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Research Article

Investigation of Miscanthus and Sunflower Stalk Fiber-Reinforced Composites for Insulation Applications

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The development of materials based on renewable and low-cost resources is today's crucial interest for civil engineering. This work intends to investigate two new vegetable fibers as composite reinforcement for insulation application in the building industry. Miscanthus and sunflower stalk have mainly been selected for their high fiber content, low water content after harvesting, and availability. These criteria lead to good structural properties and allow a reduction in production and transportation costs. This study aims at demonstrating the feasibility to produce cohesive panels from these lignocellulosic fibers and their related interesting mechanical and thermal properties according to various natural binders.

1. Introduction

In recent years, the environmental awareness and the increase in production costs have encouraged institutions to develop biocomposites. Civil engineering is especially affected by such issues as building industry generates about 40% of the global carbon dioxide emission and 40% of global solid waste production [1–3]. Compared to conventional building materials, biocomposites made from vegetable fibers allow long-term benefits to infrastructures (biodegradability, reduction of waste, energy efficiency, less health risk, etc.), thus leading to a much lower environmental impact [4, 5]. The advantages of vegetable fibers for producing building materials are in fact multiple [6]. Firstly, they are abundant, cheap (the majority are agricultural residues), and independent from fossil resources. Secondly, due to their renewability, their environmental impact is minimal. Lastly, vegetable fibers reveal natural thermal insulation ability.

Looking at their high lignocellulose content, vegetable fibers can be used as reinforcement inside wood-polymer composites (WPCs) [7–11], the latter being usable in houses, for example, as slats for outdoor terrace or wall cladding. Lignocellulosic by-products from agriculture or agro-industry can also be used as bioaggregates for producing bio-based insulating concretes [12–14].

Another possible use of vegetable fibers inside buildings results in their promising thermal insulation properties. For instance, lignocellulosic fibers from hemp [15], flax [15], or sunflower [16–19], all three cultivated in France, and from date palm [20] have already been used as insulating materials. The density of insulation boards often influences their thermal conductivity [20–28], and the lowest thermal conductivities are those of low-density materials. As an example, low-density insulation boards made from sunflower pith [26, 28] reveal thermal conductivity values (38–41 mW/m·K at 25°C with density in the 36–47 kg/m^3 range) perfectly comparable to those of conventional insulation materials such as expanded polystyrene (37 mW/m·K with 50 kg/m^3 density), rock wall (36 mW/m·K with 115 kg/m^3 density), and glass wall (35 mW/m·K with 26 kg/m^3 density).

On the contrary, medium-density materials have usually higher thermal conductivities, making these materials less efficient insulators. However, the use of these medium-density materials may be considered in building insulations (e.g., walls and ceilings). Many examples of insulation boards are thus described in the recent literature (from the most insulating to the least one), e.g., from coconut husk (46–68 mW/m·K thermal conductivity with 250–350 kg/m^3 density) [25], coriander stalk fibers (56 mW/m·K thermal conductivity with 155 kg/m^3 density) [29], cake generated during biorefinery of sunflower whole plant in a twin-screw extruder (78–99 mW/m·K thermal conductivity with 358–687 kg/m^3 density) [17, 19], cotton stalk fibers (82 mW/m·K thermal conductivity with 450 kg/m^3 density) [24], hemp fibers (90–108 mW/m·K thermal conductivity with 369–475 kg/m^3 density) [27], coconut coir (104 mW/m·K thermal conductivity with 540 kg/m^3 density) [21], and date palm fibers (150 mW/m·K thermal conductivity with 754 kg/m^3 density) [20]. Here, a correlation appears between the board density and its thermal conductivity: the denser the composite material, the poorer the thermal isolation ability is. Producing cohesive panels for which the density would be minimized as much as possible, thus leading to improved insulating properties, is therefore a key point for future research.

The present study intends to investigate new alternatives for medium-density insulating composite panels based on vegetable fibers. Among potential plant resources unused in the food industry, a first selection was done from the fiber properties. Regarding the chemical composition, the cellulose content was chosen as high as possible since this straight chain polymer generally leads to good structural properties for composites. The lignocellulosic fiber content (as high as possible for better composite strength), the density (as low as possible for better thermal insulation properties), the water content after harvesting (as low as possible to reduce the production cost related to drying), and the availability in Europe (to reduce transportation cost) were other factors considered. According to these criteria, two candidates appear as relevant in the present case, namely, miscanthus and sunflower stalk fibers.

Miscanthus (*Miscanthus giganteus*) is a large perennial grass (also called elephant grass) that grows in many European countries. It is currently used as a commercial biomass crop, as a source of heat and electricity, or converted into biofuel products such as bioethanol. Injected thermoplastic composites based on miscanthus fibers and Mater-Bi® or poly(lactic acid) (PLA) or polypropylene (PP) matrices have been extensively investigated and show interesting mechanical reinforcement [7, 9]. Nevertheless, the question of their use within thermal insulating panels has not been studied yet.

Sunflower (*Helianthus annuus* L.) is an herbaceous plant mainly cultivated in China, USA, Russia, and Western Europe, whose seeds are used to gain oil. The stalk is then a waste product of its cultivation. Insulation boards with satisfactory thermal conductivity have already been prepared from these fibers, either under pressure with epoxy binder [16], by thermopressing with protein mixture [17] or by compression molding after addition of chitosan [18]. In the first case, the addition of epoxy binder to produce cohesive materials can lead to the emission of COVs during time, making these materials not really environmentally friendly [16]. In addition, in the two first cases, the use during molding of pressure [16], and both pressure and temperature [17], will contribute undoubtedly to significantly increase the production cost of such insulating materials. Lastly, even if the natural binder (i.e., chitosan) used by Mati-Baouche et al. [18] to produce bio-based composites from sunflower stalk particles is commercially available, it is known as a relatively expensive binder.

The first originality of this work stands in the new application field, regarding thermal insulation expected for miscanthus fibers. At the same time, we intend to promote the sunflower cultivation for which the stalks could also be seen as potential commercial products for farmers. Moreover, obtaining an acceptable finished product from purely renewable resources and at low-cost constitutes another main challenge. Accordingly, natural water-soluble binders have been considered to provide the cohesion to composite materials. In addition to their commercial availability and low price, the use of these binders leads to fully renewable and biodegradable composites. Also, manufacturing process by low pressure at ambient temperature was favored to get low-density panels and therefore to improve thermal insulation properties. This preliminary study thus aims at evaluating manufacturing aspects and related mechanical and thermal properties of miscanthus and sunflower stalk composite panels.

2. Materials and Methods

2.1. Materials and Manufacturing Process. The miscanthus fibers bought from the Burlerrow Farm company (UK) can directly be used for the panel production. They are delivered at their natural moisture content (9.0%), which avoids fungi contamination during storage, and milled, meaning that such a material is directly moldable. The sunflower stalks were manually harvested from a field of Arterris company (France). It was first dried during 24 h at 70°C and then milled with a 1.5 cm screen. To remove small solid particles, the material was manually sieved to obtain raw fibers equal or bigger than 1 cm. It was also equilibrated in a climatic chamber (60.0% relative humidity, 25°C) during 3 weeks before compression molding. After equilibration, its moisture content was 9.6% [30]. The density of fibers stands around 160 kg/m^3.

Regarding natural binders, three different binders were tested. They were chosen because they were all available commercially. Firstly, a starch-based binder was used. Supplied by Bostik (France), it is usually used as wallpaper glue. Batch number 28474 used for this study was made of starch, a polymer of glucose, with 85% purity. The second binder was wood glue made of casein (97% purity) from Ipharos (France). Precipitated from milk, this protein-based binder is sold under the name Collaseum Caseo Bois. Lastly, bone glue made of gelatin from Briançon Production (France) was also used. With 90% of purity, this second protein-based binder is usually used in crafts, e.g., for restoring antique furniture, in marquetry, etc. The two first binders are delivered in the form of powders, while gelatin is as granules. These three natural binders are known as external binders with physical curing. Before using them, they are firstly solubilized in water, and they are then mixed to the lignocellulosic residue chosen, i.e., miscanthus or sunflower stalk fibers here, before compression molding using low applied pressure. The adhesion can then be obtained through the mixture drying inside a ventilated oven, which allows water evaporation. This results in the appearance of new bonds of hydrogen type between the biopolymer inside the binder (i.e., starch, casein, or gelatin) and the ones from the lignocellulosic residue used (i.e., mainly cellulose and hemicelluloses). After complete drying, the use of natural binders thus contributes to good cohesion for the obtained composite material while preserving a low density, which is required for thermal insulation applications.

In a practical point of view for this study, firstly, each binder was dissolved in cold water (for starch) or up to 60°C hot water (for gelatin and casein). This step should therefore take into account the water absorption of the raw plant material. In this case, the dissolution of binder inside water was executed by a simple blender and the mixture between the binder in aqueous solution and the material was done manually. Several binder contents have been considered, namely, between 10% (to get the required cohesion) and 40% (higher values become useless for the composite). The mixture was then filled manually into a rigid squared mold, and the molding process was executed at ambient temperature by means of a hydraulic press with low pressure (only 90 kPa). The last step was to dry panels in order to get final moisture content of composites of less than 10% to guarantee the material durability. To avoid any damage, a constant temperature in the ventilated oven of 80°C for a drying time of 50 h was considered. Note that panels were then equilibrated in a climatic chamber (25°C, 60% RH) during three weeks before all measurements. From that procedure, composite panels with mean dimensions of 15 cm × 15 cm × 5 cm could be obtained (Figure 1).

2.2. Experimental Investigation. According to the final application, the focus was first put on the panel density as it is closely related to insulating quality. All manufactured boards are shaped squared with solid edges, so such a determination simply requires the weight of the board and its thickness (generally 5.0 ± 0.5 cm). Some configurations do not appear for all binder contents, due to difficulties in the manufacturing process (mixing problems, cohesion loss, etc.). Composite panels exhibit density values less than 450 kg/m^3, making them medium-density materials, which tend to achieve interesting insulating properties (Figure 2). Results obtained are even better with miscanthus (M) fibers (an average value of only 190 kg/m^3) compared to sunflower stalk (S) fibers (280 kg/m^3). In addition, an increase in the panel density is observed in all cases with the increase in the binder content.

The bending behavior of the biocomposites is then investigated to ensure the mechanical stability when handling panels [31]. Such a test was performed with an Instron 33R4204 (USA) universal testing machine equipped with a three-point bending test device (distance between spans of 100 mm, 93 500 N load cell, and crosshead speed of 2 mm/min). All determinations were carried out from five equal elementary samples cut in manufactured panels (a mean sample dimension of 15 cm × 5 cm × 2.8 cm). Taking into account the accurate dimensions of each sample, such a test provides the material Young's modulus and its yield bending stress.

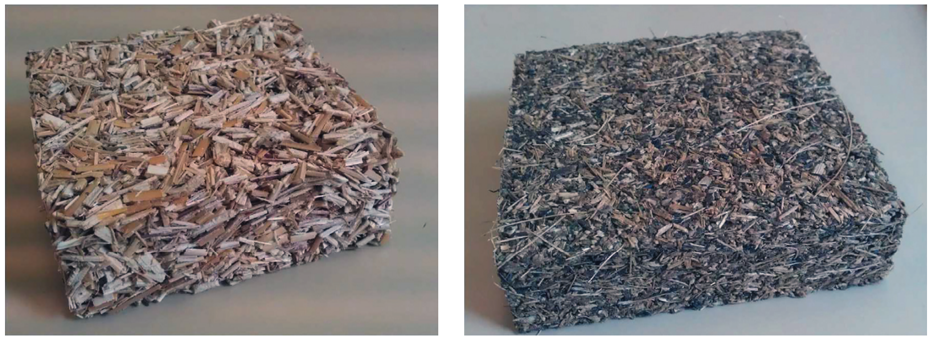
Finally, thermal conductivity measurement was done with Lambda-Meßtechnik GmbH Dresden 98 EP 500 (Germany) l meter [32]. The panel is surrounded by an insulating material, that is, rubber foam, and placed in the guarded hot plate apparatus. It is submitted to a heat flow such that the temperature difference between the faces of the sample is equal to 5 K. The test is done three times with different temperature settings to cover the range of thermal conditions encountered at different seasons (between 10°C and 40°C). From the specimen dimensions, one could deduce the thermal conductivity λ and thermal resistance R.

3. Results and Discussion

Even if the density results were promising, some manufactured panels did not satisfy building quality requirements regarding surface aspect (too porous surface) and global cohesion (lost material after the drying process, breaking while cutting the mechanical test specimens). This is generally due to the morphology of fibers and also viscosity of binder solutions that prevent to achieve a homogeneous mixture. For instance, it was not possible to obtain a satisfactory association between miscanthus fibers and gelatin, whatever the binder content. Accordingly, only nine configurations could be tested mechanically: four panels from miscanthus and five from sunflower stalks. Among them, three were based on starch, five on casein, and only one on gelatin.

Bending Young's moduli and yield stresses measured are shown in Figures 3 and 4. Mechanical properties also increase with binder content, and tendencies observed on Young's modulus and yield stress are quite equivalent. More precisely, two groups can be clearly distinguished:

(i) Panels based on sunflower/casein with at least 30% binder content exhibit high bending properties (a rigidity of around 11 MPa, and a yield strength of around 1 MPa);



(a) (b)

Figure 1: Composite materials. (a) Miscanthus fiber panel. (b) Sunflower stalk fiber panel.

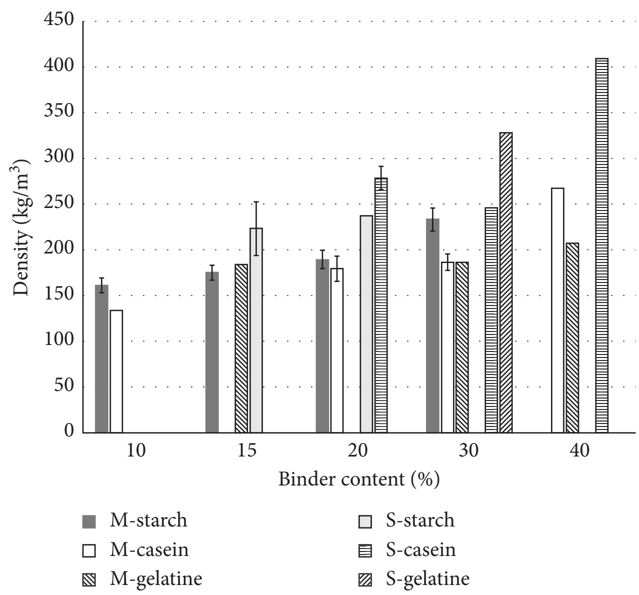


Figure 2: Panel density.

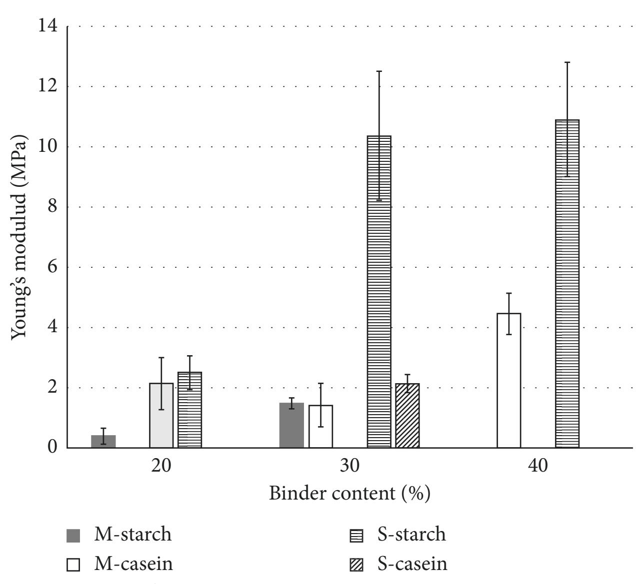


Figure 3: Bending Young's modulus.

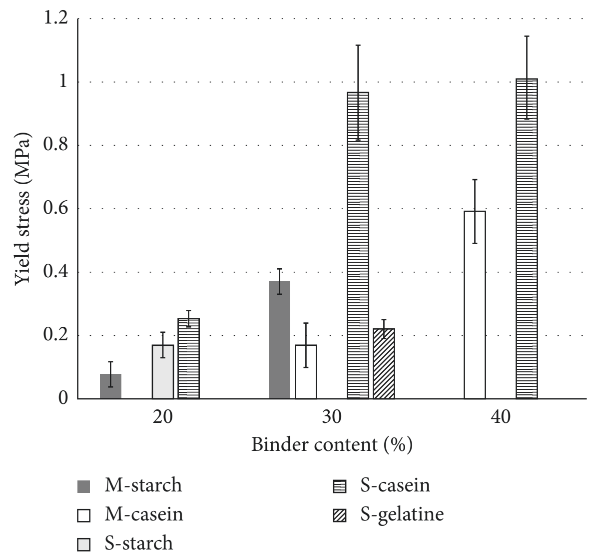


Figure 4: Yield bending stress.

(ii) All other panels lead to quite similar and much lower performances (a rigidity of around 3 MPa, and a yield strength of around 0.3 MPa). Regarding miscanthus fibers, 40% casein leads to the strongest behavior.

In order to guide thermal tests, a selection was done on the basis of these first results. Here, requirements are reasonable mechanical properties, good thermal properties, and, above all, a low cost (essential to enter the market). In this way, an optimization factor F is introduced to estimate quantitatively the performance of each panel with respect to all these constraints and to be able to compare the different configurations:

$$F = \frac{\sigma}{d^2 b^4}, \quad (1)$$

where σ denotes the mean yield bending stress, d the panel density, and b the binder content. Precisely, the mechanical performance (through the mean yield stress σ placed in the numerator of F) seems essential for handling of panels and their installation in buildings. Moreover, as said before, best thermal properties are generally associated with low density. At the same time, business issues must be taken into account. In the present case, the panels manufacturing cost mainly depends on the binder price (price of fibers is around 0.20 €/kg, whereas binders rather cost 5 €/kg) and consequently on the binder content. Commercially viable solutions for insulation applications thus require the minimization of both the density parameter d and binder content b (accordingly these two parameters appear at the denominator of F). Note finally that powers, respectively, applied to mechanical resistance (order 1 for yield stress σ), thermal performance (order 2 for density d), and manufacturing cost (order 4 for binder content b) account for the respective weights of these aspects in view of the development of new products.

From the values of factor F (Figure 5), configurations that appear as the best compromises (highest value) are Miscanthus/starch and sunflower/casein with a binder content of 20% in both cases. The focus was thus put on these two couples for the thermal analysis (Table 1). One could note that Miscanthus/starch exhibits the best thermal properties in agreement with its lower density (Figure 2). In France, a thermal conductivity value λ less than 40 mW/m·K and/or a thermal resistance value R bigger than 0.5 m^2·K/W is requested for insulation panels to be used in commercial applications. The two selected configurations satisfy the second criterion whatever the temperature and could then be employed for the building industry. In addition, looking at their densities (Figure 2) and especially at their thermal conductivities at ambient temperature (Table 1), these two optimal medium-density composite materials revealed a poorer insulating behavior than expanded polystyrene, rock wall, and glass wall. However, this behavior was comparable to that of other insulation materials made from locally available other products (e.g., hemp fibers, sunflower cake from whole plant, and coriander straw). They were less dense and revealed better thermal insulating properties than hemp fiber insulation boards (369–475 kg/m^3 density and 90–108 mW/m·K thermal conductivity) [27] and thermal insulation fiberboards made from sunflower cake, using hot pressing (687 kg/m^3 density and 99 mW/m·K thermal conductivity) [17] or also compression molding after 20% starch-based binder addition (358 kg/m^3 density and 78 mW/m·K thermal conductivity) [19]. On the contrary, a more recent experimental insulating material made of milled coriander straw (7.5 mm sieve) and 15% starch as binder with 155 kg/m^3 density revealed a slightly reduced thermal conductivity (56 mW/m·K) [29].

4. Conclusions

The feasibility of manufacturing panels with miscanthus or sunflower stalk fibers and natural binders and their related

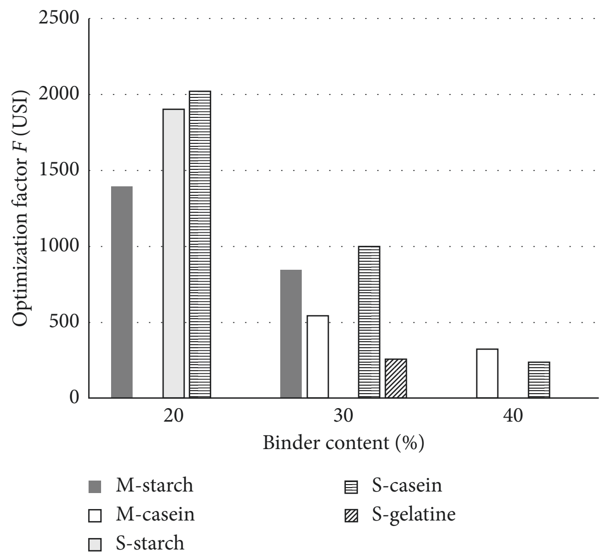


Figure 5: F performance of mechanically tested configurations.

Table 1: Thermal conductivity λ and thermal resistance R according to temperature T for composites panels with 20% binder content.

T (°C)	Miscanthus/starch		Sunflower/casein	
	λ (mW/m·K)	R (m^2·K/W)	λ (mW/m·K)	R (m^2·K/W)
10	57.02	1.070	65.24	0.680
25	61.27	0.992	70.42	0.631
40	67.55	0.900	77.42	0.573

interesting properties have been demonstrated. Compared to existing thermal insulation materials, these two experimental ones thus appear as viable options for future commercial applications. On the one hand, their bending properties allow easy handling without risk of breaking them when positioned inside buildings. On the other hand, their thermal insulation properties are situated in the same range of other medium-density materials [19, 25, 29]. However, for future work and before using them inside buildings, other characteristics should be completed among which it is already possible to mention the water vapor permeability and vapor sorption isotherm, resistance to fire, and resistance to rodents, pests, etc. In addition, the antimicrobial properties (i.e., the resistance to fungi) of these new materials will also need to be investigated. "Green" solutions consisting in coating glycerol esters at the surface of insulation materials recently demonstrated their efficiency for preventing microbial/bacterial growth over time [33]. Glycerol esters valuable by-products of agroindustry are already used for their significant antimicrobial effect, especially in the food industry. Indeed, microbial proliferation leads to the deterioration of both materials and indoor air quality (indirectly induced by the release of airborne microbial contaminants, including spores, fragments, toxins, and mVOC), and bio-based building materials are especially sensitive to such degradation because they contain large amounts of cellulose or derivatives. Lastly, accelerated aging of these two new thermal insulation composites would make possible to ensure the durability of their properties of use over time. Regarding microstructural aspects, further studies are also needed to better understand the mechanisms involved, specially the fiber wetting according to the viscosity of the binder solution and the creation process of hydrogen bonds between the binder and hydroxyl groups of cellulose. All these complementary characterizations would undoubtedly give much more information about the chances of eventually using these reinforced insulating composites inside buildings.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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