi:10.1016/j.jclepro.2015.03.008.
- [27] B.C. McLellan, R.P. Williams, J. Lay, A. van Riessen, G.D. Corder, Costs and carbon emissions for geopolymer pastes in comparison to ordinary portland cement, *J. Clean. Prod.* 19 (2011) 1080–1090. doi:10.1016/j.jclepro.2011.02.010.
- [28] Q.T. Nguyen, T. Ngo, P. Tran, P. Mendis, L. Aye, S.K. Baduge, Fire resistance of a prefabricated bushfire bunker using aerated concrete panels, *Constr. Build. Mater.* 174 (2018) 410–420.
- [29] AS 1530.4, Methods for fire tests on building materials, components and structures Fire-resistance test of elements of construction, Standards Association of New Zealand, Sydney, Australia., 2005.
- [30] Lubos, Gregor, Performance of hempcrete walls subjected to a prescribed Fire Curve.pdf, Victoria University, 2014.

- [31] EN 13823, Reaction To Fire Tests For Building Products - Building Products Excluding Floorings Exposed To The Thermal Attack By A Single Burning Item, European Committee for Standardization, Brussels, Belgium, 2010.
- [32] P. Daly, P. Ronchetti, T. Woolley, Hemp Lime Bio-composite as a Building Material Irish Construction, Environ. Prot. Agency Irel. (2012).
- [33] W. Stanwix, A. Sparrow, The hempcrete book: designing and building with hemp-lime, Green Books Cambridge, UK:, 2014.
- [34] BS EN 1365-1, Fire resistance tests for loadbearing elements. Walls, British Standard Institute, London, UK, 1999.
- [35] L. Arnaud, E. Gourlay, Experimental study of parameters influencing mechanical properties of hemp concretes, Constr. Build. Mater. 28 (2012) 50–56. doi:10.1016/j.conbuildmat.2011.07.052.
- [36] N. Stevulova, L. Kidalova, J. Junak, J. Cigasova, E. Terpakova, Effect of Hemp Shive Sizes on Mechanical Properties of Lightweight Fibrous Composites, Procedia Eng. 42 (2012) 496–500. doi:10.1016/j.proeng.2012.07.441.
- [37] AS 1141.6, Methods for sampling and testing aggregates Particle density and water absorption of coarse aggregate - Weighing-in-water method, Standards Association of Australia, Sydney, Australia., 2000.
- [38] AS 1012.3.5, Methods of testing concrete - Determination of properties related to the consistency of concrete - Slump flow, T500 and J-ring test, Standards Association of Australia, NSW, Australia, 2015.
- [39] AS 3582.2, Supplementary cementitious materials for use with portland and blended cement - Slag - Ground granulated iron blast-furnace, Standards Association of Australia, NSW, Australia, 2001.
- [40] AS/NZS 3582.3, Supplementary cementitious materials for use with portland and blended cement-amorphous silica, Standards Association of Australia; Standards Association of New Zealand, NSW, Australia, 2002.
- [41] D. Sedan, C. Pagnoux, A. Smith, T. Chotard, Mechanical properties of hemp fibre reinforced cement: Influence of the fibre/matrix interaction, J. Eur. Ceram. Soc. 28 (2008) 183–192. doi:10.1016/j.jeurceramsoc.2007.05.019.
- [42] T. Drzymała, W. Jackiewicz-Rek, M. Tomaszewski, A. Kuś, J. Gałaj, R. Šukys, Effects of high temperature on the properties of high performance concrete (HPC), Procedia Eng. 172 (2017) 256–263.
- [43] Z. Zhang, J.L. Provis, A. Reid, H. Wang, Geopolymer foam concrete: An emerging material for sustainable construction, Constr. Build. Mater. 56 (2014) 113–127. doi:10.1016/j.conbuildmat.2014.01.081.
- [44] J.-Y. Wang, M.-H. Zhang, W. Li, K.-S. Chia, R.J.Y. Liew, Stability of cenospheres in lightweight cement composites in terms of alkali–silica reaction, Cem. Concr. Res. 42 (2012) 721–727. doi:10.1016/j.cemconres.2012.02.010.
- [45] D. Montgomery, S. Diamond, The influence of fly ash cenospheres on the details of cracking in flyash-bearing cement pastes, Cem. Concr. Res. 14 (1984) 767–775.
- [46] K. Senthamarai Kannan, L. Andal, M. Shanmugasundaram, An investigation on strength development of cement with cenosphere and silica fume as pozzolanic replacement, Adv. Mater. Sci. Eng. 2016 (2016).
- [47] M. Chabannes, E. Garcia-Diaz, L. Clerc, J.-C. Bénézet, 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. doi:10.1016/j.conbuildmat.2015.06.032.

- [48] E.A.J. Hirst, P. Walker, K.A. Paine, T. Yates, Characteristics of low-density hemp-lime building materials, *Proc. Inst. Civ. Eng. - Constr. Mater.* 165 (2012) 15–23. doi:10.1680/coma.1000021.
- [49] T.-T. Nguyen, V. Picandet, S. Amziane, C. Baley, Influence of compactness and hemp hurd characteristics on the mechanical properties of lime and hemp concrete, *Eur. J. Environ. Civ. Eng.* 13 (2009) 1039–1050. doi:10.1080/19648189.2009.9693171.
- [50] ASTM C 1018, Standard test method for flexural toughness and first crack strength of fibre reinforced concrete (Using beam with third-point loading), American Society of Testing and Materials, Philadelphia, United States, 1998.
- [51] Hempcrete Installation Facts Sheet, Hempcrete Aust. Pty Ltd. (2014). http://www.hempcrete.com.au/index.php?option=com_content&view=article&id=2&Itemid=3 (accessed March 14, 2017).
- [52] E. Sassoni, S. Manzi, A. Motori, M. Montecchi, M. Canti, Experimental study on the physical–mechanical durability of innovative hemp-based composites for the building industry, *Energy Build.* 104 (2015) 316–322. doi:10.1016/j.enbuild.2015.07.022.
- [53] W. Mozgawa, The relation between structure and vibrational spectra of natural zeolites, *J. Mol. Struct.* 596 (2001) 129–137.
- [54] C.A. Rees, Mechanisms and kinetics of gel formation in geopolymers, PhD Thesis, 2007.
- [55] C.A. Rees, J.L. Provis, G.C. Lukey, J.S. Van Deventer, The mechanism of geopolymer gel formation investigated through seeded nucleation, *Colloids Surf. Physicochem. Eng. Asp.* 318 (2008) 97–105.
- [56] W.K.W. Lee, J.S.J. Van Deventer, Use of infrared spectroscopy to study geopolymerization of heterogeneous amorphous aluminosilicates, *Langmuir*. 19 (2003) 8726–8734.
- [57] C.A. Rees, J.L. Provis, G.C. Lukey, J.S. van Deventer, Attenuated total reflectance fourier transform infrared analysis of fly ash geopolymer gel aging, *Langmuir*. 23 (2007) 8170–8179.
- [58] I. García-Lodeiro, A. Palomo, A. Fernández-Jiménez, D.E. Macphee, Compatibility studies between NASH and CASH gels. Study in the ternary diagram Na₂O–CaO–Al₂O₃–SiO₂–H₂O, *Cem. Concr. Res.* 41 (2011) 923–931.
- [59] X. Guo, H. Shi, W.A. Dick, Compressive strength and microstructural characteristics of class C fly ash geopolymer, *Cem. Concr. Compos.* 32 (2010) 142–147.
- [60] P.N. Lemougna, K.J. MacKenzie, U.C. Melo, Synthesis and thermal properties of inorganic polymers (geopolymers) for structural and refractory applications from volcanic ash, *Ceram. Int.* 37 (2011) 3011–3018.
- [61] N.K. Lee, K.T. Koh, G.H. An, G.S. Ryu., Influence of binder composition on the gel structure in alkali activated fly ash/slag pastes exposed to elevated temperatures, *Ceram. Int.* 43 (2017) 2471–2480. doi:10.1016/j.ceramint.2016.11.042.
- [62] I. Netinger Grubeša, B. Marković, A. Gojević, J. Brdarić, Effect of hemp fibers on fire resistance of concrete, *Constr. Build. Mater.* 184 (2018) 473–484. doi:10.1016/j.conbuildmat.2018.07.014.