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Preliminary study of the mechanical and hygrothermal properties of hemp-magnesium phosphate cements

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HIGHLIGHTS

- A new hemp-magnesium phosphate cement (*heMPC*) was developed and assessed.
- *HeMPC* can incorporate higher hemp amounts with enhanced mechanical properties.
- It can achieve a $\sigma_{max} = 0.714 \pm 0.11$ MPa with a 20 wt.% of hemp content.
- It also presented enhanced thermal properties ($\lambda_D = 0.103\ W \cdot m^{-1} \cdot K^{-1}$).
- The improved performance is attributed to the effective formation of K-struvite.

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ABSTRACT

The use of bio-based materials in the construction field is of great interest to society and the scientific community because of its highly sustainable character. They are formed by plant fibres and a binder, usually cement, lime or pozzolanic additives. Among the new vegetable fibres used, hemp-based biomaterials have attracted great attention in the recent years due to its excellent thermal and hygroscopic properties. However, they present a very low mechanical performance, which has intensified the search for better alternatives. In this research, the use of magnesium phosphate cement (MPC) as binder with different hemp additions (8%, 12%, 16% and 20% by weight) was evaluated. Thus, a new material made of hemp and MPC (*heMPC*) was developed. According to the results obtained, the *heMPC* could be used in floor or pre-cast structural applications since it presented enhanced mechanical ($\sigma_{max} = 0.714 \pm 0.11$ MPa with a 20 wt.% of hemp content) and thermal ($\lambda_D = 0.103\ W \cdot m^{-1} \cdot K^{-1}$, density = 600 $kg \cdot m^{-3}$) properties with respect other lime-based hemp biomaterials. Furthermore, the material exhibited good hygroscopic properties (water absorption by capillarity). Accordingly, this preliminary study allowed opening a new research line in the use of hemp bio-composites, in which other important properties are currently under investigation. The MPC used as a binder in this study was formulated with a by-product from the MgO industry, which increases the sustainability and recyclability criteria of the material developed.

1. Introduction

The environmental concerns of energy utilization in buildings while ensuring indoor comfort has given rise to an intense research over alternative construction materials. The main research criteria have been based in environmental and economic factors while favouring the use of local resources [1]. In this sense, the use of materials with plant fibres – bio-based materials or biocomposites – is very attractive because its renewable character allows reducing energy and raw materials [2]. Moreover, their environmental impact is lower than traditional building materials because relatively large amounts of atmospheric CO_2 can be sequestrated through photosynthesis [3–5]. Among the new vegetable fibres used, hemp stands out from the rest because of its wide availability, low requirements of fertilizer and irrigation, permanent renewal character, good humidity control and very favourable energy and ecological balances [1,6]. It is hardly attacked by insects and moths due to its lack of proteins [7]. The seeds, fibres and woody core (shives) can be profitable in many manners. The woody core or hemp shives make up 40–60% of the mass of the hemp stalk and can served as animal bedding or more recently

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as aggregate for bio-composites [5,8]. A hemp bio-composite is commonly formed by the shives and a mineral binder. The initial studies considered Portland cement as the binder although other compounds such as lime and pozzolanic admixtures were later under consideration in order to improve the performance. Thus, a hemp concrete (*hempcrete* or HC) or hemp-lime concrete (HLC) can be obtained. Both materials present an excellent hygrothermal behaviour (especially with respect concrete) and depending on their composition they can be used as insulators in walls, floor and roof [9]. According to Collet and Pretot (2014), HC/HLC induce a reduction ranging from 15% to 45% in energy consumption, depending on the ventilation strategy used [10]. Despite of its great hygrothermal performance, HC/HLC presents low mechanical properties that restrict its use for structural purposes, being often associated with a rigid frame [11]. These low compressive strength values have fostered the search for alternative binders, which can provide an enhanced mechanical performance while complying with sustainability and recyclability aspects.

In this regard, magnesium phosphate cements (MPC) are a type of the commonly known Chemically Bonded Ceramics (CBCs) that possess certain characteristics of cement and thus can be considered as such. In general, CBCs are obtained from an acid–base chemical reaction into an aqueous phase between a metal cation and an oxyanion source. When phosphates are used as the oxyanion source, the CBC becomes chemically bonded phosphate ceramic (CBPC), which are commonly used as stabilizing or encapsulation agents for hazardous and radioactive wastes [12–16]. Due to their very fast setting time and good mechanical properties, they can be also used as effective repairing materials. When MgO and monopotassium phosphate (KH_2PO_4) are used as the metal oxide and phosphate sources respectively, a crystalline structure – $KMgPO_4{\cdot}6H_2O$-named K-struvite is formed. This compound is known as *ceramicrete* and belongs to the category of MPC [17,18]. In order to reduce the cost of the Mg source, a by-product from the calcination of natural magnesite (low grade MgO, LG-MgO) can be used instead of pure or high grade magnesium oxide. Furthermore, since the by-product has been partially calcined (~1200 °C), it also offers the extra advantage of presenting a more suitable reactivity for formulating MPC. In this sense, the authors have a long experience on the development of MPC using LG-MgO and its sustainable and environmentally friendly application [12,19–22]. Taking into account all the above mentioned and in order to increase the mechanical performance and sustainability criteria of *hempcrete*, the aim of this study was to develop a new bio-composite and eco-material composed of hemp shives and MPC formulated with LG-MgO: *heMPC*. The novelty of the present investigation led to start from preliminary trials until identifying an adequate range of study and subsequently evaluate the most important mechanical, thermal and hygroscopic properties.

2. Materials and methods

2.1. Raw materials and heMPC formulations

About 50 kg of Mg by-product was supplied by Magnesitas Navarras S.A. located in Navarra (Spain). This by-product is collected during the calcination process of natural magnesite and it can be generally described as follows. Once the magnesite ore is extracted from the natural deposits and benefited through classifiers and collectors, it is fed into two rotary kilns at 1200 and 1600 °C for calcination. The gases generated from these two kilns are taken to an air pollution control system where the fly particles are collected in fabric filters. This dust material is classified as low-grade MgO (LG-MgO) and is considered the bulkiest by-product obtained in the production train process. It contains a 55–60 wt.% of MgO and is about 10 times cheaper than pure MgO [12].

The hemp shives were obtained in Toulouse (France) and the monopotassium phosphate (MKP 0-52-34 food grade) was acquired from Rotem Amfert Neger Ltd. (ICL Fertilizers) in Barcelona (Spain). Tap water was used for all formulations.

In order to define a range of study in what respect to the manufacturing process, several preliminary tests considered varying different parameters for determining the quantity of material needed per batch and the best mixing method. Other important aspects that were also considered were the: (i) state of the hemp shives; (ii) pouring of the material mixture in the moulds; (iii) method for mixing hemp and MPC and (iv) workability of the resulting paste. The authors want to emphasize that the setting time of MPC is considerably much shorter than the widely used lime. Therefore, the results from this preliminary stage allowed defining a range of study in what respect to dosages. Thus, the different formulations used in this study were cast based on variations in the percentage of hemp with respect the total weight of solids (8%, 12%, 16% and 20%). The composition (weight basis) of the binder (B) was fixed in 60% of LG-MgO and 40% of MKP for all experimental trials, as reported as the optimum formulation by Formosa et al. (2012) [21]. Due to the water (W) demands from the B and the hemp shives (H), two different W amounts (kg) were distinguished: the water needed to mix the binder (W_B) and the additional to mix the hemp (W_A). According to the preliminary trials, W_B/B and W_A/H were fixed in 0.26 and 0.8 respectively. The B was prepared and mixed in the first place following the next procedure:

I. The solids (LG-MgO and MKP) were weighted separately and mixed.
II. After adding WB to the solids, the paste was mixed with an automatic mixer during 1 min at low speed (140 ± 5 rpm) and 1 min at high speed (285 ± 10 rpm). Immediately after,
III. The H was added in five parts. WA was added in five parts as well, each one after the H additions. After each step of H and WA addition, the mixture was mixed during 30 s at high speed. After the final step, the paste was mixed again for 1 min at high speed. Steps II and III could never exceed 8 min.
IV. After mixing, the material used (bowl, stirrer and spatula) was cleansed with tap water. This process could not exceed 15 s.
V. The final paste was poured in the corresponding moulds in 2 steps, with the first one filling half the volume of each mould. After each pouring step, each specimen was vibrated two times, 5 s each. At the end, the surplus of material was eliminated with a spatula.

Finally, the specimens were kept in a climatic chamber (20 ± 2 °C and a relative humidity of 50–55%) for 24 h, then demoulded and stored in the same curing conditions for 1, 7 and 28 days.

2.2. Evaluation of heMPC

Many researches on HC/HLC have focused in evaluating the hygroscopic behaviour as well as the thermal and mechanical performance by means of testing several parameters [7,5,23–27]. These properties are closely related and each one exerts a synergistic effect over the other to a different extent. According to the formulations based in varying the H content (8, 12, 16 and 20 wt.%) and the time of curing (1, 7 and 28 days), different specimens were prepared for evaluating the mechanical, thermal and hygroscopic performance by triplicate using $40 \times 40 \times 160$ mm^3 polystyrene moulds for mechanical and hygroscopic testing and $50 \times 150 \times 150$ mm^3 PVC moulds for determining thermal properties. The parameters measured were aimed at assessing the following properties.

2.2.1. Mechanical performance

The most important parameters characterizing the mechanical performance of construction materials can be obtained from the compressive strength (σ) vs. strain (ε) graph [4]: compression resistance (σ_{max}), the maximum stress supported by the material prior rupture; Young's modulus (E), defined as the material stiffness and obtained as the slope of the linear section in the elastic field; and strain to maximum stress ($\varepsilon_{\sigma max}$). However, bio-composites usually present an atypical mechanical behaviour that requires adapting the standard tests [23]. These issues have been accurately described by Cerezo (2005), Nguyen (2010) and Dinh (2014) [4,28,29] and they can be described as follows. During compaction of the H + B mixtures, a stratification and particle orientation in the radial direction can be promoted, which may result in a structure with a linear elastic orthotropic behaviour in the first phase of the mechanical tests [4]. Moreover, it should be noted that an implementation phase, where the supports of the testing machine adjust to the rather irregular surface of the specimens, can exist. These events required a special treatment of the data. Thus, the displacement of ε at a non-existent σ was corrected by eliminating the corresponding section. As for the orthotropic behaviour, two deformation references were considered: 1.5% and 7.5% [28]. Therefore, the analysis of the mechanical behaviour of the *heMPC* material formulated in this study was evaluated by means of σ_{max}, $\sigma_{1.5\%}$, $\sigma_{7.5\%}$, $\varepsilon_{\sigma max}$ and E using a 100 kN capacity hydraulic press (universal testing machine).

2.2.2. Thermal performance

The insulation performance of a material is usually evaluated by means of the thermal conductivity (λ) and diffusivity (α). The value of λ is an intrinsic characteristic that depends on the material structure. Thermal conductivity was measured in the steady state by the guarded hot plate method, with a λ-Metre EP 500. Measurements were performed according to standard NF EN 12664 [30]. The accuracy of the device was of the order of 5%. The samples used had a volume of