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Céline Badouard, Christophe Bliard, Guillaume Polidori, Fabien Bogard. Development and characterization of a new composite based on grape stalks and potato starch for building insulation. ICoGB 2022, Ms. Chris Y. Cheung, Jun 2022, Stockholm, Sweden. hal-04247813

HAL Id: hal-04247813

<https://hal.science/hal-04247813>

Submitted on 18 Oct 2023

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Development and characterization of a new composite based on grape stalks and potato starch for building insulation

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Abstract. Several agricultural waste products can be recycled into biomaterials. This article describes the potential application of grape stalks in the manufacture of ecological lignocellulosic insulation panels. Panels such as these can reduce the energy consumption of buildings, thereby reducing the CO₂ emissions, as well as providing good thermal, hydrothermal, and acoustic insulation. This study describes the properties of agrocomposites made from agricultural waste: stalks and crushed stalks. Building materials made from bio-composites made from stalks and starch exhibit excellent thermal, acoustic, and moisture-buffering properties, making them suitable for use as building materials.

1 Introduction

Nowadays, energy consumption is increasing in all areas around the world. Meanwhile, the cost of energy has increased due to the exhaustion of energy sources and the high environmental impact (greenhouse gases, global warming, etc) [1]. The French building industry represents 43% of energy consumption and 25% of total greenhouse gas emissions. It is an industry that is evolving to reduce its environmental footprint.

France has committed to drastic reductions in its carbon emissions. In order to meet this commitment, a new environmental regulation of the energy transition of buildings has been implemented, RE 2020 [2]. It requires the improvement of building insulation performance (thermal insulation, absorption, and acoustic insulation), by promoting low-emission construction methods, biosourced construction materials, and finally by integrating a summer comfort objective to adapt buildings to global warming. The development of materials that respect health and the environment is a priority issue and these new materials will have to meet new criteria such as a renewable resource, the recyclability of a material, and a moderate cost.

Insulation is a key factor in the energy performance of buildings. Hence the necessity to develop materials for civil engineering using minimal fossil resources. Agricultural waste represents a vast potential to be used as biosourced materials for insulation. The valorization of these raw materials as agromaterials would diversify the sources of supply, provide a complementary source of revenues for farmers and adapt the composition of particleboard to locally available resources.

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Grapes are one of the most cultivated fruits in the world. The production of wine generates a considerable amount of grape pomace, in France, 850,000 tons are produced per year. The grape pomace is called “marc” or “aignes”, in the Champagne region which corresponds to the totality of the solid parts (skins, stalks, and seeds) that remain at the end of the pressing.

Starch is a polysaccharide widely present in agricultural products such as cereals, tubers, and legumes. Starch has already been used for the development of bio-based composites for civil engineering applications such as hemp/starch composites, date palm fiber/cassava starch, and beet pulp/potato starch [3,4,5].

In this work, we describe new agrocomposites based on stalks or crushed stalks and a biosourced binder based on potato starch. The fabrication and characterization of these agro-composites by different methods and techniques are presented. An investigation of thermal, acoustic, and hygric behavior was carried out. Two parameters are studied: the influence of stalk grinding and the stalk to starch ratio. The ratio of stems to starch is fixed at 10, 20, and 30 %, and two types of stalks are used: whole stalks and crushed stalks.

2 Materials and Methods

2.1 Composite Preparation Methods

The wine co-products, stalks, and crushed stalks were recovered from a local plant during harvest. The stalks (S) and crushed stalks (CS) were mixed with a binder at different ratios (10, 20, and 30%). The chosen binder was potato starch (A), which was supplied by Roquette (Lestrem, France). By heating the starch powder to 95°C in water, one obtains an irreversible gelatinisation caused by the swelling of the grains. The resulting gel serves as a binding agent for solid aggregates. The starch/water ratio was set at 0.1. The water and starch were heated under strong mechanical agitation until they had a translucent appearance and high viscosity. Stalks, and crushed stalks were mixed and kneaded with the starch gel for a few minutes in order to obtain a good homogeneity. The resulting mixtures were formed into moulds and then compacted at a pressure of 40 kPa for five minutes. Starch gel alone was also prepared as a reference. After being frozen, the samples were freeze-dried at 3 mbars during several days.

2.2 Composite analysis

The density ρ_{bulk} of the composites is calculated by weighting the samples and determining their total volume. The volume is calculated based on the dimensions (width, length, and height or diameter and height) of the composites measured five times for each sample. The bulk density of the composite was averaged over twelve samples for each composition.

The absolute density ρ_{abs} of the composites is calculated using the absolute densities of the raw materials. The densities and porosity of stalks and starch were analyzed by the authors in a previous paper [6] and are presented in Table 1.

Table 1. Densities and porosity of raw materials [6]

<i>Raw materials</i>	ρ_{bulk} (kg/m ³)	ρ_{abs} (kg/m ³)	<i>Porosity</i> (%)
Grape Stalks (S)	61 ± 3	1022 ± 71	94 ± 1
Crushed Stalks (CS)	116 ± 5	1022 ± 71	89 ± 1
Freeze-dry starch gel (A)	178 ± 7	1200 ± 82	85 ± 2

2.3 Thermal properties

The thermal conductivity (λ) of the composites was determined using an Applied Precision ISOMET 2114 probe. The heat flux was generated by heating an electrical resistor inserted into the sample to ensure direct thermal contact with the sample. This measurement analyzes the temperature response of the measured composite to heat flux pulses. The evaluation of thermal conductivity (λ) is based on temperature measurements taken periodically as a function of time.

A series of three cubic samples (10 x 10 x 10 cm) for each composition was made. After drying, the samples were equilibrated at 23°C and 50% RH before measurements. A total of ten measurements was made for each sample and the results were averaged.

2.4 Acoustic properties

The Kundt tube 4206 (tube with two fixed microphones) from Brüel & Kjaer was used to measure the sound absorption coefficient α . This device consists of a cylindrical tube with two quarter-inch microphones type 4187 BK, a power amplifier BK 2735, and a 3160-A-042 analyzer type. The Kundt tube measures sound absorption as well as surface impedance according to NF EN ISO 10534-2.

The sound absorption coefficient was measured in a frequency range of 50 Hz to 6 400 Hz, which corresponds to the frequency range of noise usually emitted in buildings.

Cylindrical test pieces of each formulation were prepared with various thicknesses. The edges of the samples were sealed with adhesive tape and an adapter was used to fill in the small gap caused by shrinkage during the drying procedure and avoid acoustic leakage. Each batch of measurements was averaged over six tests with a 2 Hz measurement resolution.

According to ISO 11654, the sound absorption coefficient (α_w) is calculated by using the practical sound absorption coefficient (α_p) values based on standard frequencies and compared to a reference curve. The α_w parameter corresponds to the value of the reference curve at 500Hz.

2.5 Moisture Buffer Value (MBV)

For this test, the edges and the backsides of samples were sealed to obtain a one-dimensional moisture flow. Samples were stabilized at 23°C and 50% RH in a climatic chamber and weighted until they reach equilibrium. Three 10cm diameter and 4 cm thick cylindrical samples were tested for each formula. All results have been averaged. After stabilization, the Nordtest protocol [7] defines cyclic step-changes in relative humidity between high (75%) and low (33%) values for 8 and 16h, respectively. During the periodic exposure, the samples were weighed five times during the absorption phase and twice during the desorption phase. The moisture buffering value (MBV) was calculated using to Eq.1:

$$MBV = \Delta m / (A \times (RH_{high} - RH_{low})) \quad (1)$$

Where A (m²) is the area of the sample in contact with air. RH_{high} and RH_{low} represent, respectively, the high relative humidity (75% RH) and the low relative humidity (33% RH), and Δm represents the change in mass during the absorption/desorption phase (g).

3 Results

3.1 Density and porosity

Results of densities and porosity are shown in Table 2. These composites have a bulk density of less than 350 kg/m^3 . Absolute density increases with the starch ratio, but bulk density evolves differently. Composites containing 10 and 20% starch have a higher bulk density. From 30% starch on, the bulk density of the composites decreases. The presence of starch aerogel fills the inter-particle space between the stalk aggregates. However, during the drying step, water escapes from the samples and forms capillary voids between the stalk aggregates and the starch binder. In consequence, the composites contain both closed pores and more or less accessible open pores. The porosity of the composites ranges from 67% to 82%.

Among the composites with the least starch, S/A-0.1 and CS/A-0.1 have the lowest bulk density and the highest porosity, 82%. The grinding of the stalks increases the bulk density of the composites. Moreover, the complex structure of the stalks allows to create composites with high porosities. **With regard to vegetable insulation materials, hemp/starch mixtures have densities of about 130 kg/m^3 [3] and cork-based panels have very variable densities depending on particle size and range from $108\text{-}390 \text{ kg/m}^3$ [8]. However, commercial petroleum and mineral insulation materials generally have densities less than 100 kg/m^3 .**

Table 2. Densities and porosity of composites

	S/A-0.1	S/A-0.2	S/A-0.3	CS/A-0.1	CS/A-0.2	CS/A-0.3	A-1
ρ_{bulk} (kg/m^3)	177 ± 38	227 ± 47	223 ± 28	183 ± 25	345 ± 35	261 ± 15	178 ± 8
ρ_{abs} (kg/m^3)	1037 ± 70	1053 ± 85	1069 ± 62	1037 ± 70	1053 ± 85	1069 ± 62	1200 ± 71
Porosity (%)	82 ± 3	78 ± 4	79 ± 4	82 ± 3	67 ± 3	76 ± 3	85 ± 2

3.2 Thermal characteristics

Table 3 shows the thermal measurements λ for different aggregate-starch mixtures and freeze-dry starch gel. The thermal conductivity is close for all composites around (ca 0.0663 W/(m.K)).

Table 3. Thermal conductivity of the different compositions

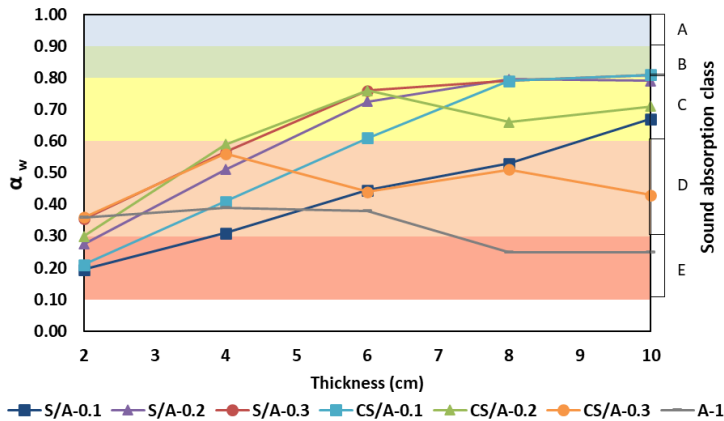
λ W/(m.K)	Stalks (S)	Crushed stalks (CS)	Starch (A)
Starch Ratio	0.1	$0,0651 \pm 0,0005$	$0,0656 \pm 0,0004$
	0.2	$0,0693 \pm 0,0039$	$0,0771 \pm 0,0015$
	0.3	$0,0590 \pm 0,0015$	$0,0617 \pm 0,0006$
	1	-	-
			$0,0658 \pm 0,0042$

Composites with 20% starch had a higher thermal conductivity than composites with 10% or 30% starch as well as starch aerogel. S/A-0.2 and CS/A-0.2 have lower porosities than the other composites, which may explain the observed difference.

The incorporation of stalks in the starch aerogel lowers the thermal conductivity at 30% starch, on the other hand; the grinding of the stalks does not lower thermal conductivity of the obtained composites. As a matter of fact, the value of λ for composites based on whole stalks is lower than the value for composites based on crushed stalks, no

matter how much starch is added. Considering that starch aerogel has had a conductivity of 0.0658 W/(m.K), the arrangement of aggregates in the composites does affect the thermal conductivity.

Hemp-starch composites have a thermal conductivity of ~ 0.066 W/(m.K) [3], as do cork-based panels, which have a thermal conductivity of between 0.047 and 0.081 W/(m.K) (due to differences in particle size in the panels) [8]. The thermal conductivity of commercial petroleum or mineral insulation materials ranges from 0.02 to 0.08 W/(m.K).



3.3 Acoustic characteristics

Fig. 1 shows the evolution of the weighted absorption coefficient α_w as a function of sample thickness and different classes of sound absorption. From A to E, the parameter α_w ranges

Fig. 1. Evolution of the weighted absorption coefficient α_w as a function of sample thickness for the different composites and sound absorption classes

from 0 to 1. Class A is the highest absorption class.

For the starch aerogel, increasing the thickness does not improve the α_w parameter. As a matter of fact, from 6cm on, there is even a decrease of the α_w from 0.40 to 0.25. For the stalks-based composites, there is a progressive increase of the α_w with the thickness but also with the starch content in the composites. The S/A-0.2 and S/A-0.3 composites have had a weighted absorption coefficient that seems to stabilize from 8 cm thickness at 0.80. For the composites based on crushed stalks, the evolution of the acoustic properties has not been so well defined. It is interesting to notice that for the composite CS/A-0.2, there is a progressive evolution followed by stabilization, whereas for the composites CS/A-0.1 and CS/A-0.3, there is a slight increase at first followed by a fluctuation of α_w with thickness. The porosity with the presence of open or/and closed pores has a noticeable influence on the acoustic properties of the materials.

3.3 Moisture Buffer Value (MBV)

It is possible to simulate realistically a house's daily climatic conditions using this method of dynamic characterization of water buffering capacities and to translate the relative humidity control ability of the material. During these cycles, composite materials will absorb and desorb water at a rate of 75% and 33% respectively. As the mass variation of

the samples appeared to have stabilized in the last three cycles, the MBV value was calculated and averaged.

According to the classification proposed by Rode [7], the results presented in Fig. 2. clearly show that all composite materials are excellent relative humidity regulators ($MBV > 2 \text{ g}/(\%RH.m^2)$). The grinding of the stalks and the starch content do not seem to have a direct influence.

By absorbing and desorbing water vapor, these composites can regulate the humidity of the interior air, reducing the ventilation rate and the need for heat in the winter and air

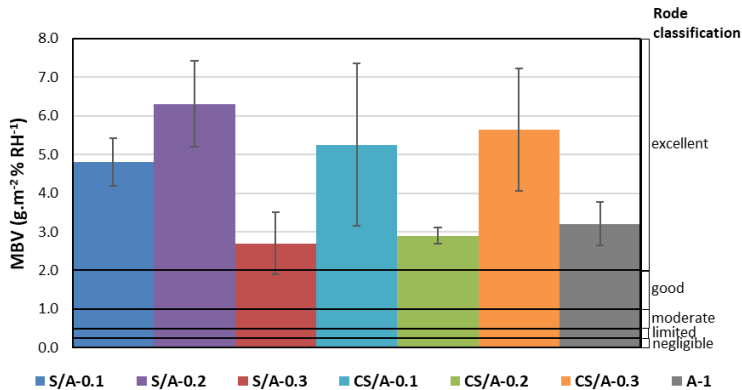


Fig. 2. Moisture buffer values of composites and the Rode classification conditioning in the summer.

4 Conclusion

The composite based on stalks with 30% starch, S/A-0.3, displays the best thermal and acoustic characteristics, it does not have the best MBV among all the studied composites, but it is still classified as an excellent moisture regulator.

Furthermore, materials of plant origin are sensitive to excess moisture since it favors the development of microorganisms, discomfort, but also deterioration of performance and thermal stability. Studies are conducted on the durability of composites (fungal evolution, water immersion, resistance and fire tests). In order to address these problems, other surface treatments using linseed oil or grape seeds are being studied to create a resistance to microbial degradation and make these materials usable in the building industry.

In addition, it will be interesting to examine how much carbon can be sequestered in these materials, as well as comparing life cycle analysis between vegetable materials from agro-waste and mineral and petroleum-based products.

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