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Microstructure and mechanical performance of modified hemp fibre and shiv mortars: Discovering the optimal formulation

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ABSTRACT

In this work, we investigate the effect of hemp fibre and shive addition on modified mortars microstructure and mechanical performance. The mortar formulation is adjusted with different percentages and lengths of either hemp fibres or shives. Workability of fresh modified mortars is carried out using two main techniques including maniabilimeter and flow table experiments. Microstructural effects are revealed using X-ray μ-tomography where the pore content and 3D spatial arrangement and content of hemp are all investigated. Mechanical performance is derived from bending and compression testing at different curing times. Results show the potential of using natural fibres as substitutes leading, in this study, to improvement of mechanical strength but deterioration of stiffness. Superior mechanical performance with compression full strengths as large as 66 MPa is achieved using high-energy ball milling process of fly ashes that are added to unmodified mortar. Results show also that optimal formulations need to involve both hemp fillers and milled fly ashes. These formulations reveal superior mechanical performance compared to all tested conditions after only 7 curing days.

1. Introduction

A huge body of research effort is now dedicated to the use of natural fibres in civil engineering applications [1–4]. Besides of being abundant renewable resource, natural fibres have a positive environmental footprint. Environmental impact starts to be a concern in building sector pushing towards practical changes in building design [5], materials selection [6] and methods of cost evaluation [7]. Indeed, significant CO_2 emission comes from the building environment as reported in several research contributions [8–11]. The replacement of part of the cementitious matrix by natural fibres can be viewed as an effort towards a more positive ecological footprint despite negative binding capabilities of fibres. The use of natural fibres as a reinforcement or substitute is a challenging question. Natural fibres are neither regular in shape nor in dimensions. They exhibit lower mechanical performance with respect to synthetic fibres (Table 1) due to the presence of surface flaws and hierarchical internal microstructure [12]. Sensitivity to water can be a limiting factor since it requires a long setting time and induces complex drying kinetics [13,14]. The most critical question may also be the processability of cementitious mixtures with natural fibres, which enforces to review many standards in building sector. Besides, odours and discomfort can be some secondary issues which have to be part of the building specifications.

Despite all mentioned disadvantages regarding the use of natural fibres as building materials, the literature reports several successful attempts [1]. From the mechanical viewpoint, the major argument supporting the use of natural fibres is the low material density, which allows the achievement of high levels of specific properties [15–18]. As shown in Table 1, the specific Young's modulus of natural fibres, more particularly hemp, competes with those of glass and steel fibres [19–22]. In various studies related to civil engineering applications, the density of the modified cementitious material is provided as a result of weight modification due to the filler addition [23,24]. Few studies focus on positive carbon footprint as an objective and this explains why the substitution of cement by natural fibres is not correctly addressed.

Hemp or *Cannabis sativa* as an effective plant substitute of cement is a fast growing annual plant where the stem is constituted by fibre bundle (hemp fibres), shives, epidermis and hollow space. Hemp stem can reach a height of more than 4 m when the plant has good growth conditions [25]. A cross section of the hemp stem shows different layers. The outside of the stem is covered with bark, also called epidermis [25]. Inside the hemp stems are fibre bundles and the woody core, called shives. The fibres are bound by the middle lamella and arranged in bundles running from the top to the bottom of the stem [26]. The hemp fibres contain 73–77% w/w cellulose, 7–9% w/w hemicellulose

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Table 1
Mechanical properties of natural fibres compared to conventional reinforcement available from the literature [2,19,20,52,53].

Fibre	Density (g/cm^3)	Elongation at break (%)	Tensile strength (MPa)	Young's modulus (GPa)	Specific Young's modulus ($GPa \times cm^3/g$)
Hemp	1.14–1.51	1.6	500–900	30–80	20–57
Flax	1.20–1.50	2.7–3.2	345–1035	28–90	19–75
Sisal	1.45–1.50	2.0–2.5	67–635	4–22	3–15
E-Glass	2.50–2.60	2.5	2000–3500	70–80	27–32
Carbon	1.40–1.78	1.4–1.8	4000–5000	230–240	129–171
Steel	7.75–8.05	0.5–3.5	500–2000	200–210	25–27

and 4–6% w/w lignin, whereas the hemp shives contain 48% w/w cellulose, 21–25% w/w hemicellulose and 17–19% w/w lignin [25].

Most of the literature work related to hemp fibre reinforcement of cementitious composites highlights compromise decisions in processing that have to be taken to deal with disadvantages of using such natural fibre. Arnaud and Gourlay [14] establish a window parameter for the curing conditions (relative humidity conditions) of shiv modified concretes. The authors highlight that performance of hemp modified mortars is sensitive to both content and length of shiv addition. A more complete study needs to handle both parameters to design optimal modified mortars. Colinart and co-workers [27] show that the spraying setting process is more appropriate to achieve a faster drying of the hemp shiv modified concrete. The same authors conclude that kinetic of moisture diffusion are decoupled from the overall weight loss during curing. An attempt to lower the effect of water absorption using projection setting process is illustrated in the work of Elfordy and co-workers [13]. The authors demonstrate the possibility of achieving better mechanical performance with a large density of hemp in modified concrete.

All these studies show that modulation of the hemp content has to take into account the balance between searched properties (thermal insulation versus structural integrity). Fibre content and dimensions are by far the most important tuning parameters to modulate cementitious composite properties [28,29]. Even with these levers in hand, superior mechanical performance is not yet obtained without considering chemical modification of the fibre itself that exposes more cellulose properties. Besides the fact that such treatment is costly, it pulls back the involved green process.

Some of the recent contributions suggest the use of additives to ensure better durability and optimum mechanical performance of hemp modified cementitious composites [28,30]. This approach proves to be effective for pure cementitious materials [31].

In the same line of thought, this paper investigates the mechanical performance of a modified mortar using either hemp fibre or shiv with pozzolan binder. The process variables include varied concentrations and lengths of hemp fibre or shiv combined with a fixed content of milled fly ashes as cement substitutes. The use of coal fly ash in concrete technology has gained attention because of its positive role on improving mechanical performance and effective cost reduction in civil engineering applications. In the present study, all modified mortars including formulations with milled fly ashes are compared. Compressive and flexural experiments are performed for this purpose and discussed up to 90 curing days.

2. Experimental layout

The cement used in this study is CEM II/B-M (S-LL) 32.5 R from Ciment Calcia. It contains more than 62% of clinker, slag (>22%) and other mineral additions according to EN 197-1 norm. The cementitious matrix is also composed of standardized sand, which is a dry siliceous natural sand calibrated to 0/2 mm and conforming to the norm EN 196-1. Among the other additives to the cementitious matrix, fly ashes are byproducts from coal combustion, which are widely used as a cementitious and pozzolanic ingredient in hydraulic cement concrete. Fly ash is provided by Effage Company with chemical composition given in Table 2.

Hemp fibres and shives are provided without any chemical treatment by INRA UMR FARE of Reims, France. Regular hemp fibres and shives are obtained by sectioning the raw materials into lengths of 1 mm, 10 mm and 20 mm (Fig. 1). The primary aspect ratio of the fibre (ratio between length and largest transverse dimension) varies significantly because of the variability in lateral dimensions of bundles. With regard to the considered lengths, this aspect ratio evolves between 1 and 1000. In addition, the hemp fibres exhibit a transverse aspect ratio (ratio between transverse dimensions) of 3.05 ± 0.67 as shown in [28]. The untreated fibres are composed of bundles with a significant amount of surface defects. Hemp shiv exhibits more regular shape with a smoother surface topography and a lower density of surface defects. The associated primary aspect ratio dispersion (from 1 to 10) is smaller but the secondary aspect ratio is at least two times larger than the one for hemp fibre. Different percentages are added to the cement in the range (1–10) % in weight. In all formulations, hemp is substituted to the cement, which implies adjustment of sand and water contents. Maximum filler content is only achieved with the smallest lengths due to lack of processability. Table 3 summarises all possible combinations.

Soaking of hemp fibres and shives is performed based on a former study showing the beneficial effect of using wet hemp on mechanical performance of modified mortar [28]. Soaking is performed at room temperature for a duration ranging between 15 and 20 min (Fig. 2). Fibres are gently spun and excess of water is further removed using absorbent paper. Hemp is added, when needed, to the mixing water taking into account weight ratio rules for sand, water and cement as detailed in [28].

Milled fly ashes are added to the mixture at a fixed content of 15% in weight by substituting cement. The milled powder is obtained using a standard route of high energy ball milling. This process is carried out using a planetary high-energy ball mill (Retsch PM 400). The instrument is composed of four vials mounted on a planar disc. With the rotation of the disc, the vials move in a circular and in opposite direction compared to the disc rotation. The rotation speeds of the disc and the vials are $\Omega = 400$ rpm and $\omega = 800$ rpm, respectively. Thirty millimetre diameter steel balls and 500 ml volume steel vials are used. The weight of powder samples is 200 g per vial. The ball-to-powder weight ratio is 4. Milling of fly ashes during 3 h modifies the colour and structure of the powder as shown in Fig. 3. Structural analysis of milled and unmilled powders are carried out using Bruker D2 phaser diffractometer under a continuous scanning mode with Cu Kα radiation ($\lambda = 0.1541$ nm). The lines are measured in the 2θ range (5–100)° in steps of 0.02° for 10 s. The software used for evaluation is DIFFRAC.EVA with ICDD PDF2. The milled powder exhibits an amorphous structure of magnetite with a reduced crystalline size of Quartz and nanosised Mullite (Fig. 3). The milling process is responsible for the increase of the specific surface of fly ashes from 0.8 m^2/g to 2 m^2/g measured using the Brunauer–Emmett–Teller (BET) adsorption method with the Gemini VII 2390 Micrometrics instrument. Further description of milling conditions and effects on modified fly ashes properties are beyond the space of this study. An in-depth study related to these effects is currently under investigation and will be published elsewhere.

The reference mortar free of addition is prepared according to the European classification under the standard Norm NF EN: 196-1 [32]. Following this standard, cement to sand (C/S) weight ratio is 1:3 and water to cement (W/C) ratio is 1:2. Each batch of three test specimens

Table 2
Chemical composition of fly ash in % wt.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	TiO_2	P_2O_5	SO_3	SrO	BaO
43.7	25.1	13.5	5.86	3.41	2.65	1.96	1.38	1.31	0.28	0.168