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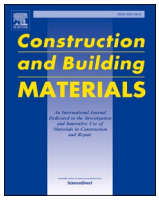

Mechanical and hygrothermal properties of hemp-silica bio-composites

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ABSTRACT

This research investigated the development of a fast-drying silica-based binder for hemp concrete products with enhanced mechanical and thermal properties. Hemp-silica bio-composites were prepared by mixing hemp shivs with a two-component binder system composed of liquid sodium silicate and tributyl citrate (TBC). Compressive strength, thermal conductivity, moisture buffering value, cyclic moisture resistance and microstructure of hemp-silica composites were analysed, and the results were compared with those of hemp-lime concrete. Hemp-silica blocks with shiv-liquid sodium silicate mass ratio of 1:3.75 and TBC content of 37.5 wt% of sodium silicate dry matter produced a compressive strength of 0.56 MPa only after 3 days of drying and 1.92 MPa after 28 days. These were higher than hemp-lime blocks at the same density range. Hemp-silica panels showed a thermal conductivity of 0.101 W/mK and an excellent moisture buffering value of 3.49. Hemp silica formed an open porosity with large air gaps between the particles and a water-resistance silica-based layer on the shiv surface producing a higher moisture resistance compared to hemp-lime systems. This paper focuses on the development of a novel fast-drying binder system with a potential for use in conjunction with other lingnocellular plant aggregates to form low-carbon and efficient multifunctional building materials.

1. Introduction

The construction and building industry is responsible for 39 % of CO_2 emissions globally 28 % of which is related to building operations and 11 % to materials embodied carbon [1]. Storing the biogenic carbon across a range of bio-products coupled with sustainable re-growth of biomass presents a remarkable opportunity to reduce atmospheric CO_2 emissions [2]. The use of harvested wood from forests, of which about 50 % of the dry weight is carbon, as a construction material has been widely practised [3]. However, it has been argued that storing carbon in fast-growing plants is much more efficient than in timber products. This is because the carbon stored in fast-growing biogenic materials is fully captured by crop regrowth only one year after construction, while a longer time is expected for forest products due to the long rotation period required for forest regrowth [4].

The development of "agro-concrete" products and plant-based composites for buildings, which can be qualified as environmental friendly and efficient multifunctional materials has been rapidly expanding [5]. In such applications, usually a lignocellulose-based plant aggregate (bio-aggregate) such as hemp (shivs), flax, straw, bagasse, etc. is mixed with a mineral binder to form a heterogeneous material aimed for various construction applications. The bio-aggregate provides not only excellent insulation properties but also enhanced ductility and post-fracture behaviour due to the porose and elongated structure of the plant stem.

One of the most common plants that have been used in agro-concrete products is hemp. Hemp (*Cannabis sativa L*) is a fast-growing annual plant that can grow up to 4 m in 4 months, with low fertiliser and irrigation demand, making it very efficient in the use of time and material resources [6]. The woody core of the hemp plant (shivs/hurds) is usually used as bio-aggregate in conjunction with inorganic binders such as lime to produce hemp-lime (hempcrete) bio-composites. These systems have received a great deal of attention as building materials due to their superior hygric and thermal properties [7,8]. Mechanical properties of hemp-lime concrete such as density and compressive strength are heavily dependent on hemp-to-binder mass ratios. Studies focused on the compressive strength of hemp-lime systems have reported strengths in the range of 0.2 – 1.2 MPa for hemp-to-binder ratios ranging from 1:1–1:4 [9]. Hemp-lime concrete is mainly used as a non-load-bearing material. The addition of Portland Cement and pozzolanic materials as binder replacement has been studied to improve the structural capability of hemp-lime [10–13]. A number of studies have also investigated

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the environmental profile of hemp-lime systems by conducting a life-cycle assessment (LCA) highlighting the lower impact of hempcrete compared to traditional/synthetic construction materials [14–18].

Hemp shivs have a cellular structure which is mainly composed of cellulose, hemicellulose, and lignin. The excellent hygric and thermal properties of hempcrete products are due to the high porosity and hydrophilic nature (hydroxyl groups of cellulose and hemicellulose molecules) of the hemp shives [19]. However, this also results in hemp shivs having large water absorption capacities which has a negative effect on the hydration/drying of lime-based binders as both hemp shivs and lime compete for the available water in the wet mix [20]. Therefore, hemp-lime systems require long drying durations (in months) to achieve sound structural integrity. Hemp-lime walls usually have a brittle inner core due to the binder incomplete hydration [21]. Alternative silica-based binders for hempcrete have shown promise to provide sufficient hygroscopic, thermal and mechanical properties while reducing the drying times to just a few days [22].

Liquid sodium silicate can be considered a low-cost silica-based binder for hemp concrete. However, the main challenge with the use of sodium silicate is the low water resistance of the material in its dry form. Esters have been used as an additive in silica-based grout to improve mechanical properties and moisture resistance [23,24]. This paper explores the use of sodium silicate as a quick-drying binder mixed with Tributyl citrate (TBC) for hemp concrete. TBC is used as a nature-based plasticiser to improve the moisture resistance and mechanical properties of bio-degradable polymers [25]. The hemp-silica composites were prepared and characterised for mechanical properties, thermal conductivity, moisture buffering capacity and water resistance, and the results were compared with conventional hemp-lime concrete products.

2. Materials and methods

Hemp shivs with an average particle size range between 4.0 mm and 30.1 mm and a bulk density of 145 g/cm^3 were used in this study (KANABAT - France) as shown in Fig. 1. For hemp-silica formulation, liquid sodium silicate (Chemiphase) with 37.5–39.5 % dry matter and 3.3–3.5 SiO_2/Na_2O molar ratio, and TBC (≥97.0 % $C_{18}H_{32}O_7$ – Sigma-Aldrich) were used. For hemp-lime formulation, Natural Hydraulic Lime (SECIL NHL-5) was used.

2.1. Formulation and casting

Hemp-silica composites were made by mixing hemp shivs with TBC for 1–2 min and then adding sodium silicate until a uniform and workable paste was obtained. The mixes were prepared based on the proportions shown in Table 1 and mixed manually for 5–6 min. The mix was cast in two stages by gently tamping down (30–40 times) the paste in polystyrene moulds. A shiv-liquid sodium silicate mass ratio of 1:3.75 was used throughout this study for hemp-silica composites. An initial experiment to determine the optimal TBC content in the hemp-silica matrix was carried out. TBC was added at 12.5 wt%, 25 wt% and 37.5 wt% of sodium silicate dry matter to hemp-silica (1:3.75) mixes and the compressive strength at 7 days of drying was measured. The mix with 37.5 % TBC produced the highest compressive strengths as shown in Fig. 2. Therefore, this formulation was used for further thermal conductivity, moisture buffering capacity, water resistance and microstructural analysis. Hemp-lime composites with a shiv-NHL5-water mass ratio of 1:2:3 was prepared to replicate the hemp-silica 37.5 % TBC samples dry matter content and allow the comparison of the two systems. Water was added to hemp shiv and NHL5 dry mix and the paste was manually mixed for 5–6 min to achieve suitable workability. Hemp-silica and hemp-lime samples were de-moulded after 1 and 3 days respectively and placed in a cabinet dryer at 50 °C for drying.

2.2. Mechanical, thermal and hygric properties

The effect of drying time on compressive strength was evaluated. Compressive strength of 100 mm cubes at 3, 7, 14 and 28 days of drying was measured in triplicate using a 250 kN Wykeham Farrance load frame at a rate of 2 mm/min with loading perpendicular to the tamping

Fig. 1. Hemp shivs used in this study.

Table 1
Composition of hemp-silica and hemp-lime mixes.

	Hemp shiv (kg/m^3)	Liquid Sodium Silicate (kg/m^3)	TBC[a] (kg/m^3)	NHL-5[b] (kg/m^3)	Water (kg/m^3)	Fresh density (kg/m^3)	Hemp to binder (dry matter) ratio	Binder (dry matter) (kg/m^3)
Hemp-Silica	150	562.5	0.0	-	-	712.5	0.41	368.3
	150	562.5	27.3	-	-	739.8	0.38	395.5
	150	562.5	54.6	-	-	767.1	0.35	422.8
	150	562.5	81.8	-	-	794.3	0.33	450.1
Hemp-Lime	150	-	-	300	450	900.0	0.33	450.0

[a] Tributyl citrate.
[b] Natural hydraulic lime.

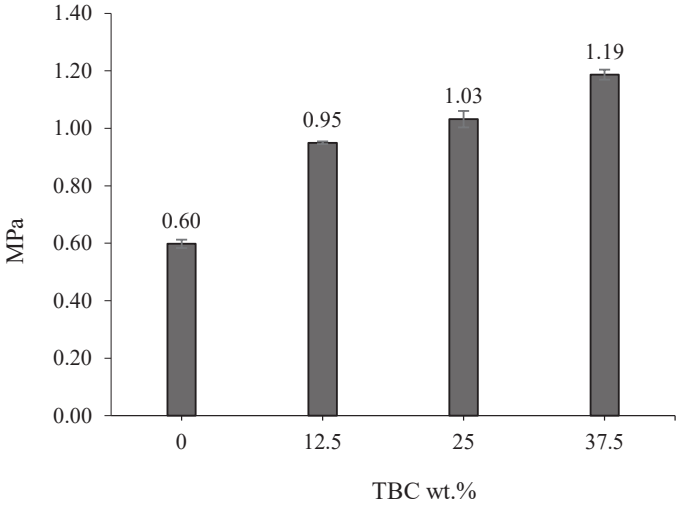

Fig. 2. The effect of tributyl citrate TBC wt% of sodium silicate dry matter on compressive strength of hemp-silica composites.

direction. The max load at sample failure or 30 % displacement was recorded whichever came first.

Panels of 20×5×5 cm were prepared and tested for thermal conductivity after 28 days of drying. The thermal conductivity λ (W/mK) of the samples was measured using a heat flow meter (Netzsch HFM 446 Lambda Small) at user-defined temperature difference between plates at 0 °C and 20 °C. An expanded cork with a declared thermal conductivity of 0.04 W/mK was tested for comparison. Thermal conductivity in W/mK was measured using the formula:

$$\lambda = (Q.d)/(A.\Delta T) \quad (1)$$

Q: energy transferred in W
d: thickness of the panel in m
A: area of the panel in m^2
ΔT: temperature difference between two chambers at steady state.

Moisture buffering value (MBV) as a measure of the materials ability to regulate the changes in the surrounding relative humidity (RH) was determined according to the NORDTEST method [26]. The samples were subjected to cyclic RH conditions at 20 °C with 8 hours of 75 % RH followed intermittently by 16 hours of 35 % RH. Samples of 10×10×3 cm after 28 days of drying were sealed on all but one surface and placed on a scale inside a dynamic test chamber (Binder MKF 56) using a step program to alternate between RH levels. The air velocity on the samples surface was kept in the range of 0.9–1.0 m/s using the chamber fan speed control. The air velocity was measured using an anemometer (Testo 416 Vane). The test was conducted in duplicate with for at least five cycles. The MBV (g/m^{2}RH) was calculated using the formula:

$$MBV = \Delta m/(A.(RH_{high} - RH_{low})) \quad (2)$$

Δm: mass change due to moisture uptake/release (g)
A: open surface area (m^2)
RH: relative humidity level high/low (%)

Sodium silicate is highly soluble in water. The moisture resistance of hemp-silica composites with and without the addition of TBC was evaluated according to the British Standard method (BS 321:2002) [27] and the results were compared with hemp-lime. In this test, 100 mm cubes were subjected to cycles comprised of 3 stages: immersion in water, freezing and drying according to the conditions given in Table 2 and the changes in compressive strength were reported after 3 cycles. The hemp-silica and hemp-lime cubes had undergone drying/curing for 28 days prior to the test.

Surface morphology and microstructure of hemp-silica and hemp-lime composites were analysed using scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) (JEOL JSM-6460 equipped with an Oxford Instruments Xplore EDS detector with 15 mm^2 sensor size). The specimens were mounted onto double-sided carbon tape on an SEM stub and then coated with a conductive layer of carbon prior to the analysis.

3. Results

3.1. Compressive strength

Fig. 3a and b show the effect of drying time on the compressive strength and density of hemp-silica and hemp-lime composites. Hemp-silica developed 0.56 MPa compressive strength just after 3 days. This increased to 1.37 MPa at 7 days and eventually to 1.92 MPa after 28 days. It was not possible to de-mould the hemp-lime sample after 24 hours due to insufficient setting. The hemp-lime samples were demoulded after 3 days and tested for compressive strength after 7 days from casting. The compressive strength of hemp-lime samples was 0.4 MPa at 7 days and reached 1.53 MPa at 28 days. The water loss in the hemp-silicate specimens occurred rapidly as the density of the sample reduced from 0.79 g/cm^3 to 0.48 g/cm^3 in the first 3 days of drying. The density of hemp-silica composites remained between 0.48 g/cm^3 and 0.44 g/cm^3 up to 28 days of drying. The hemp-lime showed a higher density of 0.61 g/cm^3 at 7 days and 0.49 g/cm^3 at 14

Table 2
Cyclic moisture resistance test conditions adapted from BS 321:2002.

Stage		Temp °C	Duration (h)
1	Immersion in water (minimum depth of 25 ±5 mm) with pH 7±1.	20 (± 1)	70 (± 1)
2	Freezing	-12 - −25	24 (± 1)
3	Drying at 105 °C	70 (± 2)	70 (± 1)

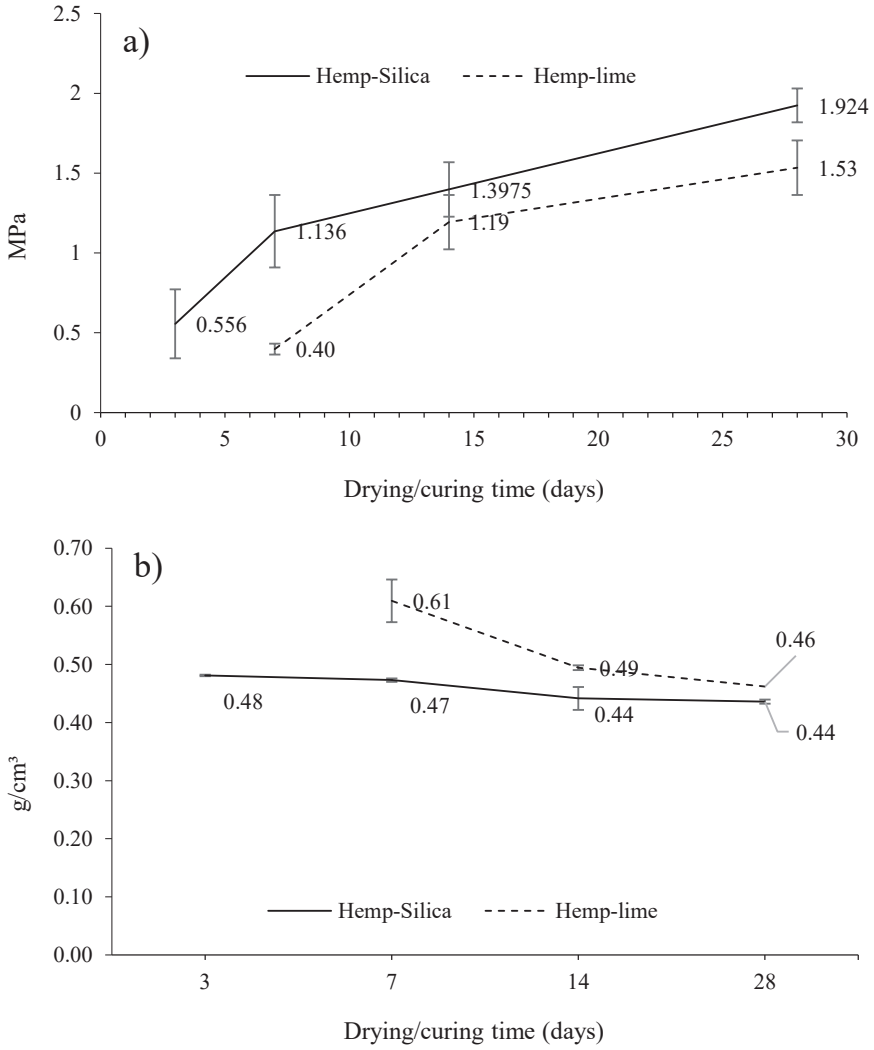

Fig. 3. Effect of drying time on the compressive strength (a) and density (b) of hemp-silica and hemp-lime composites.

days due to the higher moisture content present in the material at an earlier drying duration. Hemp-lime reached a density of 0.46 g/cm^3 after 28 days.

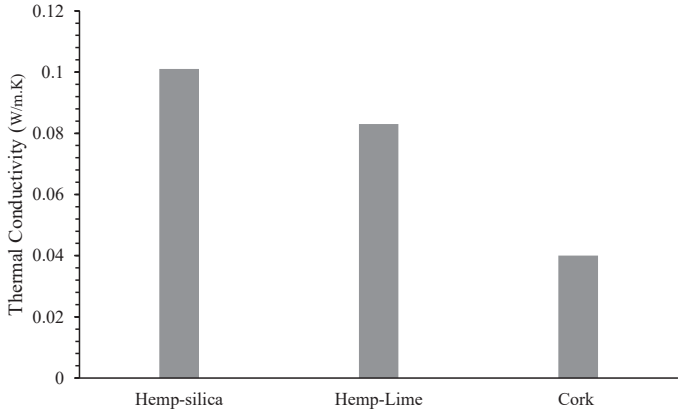

Fig. 4. The thermal conductivity of hemp-silica, hemp-lime and cork panels measured using a Heat Flow Meter.

3.2. Thermal conductivity

Fig. 4 shows the HFM thermal conductivity values measured for hemp-silica and hemp-lime panels at 0.101 W/mK and 0.083 W/mK, respectively. The thermal conductivity of cork was measured at 0.04 W/mK which was similar to the supplier declared value.

3.3. Moisture buffering value

Fig. 5a–c show the changes in the mass of hemp-silica, hemp-lime and cork against the fluctuations in relative humidity. The average MBV for hemp-silica samples was measured at 3.49 g/m^{2}RH which was higher than that for hemp-lime samples at 3.11 g/m^{2}RH. MBVs in the range of 0.5 – 1.0 g/m^{2}RH is considered Moderate while MBV at 1.0–2.0 g/m^{2}RH and above 2.0 g/m^{2}RH are considered Good and Excellent, respectively. Cork samples showed a lower MBV at 0.99 g/m^{2}RH compared to hemp-silica and hemp-lime bio-composites.

3.4. Cyclic moisture resistance

Fig. 6 shows the compressive strength of hemp-silica and hemp-lime composites after being subjected to 3 cycles of immersion in water, freezing and drying according to the conditions expressed in the British Standard (BS 321:2002) [27]. The compressive strength of hemp-silica dropped from 1.92 MPa to 1.13 MPa (41 % decrease) after the third

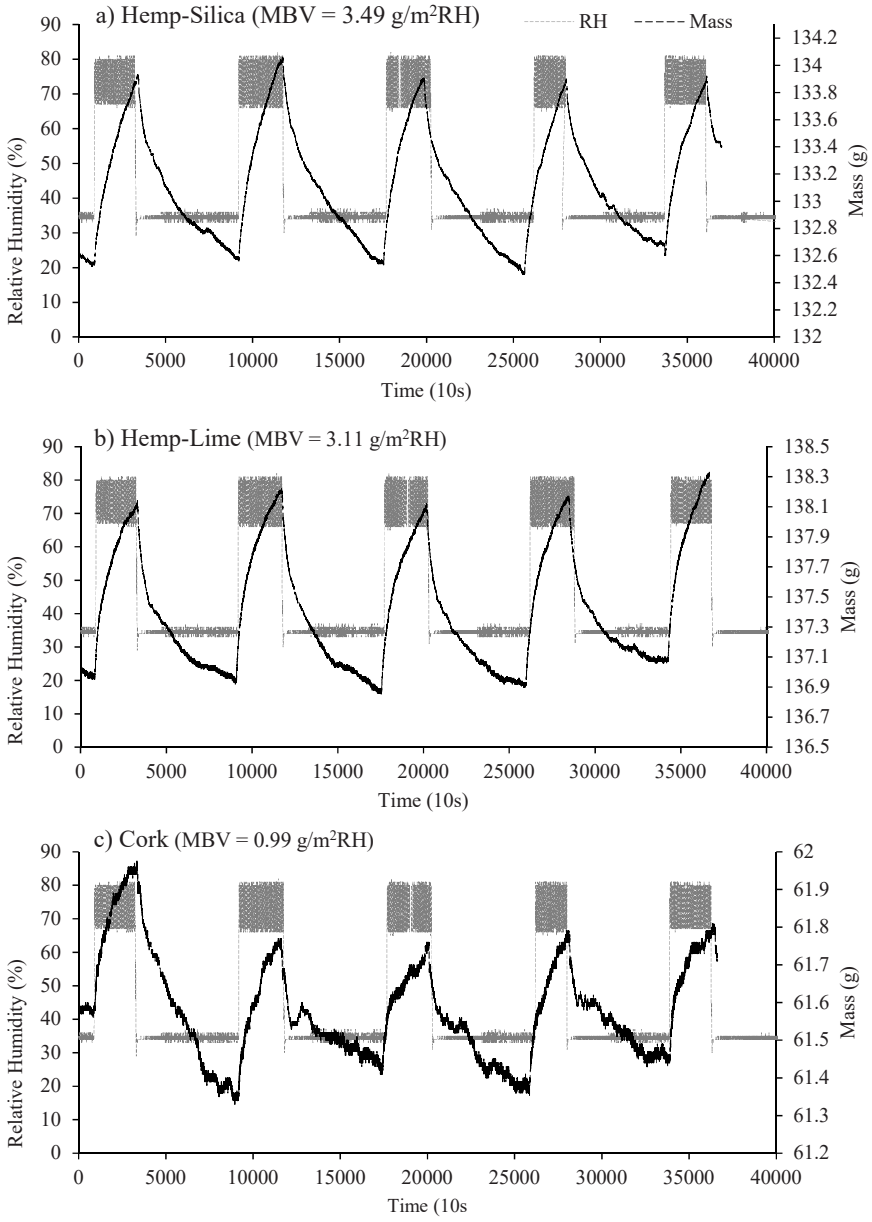

Fig. 5. Moisture buffering values and the changes in the mass of hemp-silica and hemp-lime composites against the fluctuations in relative humidity.

cycle. Samples of hemp-silica prepared without the addition of TBC were disintegrated during the immersion stage of the first cycle showing the effectiveness of TBC in increasing the water resistance of the material. The compressive strength of hemp-lime samples showed a 54 % decrease reaching 0.7 MPa from 1.53 MPa after the third cycle.

4. Discussion

Hemp-silica composites exhibited a higher compressive strength compared to hemp-lime over 28 days of drying time. It took only 3 days for hemp-silica to reach 40 % and 7 days to reach 60 % of strength at 28 days. This is because the setting of the silica binder only requires the removal of water which occurs either by evaporation or by sorption through the porous structure of the hemp. Another reason for the fast-drying capability of hemp-silica is the low water addition required to reach a workable mix. For the same density range and hemp-to-binder ratio, the hemp-silica system produced a workable mix with 25 % less water content compared to hemp-lime. The improved workability is due to the plasticising effect of TBC. The initial mixing of shivs with TBC inhibits the water ingress to the porosity and makes the mix more workable at a lower water content. On the contrary, the hemp-lime curing process requires a longer time due to the lower diffusivity of the porous paths into the hardened material [28]. In addition, as the drying continues, the lack of liquid water to sustain the pozzolanic reaction of hydraulic lime results in the formation of a powdery (un-set) core and an overall lower compressive strength.

Hemp-silica composites had a similar 28-day density compared to hemp-lime. However, NHL-5 undergoes carbonation increasing the material density which can affect mechanical, thermal and hygric properties. SEM micrographs of Fig. 7 showed a higher thickness of the lime binder filling up the gaps between the shiv particles with a powdery phase while in hemp-silica, the binder was mainly deposited on hemp as a thin film providing a strong interfacial adhesion between the particles. This effect resulted in hemp-silica not reaching a failure on more than 30 % of displacement but hemp-lime failed at much lower displacement levels.

Hemp-silica systems exhibited an excellent MBV due to the microstructures developed by the shiv-binder interactions. Fig. 7**c** shows an

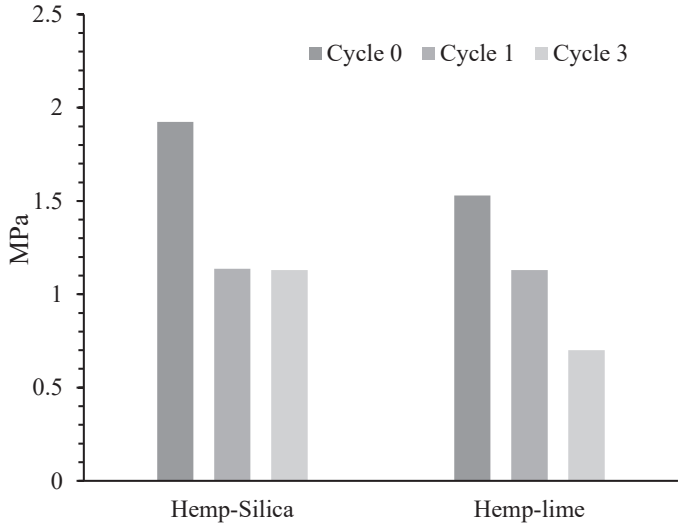

Fig. 6. Compressive strength of hemp-silica and hemp-lime composites after being subjected to 3 cycles of immersion in water, freezing and drying according to the conditions expressed in the British Standard (BS 321:2002).

SEM micrograph of the outer and internal structure of a fractured hemp-silica particle. The formation of a non-porous silica layer on the outer surface of hemp shivs can be observed in the EDS elemental map of Fig. 7d. However, the internal pore structure of the hemp-silica particle remained intact as the dark regions in the EDS map show the absence of silica. Hemp-lime also exhibited a high MBV (although slightly lower than hemp-silica) due to the porous microstructure of lime binder allowing an efficient moisture access to shivs internal pores as seen in SEM micrographs of Fig. 7**e** and **f**. The results of the cyclic moisture resistance test revealed the effectiveness of TBC in stabilising the sodium in the dried binder and the formation of a water-proof end-product.

In addition to the superior mechanical/thermal and water resistance properties of hemp-silica composites, the binder system can provide further environmental benefits compared to the conventional lime binder systems. Liquid sodium silicate has a lower embodied carbon (0.424 $kgCO_2/kg$, [29]) compared to hydraulic lime (0.89 $kgCO_2/kg$ – EcoInvent 3.9.1). Sodium hydroxide is susceptible to carbonation following the reaction:

$$2NaOH{\cdot}SiO_2 + CO_2 \rightarrow Na_2CO_3 + 2SiO_2\downarrow + H_2O \quad (3)$$

In addition, a significant energy saving can be provided by the low drying time of hemp-silica (days) compared to hemp-lime (months) systems. The use of TBC as a nature-based plasticiser to improve the

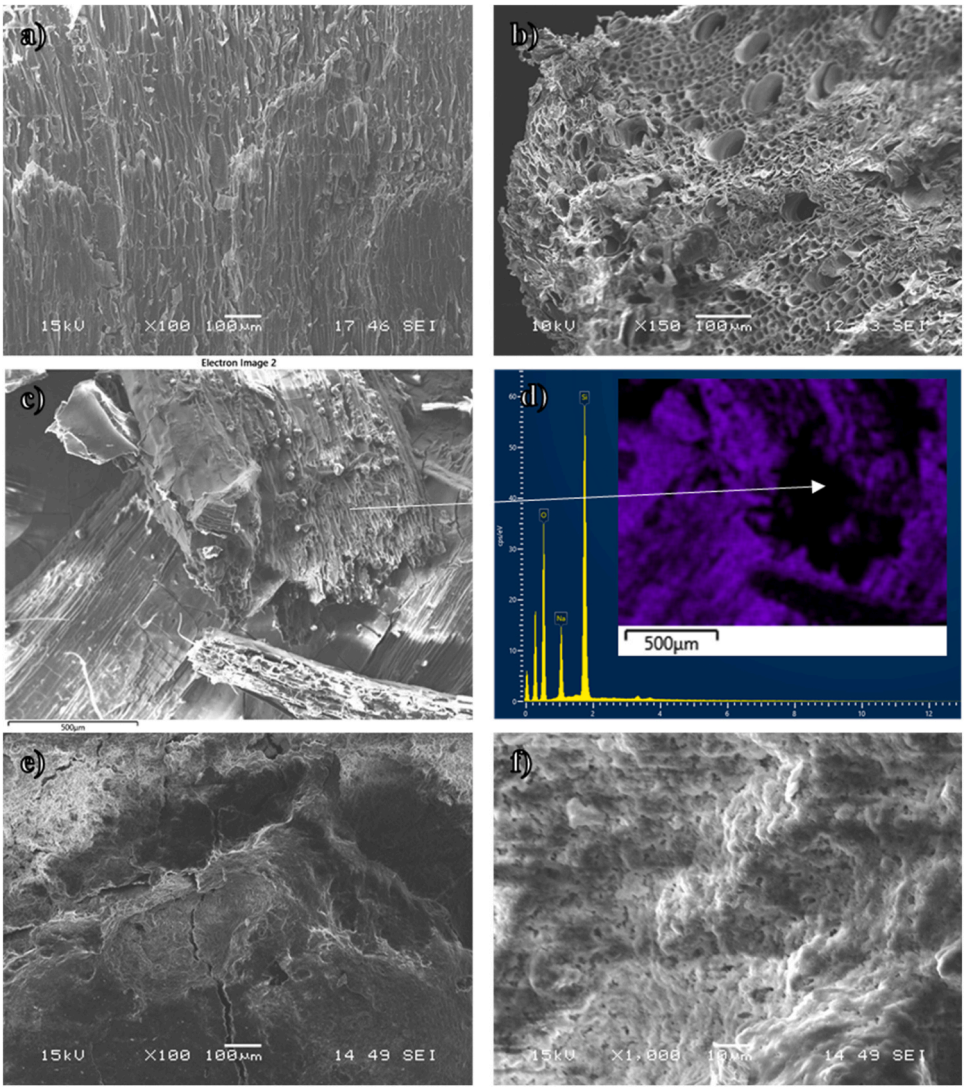

Fig. 7. SEM micrographs of hemp shive surface (a) and internal porosity (b), shivs surface and internal pores (c) with the corresponding EDS elemental map for Si-Kα (d) in hemp-silica composites and shivs surface (e) and internal pores (f) in hemp-lime composite.

mechanical properties of sodium silicate binder showed promise but the investigation of other esters can be considered by future research. The investigation of the binder system compatibility with other lignocellulose-based bio-aggregates also presents an interesting future research direction. The formulation can be further optimised to obtain better mechanical, thermal and hygric properties.

5. Conclusions

This research has demonstrated the potential for silica-based binders as a low-carbon fast-drying alternative for hemp concrete products. Hemp-silica composites with shiv – liquid sodium silicate mass ratio of 1:3.75 and TBC content of 37.5 wt% of sodium silicate dry matter exhibited superior compressive strength, hygrothermal and water resistance compared to hemp-lime concrete. The microstructural analysis of the hemp-silica composites revealed the formation of a silica-based layer on the shivs surface with strong interfacial adhesion and large air gaps between the particles to be responsible for the material enhanced mechanical and hygric and thermal properties. The binder system has potential for other commonly used plant aggregates but further formulation optimisation and the use of other nature-based esters in conjunction with sodium silicate to obtain better properties are recommended.

CRediT authorship contribution statement

Bamdad Ayati: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Armor Gutierrez:** Writing – review & editing, Validation, Methodology, Conceptualization. **Alan Chandler:** Writing – review & editing, Resources, Methodology, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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