



Thermo-mechanical behaviour of hemp fibers-reinforced gypsum plasters

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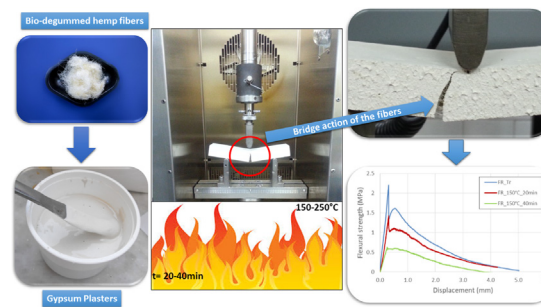
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HIGHLIGHTS

- Biologically treated hemp fibers were used as plasterboard's reinforcement.
- Bio-treated fibers modified the post-cracking behavior improving plaster toughness.
- The toughening role of the hemp fibers is still evident at elevated temperatures.
- Hemp fibers are effective for producing toughened plasterboards for fire-protection.

GRAPHICAL ABSTRACT



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ABSTRACT

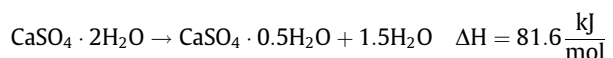
Due to the large latent heat related to dehydration into hemihydrate and anhydrite, gypsum is largely used to make firebreak systems. During a fire exposure, the dehydration can induce cracks inside the panels, since the loss of bound-water molecules leads to a large thermal shrinkage. In this study, hemp fibers were added into the plaster, acting as thermo-mechanical reinforcement of the gypsum panels and hindering the cracks formation. The effect of hemp fibers on the thermal behavior of gypsum panels was studied in terms of thermo-mechanical properties in isothermal conditions. Change in chemical composition and morphology of the gypsum matrix after the thermomechanical test were analysed by thermogravimetry (TGA), X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). Hemp fibers, even at high temperatures, preserved their “bridge action” between the fracture surfaces, increasing the tenacity of plaster and preventing its fragile collapse.

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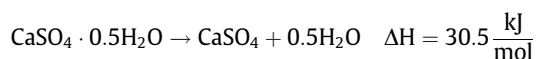
1. Introduction

Gypsum plaster is extensively used to make panels and coatings to form ducts or partitions in firebreak systems [1,2]. The fire resistance of gypsum products, consisting of calcium sulphate in the form of dihydrate crystals $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, derives directly from its chemical structure. In fact, heating gypsum provides two endothermic dehydration reactions, which generally happen in

the temperature range 120–170 °C. During the dehydration reaction, dihydrate gypsum is converted to hemi-hydrate gypsum:



and then finally to anhydrous gypsum,



The above endothermic reactions origin the loss of the bound water molecules and as consequence an isothermal stage, which slow down the rise of temperatures [3]. Consequently, the heat

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transfer through gypsum plasterboards is practically hindered until the dehydration process is complete; the elevated fire resistance characteristic of gypsum plasterboards is mainly due to this effect. Nevertheless, when all the dihydrate and hemi-hydrate gypsum have been converted to anhydrous gypsum, the board loses the capability to provide thermal protection. In fact, after this isothermal stage, gypsum starts to decompose, and cracks appear. The removal of chemically bound water results in shrinkage and a loss of strength of gypsum [4]. Moreover, further stress can develop between heating surface and unexposed surface [5].

In order to reduce the thermal shrinkage and optimize the mechanical and physical properties of gypsum products, different solutions have been proposed [6]:

- (1) Fibrous and organic additives (mineral wool, glass fibers, organic fibers, rice husk, cork) [7];
- (2) Silicate fillers (vermiculite, perlite, silica fume, fly ash, clay minerals) [8–10];
- (3) Expanded polystyrene (EPS) or foaming agent additions [11].

Recently, eco-friendly materials are playing an important role in the building materials market [12], most of all the natural fibers used to reinforce several typologies of composites materials, like polymers, geopolymers and hydraulic and lime-based binder products [13–16].

Among the natural fibres used for plaster/gypsum reinforcement, the following can be highlighted: hemp, short cellulose, sisal, palm and straw fibres [17–19]. In particular, hemp fibers showed good performances as green reinforcement leading to composites with higher physical and mechanical properties [20]. In a previous paper, Iucolano et al. (2018) showed the positive effect of biological treated hemp fibers on the bending behavior of gypsum composites. Furthermore, a significant damage reduction was observed.

The present investigation aims at verifying the effect of the hemp fibers in the improvement of the thermal resistance of gypsum composite. The influence of hemp fibers on the thermal behavior of gypsum panels was studied in terms of thermo-mechanical properties in isothermal conditions. Change in chemical composition and morphology of the gypsum matrix after the thermomechanical test were analysed by thermogravimetry (TGA), X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM).

2. Experimental

2.1. Materials

The gypsum used in the present investigation was provided by Gyproc Saint-Gobain and mainly consists in calcium sulphate hemihydrate (see X-Ray characterization). Its chemical and mineralogical composition was assessed by thermal analysis (DTA/TGA) and X-ray diffraction analysis (XRD) and was elsewhere reported [20]. The hemp fibers used as reinforcement, subjected to a biological treatment that enhances their physical and mechanical features, were provided by Fibranova Group (Pisa, Italy). The fibers were elsewhere characterized [20] and hereafter some main features are reported: diameter = 20–40 μm , density = 1.32 g/cm^3 , water absorption = 17%.

As reported in a previous paper [20], the biological treatment led to chemical modification of hemp fibers, increasing their crystalline fraction and lowering the water adsorption. Moreover, the use of biological treated fibers affected the bending behavior, giving rise to a strongly enhanced toughness of the fiber-reinforced composites.

2.2. Methods

2.2.1. Production of hemp fiber reinforced plasters

The hemp fibers were cut to 10 mm by means of an automatic cutter and were added to the binder at 1% in weight respect to binder. The dry mixture was firstly homogenized and then added to the distilled water (water to gypsum ratio = 0.7) and mixed for about 30 s. The mixture was casted into open moulds (160 × 40 ×

20 mm) and compacted with a conventional jolting apparatus (Mates, mod. E130) for about 30 s. After the setting of the plaster, the specimens were demolded, dried for 48 h at $T = 40^\circ\text{C}$ and finally stored for 1 week in a climatic chamber (MSL Humichamber, mod. EC 125) at 20°C and $\text{RH} = 50\%$. In addition, reference specimens with no fibers were also manufactured. On the whole 12 fiber-reinforced specimens (FR) and 12 reference specimens (REF) were manufactured.

2.2.2. Bending test at elevated temperatures

The influence of bio-degummed hemp fibers on mechanical behaviour of gypsum plasters at room temperature was elsewhere reported [20]. The present investigation aims at verify the efficacy of such fibers at higher temperatures, which can occur as a consequence of a fire.

Accordingly, all the specimens were kept for a fixed period (20 and 40 min) at two different temperatures (150°C and 250°C), after that the bending tests were carried out. These temperatures were chosen in order to achieve a significant decomposition of dihydrate calcium sulphate into hemihydrate (theoretically occurring at about 130°C) and of hemihydrate into anhydrous calcium sulphate (theoretically occurring at about 170°C). Results were then compared with ones obtained at room temperature (T_r , about 20°C).

For each temperature and residence time, the bending test was carried out in triplicate to evaluate the repeatability of the experimental data.

To perform the bending test, a Tensometer 2020 apparatus (Alpha Technologies; Force accuracy: 0.18%; Strain accuracy: 0.002 mm) was utilized, equipped with an oven (Fig. 1) which allowed to test the specimens until a maximum temperature of 350°C . The tests were conducted with a 500 N load cell and a displacement rate of 1.0 mm/min. At the end of the test, the flexural strength of the specimen $\sigma_{u,max}$ was calculated using the following Eq. (1):

$$\sigma_{u,max} = 1.5 \times \frac{Fl}{bd^2} \left[\frac{\text{N}}{\text{mm}^2} \right] \quad (1)$$

where F is the maximum load supported by the specimen, l is the span distance, b and d are respectively the width and the thickness of the specimen. The Young modulus was calculated according to Eq. (2):

$$E = \frac{l^3 m}{4bd^3} \left[\frac{\text{N}}{\text{mm}^2} \right] \quad (2)$$

where m is the slope of the linear portion of the stress-strain curve and the other terms are defined as in Eq. (1).

Finally, the resilience of all the samples was evaluated as the area under the stress-strain curves up to the fracture point, while toughness was evaluated as the area under the stress-strain curves up to $\varepsilon = 0.05$.

2.2.3. Chemical and physical characterization of thermally treated specimens

Immediately after the bending test at elevated temperatures, a fragment from the core of the fracture surface was taken and stored in a desiccator, to prevent any further re-hydration of the gypsum. These samples were then subjected to



Fig. 1. The device for the three-point bending test inside the oven.

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