Mechanical and hygrothermal properties of cement mortars including miscanthus fibers

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ABSTRACT. This study investigates the improvement of thermal and hygric performances of cement mortars through the introduction of micronized miscanthus fibers. Micronized fibers were chosen because they are expected to promote fine and homogeneous fiber distribution within the cementitious matrix, hence opening possibilities for the design of 3D printable mortar mixes including vegetal fibers.

An experimental protocol was first developed for preparing five mortar mixes with miscanthus fiber contents up to 7 wt.%. These bio-based mortars were then characterized at the age of 28 days, in terms of mechanical strength (under flexural and compression tests), thermophysical properties (determination of the thermal conductivity/diffusivity and the volumetric Heat Capacity by the Hot-Disk method), and in terms of hygric properties as well (evaluation of the Moisture Buffer Value MBV according to the Nordtest method). The results of this experimental campaign showed that increasing the miscanthus fiber content leads to large improvements in both thermal resistance (up to 87%) and moisture buffer capacity of mortars (with MBV values up to 2.05), suggesting that such bio-based mortar is a good insulating material and has an excellent ability to mitigate external moisture variations. On the other hand, the introduction of vegetal fibers was also found to decrease very significantly the mechanical strength of the modified mortars, making these latter incompatibles with structural applications. Nevertheless, the developed bio-based mortars retain sufficient mechanical properties for handling and are suitable for building insulation.

Keywords Micronized miscanthus fibers; Cement mortar; Thermal and hygric properties; Mechanical strength; Microstructure.

I. INTRODUCTION

World energy consumption is growing rapidly in the construction sector, causing substantial increase in CO_2 emission. This trend is mainly driven by economic development and population growth, and contributes to exhaust natural resource and degrade the environment. Therefore, current public policies promote the development of innovative technologies capable of improving energy efficiency, with specific interest in "green technologies". In this context, the replacement of conventional building materials by bio-based alternatives with low thermal conductivity has emerged as an interesting solution for upgrading thermal performance and reducing carbon footprint of buildings. In addition, these bio-based materials generally exhibit hygroscopic behavior which enable them to absorb/release moisture, and hence, to reduce humidity variations of the indoor air. Therefore, they are not only effective as thermal insulation materials, but also offer benefits in terms of comfort and health for the occupants.

Numerous studies have explored the use of vegetable fibers (hemp, flax, cork, rice husk, date palm fibers, ...) in the formulation of ultra-lightweight concrete intended for the insulation of buildings(Ashour et al., 2010; Chen et al., 2017; Chikhi et al., 2013; Liu et al., 2010; Panesar and Shindman, 2012; Haba et al., 2017; Chennouf et al., 2018). Most authors have reported that the incorporation of vegetal fibers into cement materials leads to significant improvement in the thermal performance (in particular a large reduction of the thermal conductivity) and provides superior moisture absorption capacity. On the other hand, mechanical properties can be severely degraded. These effects are generally correlated to the microstructure of these bio-based composites, which contains a high density of macro and micro-porosities.

Among the available vegetal fibers, miscanthus fibers have gained interest over the last decade. Miscanthus is a perennial plant with a high potential for biomass production and whose exploitation does not compete with human/animal feeding (Dias & Waldmann, 2020; Lewandowski et al., 2018; Lewandowski & Heinz, 2003). Miscanthus can be cultivated over a period of about 15 years, adapts easily to different climates and soil conditions, and has low requirement in terms of fertilization or pesticide inputs. In France, this crop is mainly cultivated in the northern regions of the country, and an effective valorization chain from the farm to the industry is currently being organized. However, the use of miscanthus fibers in building materials is still poorly documented in the literature and further research is needed in this field.

In this context, this study explores the possibility of incorporating micronized miscanthus fibers into a cement mortar and investigates the effect of this incorporation on the properties of the resulting bio-based composite. The fibers are micronized to allow a finer and more homogeneous distribution in the mortar matrix, with a view to developing 3D printable materials including vegetal fibers in a next part of the project. The experimental program involves the preparation of mortar specimens with various dosages of miscanthus fibers in the range 0-7 wt.%. Multiphysics characterization is then carried-out on these specimens to assess the effects of the fiber addition.

II. MATERIALS AND METHODS

A. Materials and sample preparation

The vegetal fibers selected for this study are micronized miscanthus fibers with diameters in range 200-500 μm, and conditioned in the form of a fine powder. This product was supplied by Addiplast Group (Saint-Pal-de-Mons, France) and it was used as-received. Figure 1 provides typical pictures of these fibers at various magnifications, as obtained by scanning electron microscopy (SEM), revealing their porous microstructure.

Regarding the reference cement mortar, an existing formulation from the literature was chosen (Khalil, 2018), which was initially developed for additive manufacturing applications. The mix design contains a Portland cement EXTREMAT® CEM I 52.5 N (noted CEM I), a fast-setting Sulfo-aluminous cement Alpenat R² (noted CSA), both manufactured by VICAT Company (L'Isle-d'Abeau, France), a superplasticizer (SP) SIKA VISCOCRETE TEMPO 11 and standardized siliceous sand (maximal size 2 mm).

The experimental study was carried out on 5 different mortars including the reference one. Mortar mixes denoted Mxx were designed with different dosages xx of dry fibers ranging from 0 to 10 wt. % (with respect to the total mass of all components of the reference mortar M0). The different mortar formulations are summarized in Table 1. The effective mass fractions of miscanthus fibers contained in the fresh mixes are also given in the table (these values are obtained by considering the total amount of water in the mixes). The corresponding volume fractions are difficult to evaluate, because the intrinsic density of the micronized miscanthus fibers is not precisely known; therefore, they are not mentioned in the table.

The reason for the reported increase in the water content of mortars with the added fiber content is explained in the next paragraph.

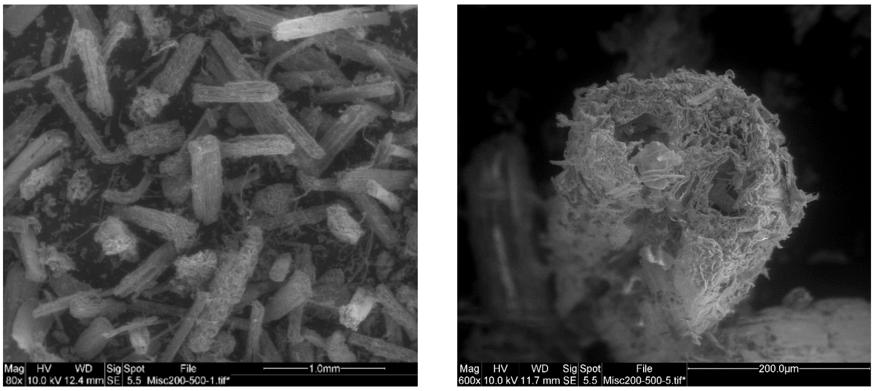

FIGURE 1: SEM pictures of the micronized miscanthus fibers used in this study

TABLE 1: Composition of the different mortars considered in this study

Type of mortar	Sand (g)	CEM I (g)	CSA (g)	SP (g)	Dry fibers (g)	Total water	Water/ (CEM I + CSA) cement ratio	Mass fraction of fibers (wt. %)
M0	1350	1059.33	79.73	2.96	-	398.67	0.35	0
M2.5	1350	1059.33	79.73	2.96	72.27	604.95	0.53	2.28
M5	1350	1059.33	79.73	2.96	144.53	843.61	0.74	4.15
M7.5	1350	1059.33	79.73	2.96	216.80	1092.99	0.96	5.70
M10	1350	1059.33	79.73	2.96	289.07	1383.85	1.21	6.94

Sample preparation was carried out with a programmable mortar mixer, following specifications of EN 196-1 standard. The mixing procedure of the reference mortar M0 includes several steps:

1. First, the mixture of the two types of cement (OPC and CSA in the proportion 97/3 wt. %) was placed in the mixer tank. The water containing superplasticizer was then added to the cement and the zero time was counted from the moment the water came into contact with the cement,

2. The mixing started at a low speed and the sand was added slowly during the first minute while mixing at low-speed. The mortar was then mixed at high speed for 30 more seconds;

3. Rotation was stopped for 30 seconds, to scrape the mortar from the bottom of the tank;

4. Another high-speed mixing was finally performed for 2 minutes 30 seconds.

Regarding mortars with miscanthus fibers, a similar mixing protocol was used but preliminary conditioning of the fibers was necessary. Preliminary tests showed indeed that the addition of dry fibers into the fresh mix leads to a rapid absorption of the mixing water by the miscanthus fibers, which degrades considerably the workability of the system. This can be attributed to the high water absorptivity of these vegetal fibers, which is reported between 390-500% depending on the size of the fibers (Chen et al., 2017). After those preliminary tests and based on a literature review (Onuaguluchi & Banthia, 2016), it was decided to pre-soak the fibers in water for 2 hours before incorporation into mortars. The soaked fibers were then placed in a tissue to eliminate the excess water, and they were added to the fresh mix at the end of step 4. An additional low-speed mixing was performed for 30 seconds followed by high-speed mixing for 1 minute to ensure homogenous fiber distribution.

Immediately after mixing, the various mortar mixtures were cast into metallic molds, to prepare parallelepiped samples of dimensions 4×4×16 cm^3 dedicated to mechanical characterizations, as well as 4x4x8 cm^3 samples dedicated to thermal and hygric characterizations. The molds were filled in 2 successive layers, and each layer was subjected to 52 blows (for about 1 min) using a shock table. After casting, the samples were covered and stored for 24 h at (20 ± 1) °C. They were demolded 24 hours later, hermetically wrapped with a plastic film, and finally conditioned at (20 ± 1) °C until the mechanical characterizations at 28 days. After removing the plastic film at 28 days, samples dedicated to thermophysical and hygric characterizations were additionally stored for few days at 23°C and 50% relative humidity (RH) to reach an equilibrium state before testing.

B. Characterization techniques

The water-accessible porosity and the apparent density of the mortars were determined according to NF P18-459 standard. Specimens of dimension 4x4x8 cm^3 were cut into smaller samples (2x2x4 cm^3) and followed all the steps of characterization described in the standard.

The mechanical strength of the mortars (under flexural and compression tests) was determined at 28 days using a 300 kN capacity press and according to the NF EN 196-1 standard. For each mortar, 6 samples were tested under three-point bending. The resulting 12 halves specimens were then tested in compression. In both cases, mean values and standard deviations were determined.

The thermal conductivity of the various mortars was determined by the Hot Disk (HD) method based on the transient plane source theory (ISO 22007-2), using a TPS 2500S apparatus. This method enables a fast, accurate, and non-destructive evaluation of the thermal conductivity (λ in $W.m^{-1}.K^{-1}$), the thermal diffusivity (α in $m^2.s^{-1}$), and the volumetric heat capacity ($\varrho.Cp$ in $MJ.m^{-3}.K^{-1}$). Experiments were carried out in a climatic chamber, at a constant relative humidity of 50% and over a temperature interval from 5°C to 45°C.

The Moisture Buffer Value (MBV) indicates the capacity of a material to store/release moisture when exposed to moisture variations. MBV determination was carried out for the various mortars based on the NORDTEST Protocol (Rode et al., 2005). This protocol consists in exposing the sample to a constant temperature, with a cyclic variation in relative humidity between high (75% RH) and low (33% RH) levels for periods of 8 and 16 hours respectively.

III. RESULTS AND DISCUSSION

A. Workability of the fresh mortar mixes

In order to evaluate the workability of the various mortar formulations, the slump of the fresh mixes was determined immediately after mixing using the Abrams mini-cone method. Figure 2 shows the evolution curve of the slump versus the weight fraction of miscanthus fibers in the mortars. As a general trend, the mortar workability is significantly reduced when the fiber dosage increases. This effect can not be attributed to the absorption of water by the fibers since these latter were already saturated before incorporation, but it may result from a modification of the overall granular distribution within the material. The same trend was observed by several authors with various types of fibers. For example, Chen *et al.* observed a similar decrease in the workability of ultra-lightweight concrete with the addition of miscanthus fibers (Chen et al., 2017, 2020). Savastano *et al.* and Panesar & Shindman observed the same trend for mortars including sisal fibers and waste cork, respectively (Panesar & Shindman, 2012; Savastano et al., 1999).

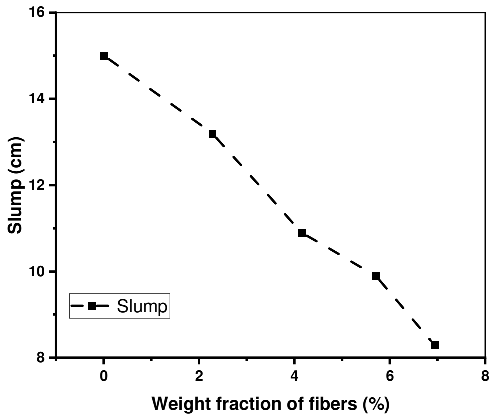

FIGURE 2: Slump of the fresh mortar mixes versus the weight content of miscanthus fibers

B. Dry density and water accessible porosity

Figure 3 shows the evolutions of the apparent dry density and the water-accessible porosity of the hardened mortars as a function of the weight content of miscanthus fibers.

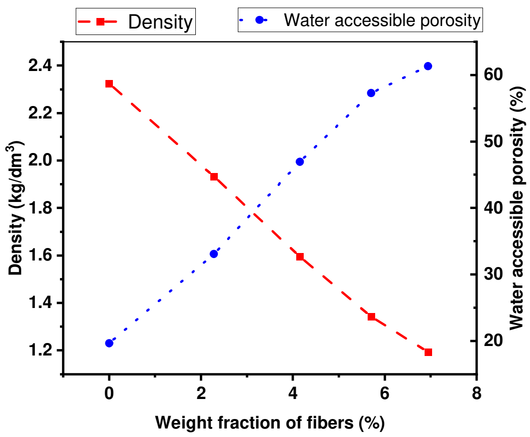

FIGURE 3: Evolutions of the apparent density and water accessible porosity of the hardened mortars as a function of their weight content of miscanthus fibers.

The apparent density of mortars decreases almost linearly as the fiber dosage increases, whereas the opposite trend is observed for the evolution of the water accessible porosity. Similar effects were reported in the literature for mortars including vegetal fibers (Chen et al., 2020; Huyen *et al*, 2011; Chikhi *et al.,* 2013). In general, the authors reported the same decrease in the dry density of mortars with the addition of vegetable fibers, and explained this phenomenon by the influence of multiple factors: i) the lower density of fibers compared to mineral components of the mortars; ii) a modification of the overall granular distribution and internal packing within the bio-based mortars; iii) the contribution of the inner porosity of vegetable fibers and the porosity at the fiber/cement paste interface to the overall porosity of the bio-based mortars, and hence to the drop of dry density. In the present case, fibers are pre-saturated by water, which may also contribute to the overall porosity of the mortar after evaporation of the excess water.

C. Mechanical properties

Figure 4 shows the evolutions of the flexural and compressive strengths of the bio-based mortars at 28 days as a function of the weight content of miscanthus fibers.

These experimental data reveal significant decreases in both the flexural and compressive strengths of mortars with the addition of miscanthus fibers. The compressive strength seems most affected among these two properties, as an addition of 2.28 wt.% miscanthus in the mortar induces a strength reduction of 70 %, from 72 MPa to 21 MPa. With higher fiber contents, the compressive strength becomes lower than the minimum requirement for structural concrete (i.e. 17.2 MPa), and hence the corresponding mortars are not suitable for structural applications.

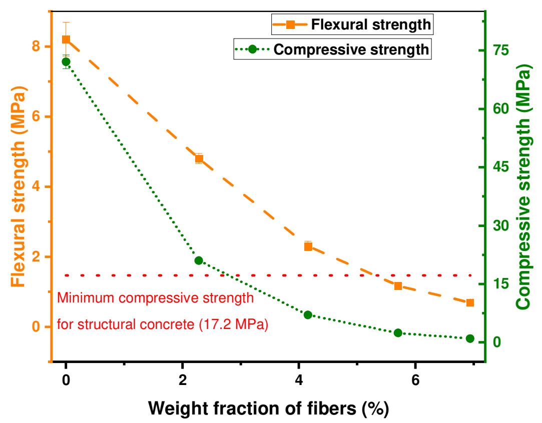

FIGURE 4: **Evolutions of the flexural strength and compressive strength of mortars at 28 days as a function of the weight content of miscanthus fibers.**

Most studies from the literature report similar detrimental effects of the addition of vegetal fibers on the mechanical properties of bio-based mortars. For instance, Chikhi *et al.* found a decrease in compressive strength of 15% at 28 days for mortar including 10 wt.% date palm fibers of 3mm compared to the neat materials (Chikhi et al., 2013). Hernandes-Oliviares *et al.* (Hernández-Olivares et al., 1999) and Li *et al.* (Li et al., 2006) also observed a decrease in compressive strength with the addition of 20 wt.% of cork granules and various ratios/proportions of hemp, respectively. Such a degradation of the mechanical performance of bio-based mortars can be explained by several factors, in agreement with the conclusions of other authors: i) the bulk density of mortar dramatically decreases, as many pores, voids and entrapped air are introduced with the addition of fibers; ii) fibers may behave like voids under mechanical loading of mortars due to their super-lightweight property; iii) bond defects between the fibers and the cementitious matrix may act as weak points, hence contributing to crack initiation/propagation and to the overall strength loss of the bio-based mortars.

D. Thermophysical properties

Figure 5 presents the evolution of the thermal conductivity (λ) of the bio-based mortars at 25°C as a function of the fiber content. It is found that λ decreases almost linearly with the addition of miscanthus fibers into the mortar (the regression line enclosed in the graph shows a R^2 coefficient higher than 0.99). The same observation was made by several authors for various types of mortars including vegetal fibers (Ashour et al., 2010; Chikhi et al., 2013; Panesar & Shindman, 2012).

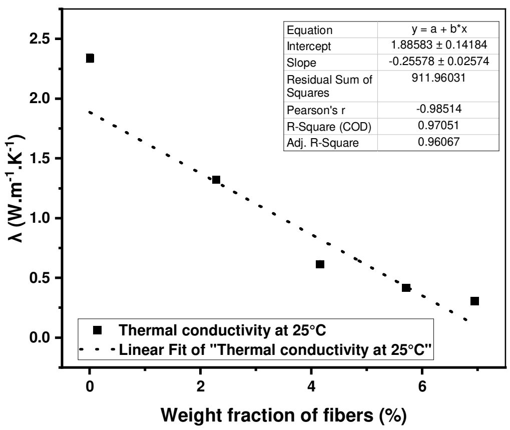

FIGURE 5: Evolutions of the thermal conductivity of mortars at 25 °C as a function of the weight fraction content of miscanthus fibers

It is worth noting that the introduction of 5.7 wt. % of miscanthus fibers leads to a decrease in thermal conductivity of the mortar of about 82%, which makes this material highly suitable for insulation applications. This large impact of the fibers on the thermal conductivity can be attributed both to the low conductivity of the miscanthus fibers themselves (a value of 0.04 $W.m^{-1}.K^{-1}$ is reported in the literature by (Schnabel et al., 2019)), but most certainly to the large increase in the overall porosity of the mortars resulting from the introduction of fibers, as previously highlighted. Indeed, several authors have suggested that the heat transfer process by conduction may be partially replaced by natural convection in the porous phase (Chikhi et al., 2013; Li et al., 2003, 2006; Onuaguluchi & Banthia, 2016; Panesar & Shindman, 2012).

E. Moisture Buffer Value

The moisture buffer values (MBVs) determined for the various bio-based mortars according to the Nordtest protocol are reported in FIGURE . The MBV clearly increases with the fiber content, as it changes from a value of 0.5 $g.m^{-2}.\%RH^{-1}$ for the plain mortar to a value close to 2.0 $g.m^{-2}.\%RH^{-1}$ for mortars with fiber contents over 4.1 wt.%, which corresponds to materials with good or even excellent moisture buffer capacity according to the Nordtest classification.

These results are in accordance with most studies of literature dedicated to bio-based mortars filled with vegetal fibers (Kreiger and Srubar, 2019). For instance, Benmahiddine *et al.* found an MBV of 2.27 $g.m^{-2}.\%RH^{-1}$ for hemp concrete (Benmahiddine et al., 2020). The outstanding moisture buffering capacity of these modified mortars can be attributed to the high intrinsic porosity and specific surface area of the added vegetal fibers (Haba et al 2017). In the present study, a good hygroscopic behavior is observed even at low fiber contents, probably due to the use of a micronized miscanthus powder which increases further the specific surface area of fibers.

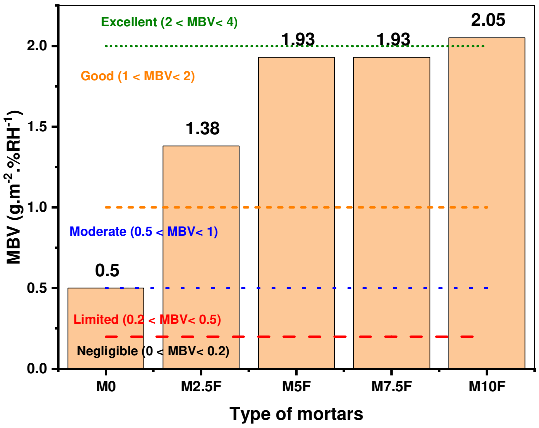

FIGURE 6: Moisture Buffer Value of various mortars including miscanthus fibers and comparison with the Nordtest classification.

IV. CONCLUSION

In this work, we explored the possibility of adding micronized miscanthus fibers into a cement mortar formulation, and investigated the effect of this incorporation on the mechanical, thermal and hygric properties of the resulting bio-based mixes.

As a first result, fiber addition was found to reduce significantly the workability of mortars despite the pre-saturation of fibers by water. This effect was mainly attributed to an alteration of the overall granular distribution and packing density of the mortar mixes.

The microstructure of mortars was also highly affected, with a quasi-linear increase in the overall material porosity as a function of the fiber content. This was considered as a main cause responsible for the degradation of the mechanical properties of the bio-based mortars under flexural and compression loading. Contrarily, a great improvement of both the thermal resistance and the moisture buffer capacity was observed after incorporation of small amounts of vegetal fibers into the mortars. The fact that fibers are micronized probably favors this beneficial effect, as their specific surface area is increased compared to fibers of larger size.

Finally, the results of the various characterizations lead to the following conclusions regarding the possible usages of the bio-based mortar mixes:

- Mortar M2.5 with 2.28 wt. % of fibers is eligible for structural applications, as it still exhibits a compressive strength over the minimum requirement; but the thermal gain and the capacity of moisture regulation remain limited.
- Mortars with dosages of miscanthus fibers over 4.15 wt.% show substantial improvement of the thermal resistance (over 70%) compared to the plain mortar, and

are classified as good or excellent material for moisture regulation according to the Nordtest protocol. These mortars still retain sufficient mechanical properties for handling and are suitable for insulation applications.

The printability of these modified mortars will be explored in a next study.

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