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Mechanical and thermal characterization of a beet pulp-starch composite for building applications

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Abstract: This work shows the making of a new bio-based material for building insulation from sugar beet pulp and potato starch. The material is both lightweight and ecofriendly. The influence of starch/ sugar beet pulp ratio (S/BP) is studied. Four binder mass dosages are considered, 10, 20, 30 and 40% (relative to the beet pulp). Samples are characterized in terms of absolute and bulk density, compressive and flexural strength, as well as thermal properties (thermal conductivity and thermal inertia). The compressive strength increases linearly with the S/BP mass ratio to reach 0.52 MPa and the compressive strain is 30%. The thermal conductivity is to around 0.070 W/m. K. The results obtained shows that increasing starch amount tends to decrease composite porosity but increases thermal conductivity and mechanical properties. Depending on the starch content, beet pulp composites have a good thermal and can be used as building materials.

1 Introduction

The building sector is responsible to around 50% of total energy consumption and 11% of CO₂ gas emissions. Therefore, the use of a renewable energy and an ecofriendly and sustainable materials in building application is needed to reduce both environmental impacts and primary energy use. The use of bio-based composites appears as a good solution while maintaining high indoor comfort [1–4]. In this context, developing innovative building material from locally available renewable resources seem a good solution to enhance the energy characteristics of buildings and induce the energy consuming behavior [4].

Several studies have already been published on the use of residual by-products for building applications. Boussetoua et al studied the properties of a cork-cement composite. The results show that increasing the amount of cork granule decreases the thermal conductivity and the mechanical properties [5]. The thermal and mechanical properties of starch-hemp composites were analyzed in different papers [6–9]. The results show that increasing hemp content tends to decrease slightly the mechanical properties. However, Le et al deduced that the mechanical properties depend strongly on the Hemp/Starch ratio and

the hemp shives sizes. The thermal conductivity values were low ($\lambda = 0.0643$ W/m. K) which provides an excellent thermal insulation. Furthermore, Niang et al was studied the potential to use an agro-material based on Typha-Clay in building application. The results show a low thermal conductivity (0.11 W/m. K) [2]. Sugar beet (see Figure 1a) (*Beta vulgaris var. saccharifera*) is widely grown in France (32 millions of tons / year). The process of producing beet sugar generates enormous quantities of by-products [10]. One of these by-products is sugar beet pulp. In the factory, clean beets emerge from the washhouse and fall into the root cutter, where knives, driven by a large diameter disk, cut the roots into thin, rigid strips called *Cossettes*. After extraction the sugar beet pulp is mechanically compressed at elevated temperature using an extrusion machine to obtain to produce the extruded sugar beet pulp (BP). BP is recognized as an excellent feed for livestock due to its high nutritional value and palatability [11]. In recent years, beet pulp has become an important source of gelling pectin. The pectin is extracted from the pulp of the sugar beet from various extraction procedures [12–14] and used in food and cosmetics industries. It has also been used as a bio-adsorbent for the removal of heavy metals [15].

One study has already been published on BP based concrete using a binder based on cement. In this study

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authors present various chemical and physical treatments carried out on BP to modify its properties [16].

Starch is the main carbohydrate reserve of higher plants. It represents a significant weight fraction in many agricultural raw materials such as cereals (30% to 70%), tubers (60% to 90%) and legumes (25% to 50%). Starch is composed of two polymers with a different primary structure: Amylose, (linear), and amylopectin (branched) [17]. It is already widely used in many industrial sectors, mainly paper, textile and also food via the beverage, confectionery and baked goods industries. The chemical industry uses starch in fermentation processes to produce bioethanol, surface treatment products, formulation of adhesives, encapsulation of pharmaceutical products, cosmetics, and biodegradable plastics [18].

Based on the above-mentioned literature review, the present work describes the elaboration of a new agro-composite based on sugar beet pulp and potato starch as a bio-sourced binder. Physical mechanical and hygrothermal characteristics are measured with different compositions designed to be used in the building sector for walls and floor insulation applications.

2 Materials and methods

2.1 Materials

2.1.1 Extruded beet pulp

The 8-10 mm diameter extruded beet pulp pellets BP (18% humidity) (Figure 1 b,c) were provided by Cristal Union factory (Pomacle, France). The BP has been immersed in the water and dried at 50 °C before use Fig. 1(d). To ensure proper conservation in the lab the pulps were kept at -20°C until use. The dominate size of BP is comprise between 2 and 4 mm (Fig. 2). The pulp size influence the mechanical properties [7].



Fig. 1. (a) Sugar beet fruit, (b) fresh sugar beet pulp, (c) extruded beet pulp pellets and (d) extruded beet pulp dried.

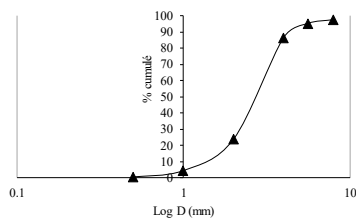


Fig. 2. Grain size distribution curve of BP.

2.1.2 Potato starch

Potato starch was purchased from ROQUETTE, Lestrem, France (Fig. 3). It has a high polymerization degree which gives a viscous binder and provides a good mechanical property for the BP – starch formulas.

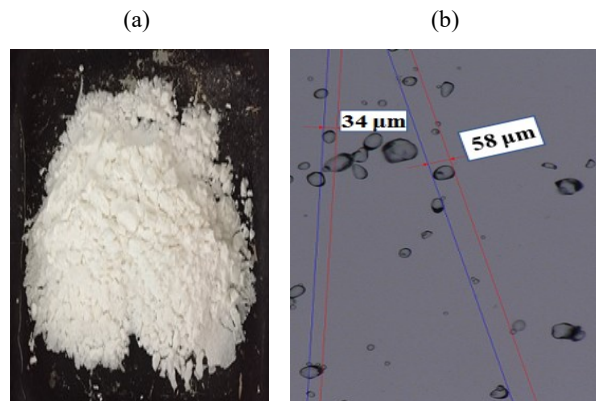


Fig. 3. (a) Powder of potato starch (b) Potato starch grains.

2.2 Formulation of composites

Starch and sugar beet pulps are hydrophilic materials which imposes a competition between these two components to absorb water from the mixture at risk of have a binder with undissolved starch grains and a mixture of wet pulps. Several studies have proposed to prepare the binder separately, with optimum dynamic viscosity and surface tension [8,14]. In our case, this solution was not effective enough, because the pulps are very hydrophilic and absorb a lot of water, which causes significant water gradients all around the sample during drying, and consequently causes dimensional deformations. For this reason, we expose a new method of preparation, which does not require the addition of water to the mixture. To avoid water competition between the beet pulps and the starch grains, the extruded pulps were soaked in distilled water to ensure saturation with a mass ratio Water / BP = 2.5 [21]. The wet BP was mixed with starch powder. The mix was putted in an autoclave to dissolve the starch under water vapor pressure. After that, the samples were compacted using the traction machine INSTRON 8801 at 0.044 MPa. Finally, the mix was frozen and dried used the freeze dryer. The samples were completely dried in a climatic chamber at 50 °C and 10 % RH.

A cubic samples 10 x 10 x 10 cm with four the influence of beet pulp amount on the thermal and mechanical properties of the S-BP formulations with four different S/BP mass ratios (0.1, 0.2, 0.3 and 0.4) were prepared to study composite (see Fig. 4). The apparent density of the composite increases linearly from 270 kg/m³ to 360 kg/m³ when the mass ratio S/BP increases from 0.1 to 0.4 (see Fig. 5).

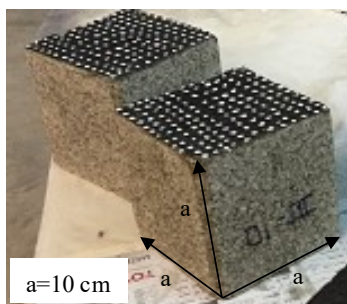


Fig. 4. The cubic samples used in the thermal and mechanical study.

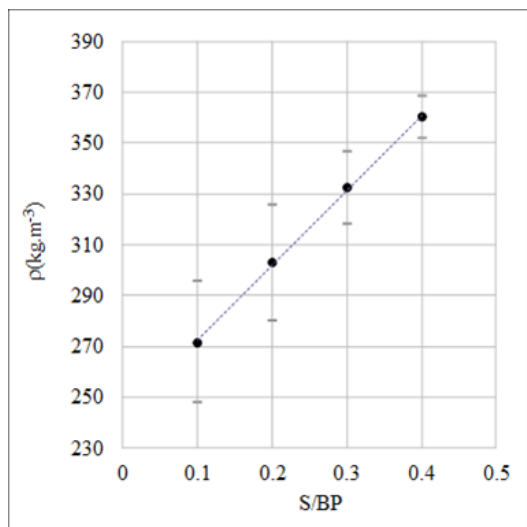


Fig. 5. Evolution of apparent density with the mass ratio S/BP.

2.3 Composite characterization

2.3.1 Porosity

The porosity is an essential characteristic for evaluating insulation properties. To evaluate the porosity of the BP composite, the bulk density was measured using equation (1):

$$\rho_{bulk} = \frac{\text{mass of the sample}}{\text{Volume of the container}} \quad (1)$$

The method used for the measurement of absolute density was the one already described by Bourdot et al., (2017). In summary, the pycnometer was filled with a given mass of BP and half its volume with cyclohexane, which is a non-polar solvent and does not affect BP composition and mass (M). The system was kept under reflux (see Figure 6) for 6 x 10 min. boiling then cooling cycles, during which air was released from the pulps voids and replaced by cyclohexane. During the 6th cycle the system is kept under Argon atmosphere to avoid humidity from being reabsorbed in BP. At ambient temperature (20

° C) the flask was filled up to the top and sealed with a dedicated glass top, allowing the excess solvent to overflow and avoiding the retention of bubbles, in order to have a constant volume inside the measuring system. The system was then weighed to a 10⁻³g accuracy. The absolute density was calculated according to this formula (2):

$$\rho_{abs} = \frac{M_1 \times \rho_{cyc}}{M_1 - (M_2 - M_3)} \quad (2)$$

where ρ_{abs} is the absolute density, ρ_{cyc} the density of cyclohexane, M_1 the mass of dry aggregates, M_2 the mass of the pycnometer (cyclohexane+ saturated aggregates), and M_3 the mass of the pycnometer (cyclohexane).

Care was taken to avoid the reflux of condensed humidity back in the mixture. Each measurement was carried out at least 3 times to be representative. The porosity was calculated using the following equation (3):

$$\Phi = 1 - \frac{\rho_{bulk}}{\rho_{abs}} \quad (3)$$

where Φ is the porosity of composite, ρ_{bulk} the bulk density in kg/m³ and ρ_{abs} the absolute density in kg/m³.

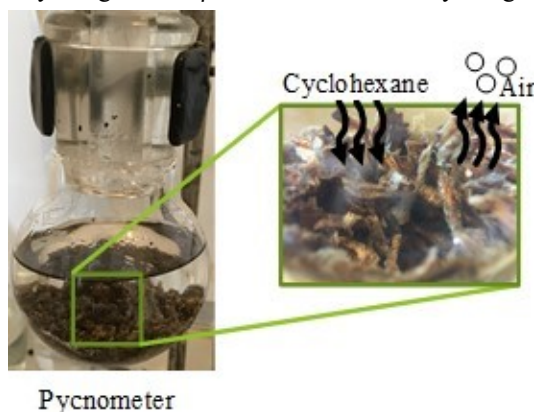


Fig. 6. The system of absolute density measurement

Four compositions were prepared with four BP- starch mass ratio S/BP 0.1, 0.2, 0.3 and 0.4. The samples were compacted under a pressure of 44 kPa. The samples were dried in a climatic chamber stabilized in a temperature at 50 °C and with humidity 10%.

2.3.2 Mechanical properties

Experimental procedure

For this test, a series of samples (12 samples 10 x 10 x 10 cm and 12 samples 16 x 4 x 4 cm) were prepared by varying the Starch/Beet pulp (S/BP) ratio (S/BP = 0.1, 0.2, 0.3 and 0.4). The samples were made by-the procedure in the paragraph 2.3.1.

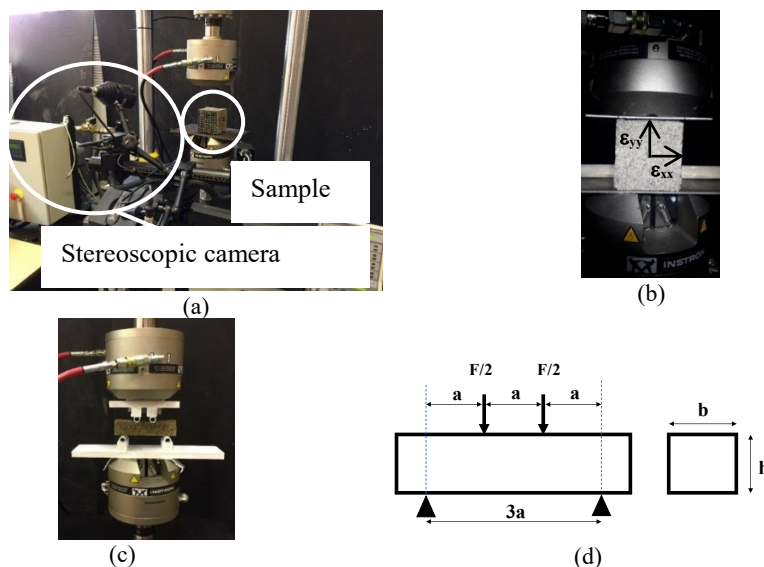


Fig. 7. (a) Aramis stereo optical system, (b) Compressive test, (c) and (d) Bending test

Compression test and bending test in four-point (see Figure 7) were recorded by using Instron 8801 machine with a constant rate of 0.1 mm/s for the bending test and 0.2 mm/s for the compression test. Compression and bending test were performed in the pressure direction. The tensile strength was determined by using the Navier equation (4):

$$\sigma_t = \frac{M_f \times y}{I} \quad (4)$$

with $M_f = (F/2) \times a$, F : the maximum load, a : the sample thickness, $y = h/2$, h : the sample height, I : the sample inertia $I = b \times h^3/12$ and b : the sample thickness (see Figure 7 d).

The transversal and longitudinal strain respectively (ϵ_{xx} , ϵ_{yy}) were determined using an Aramis optical system (see Figure 7 a). This system is a high performance optical system which determines both sample displacement and strain during loading through digital image correlation methods [8][7]. One ARAMIS stereo cameras were rigidly connected to measure the displacement fields and the 2D deformations from one pair of images corresponding to several instants of strain of samples. Before measuring, all the samples were placed at the same distance to the camera (34 cm) to obtain a sharpness image.

2.3.3 Thermal conductivity

A series of cubic samples (10 x 10 x 10 cm) were made to study the thermal behavior of the starch-Beet Pulp composite. Before the measurement, all of samples were dried in a climatic chamber at 50 °C and 10 %RH. The thermal conductivities were measured using an *Isomet*

2114 (Applied Precision) device by which applies a dynamic measurement method to reduce the measurement time in comparison with steady state measurement methods. This measurement analyzes the temperature response of the measured composite to heat flow impulses. The heat flow is produced by an electrical heating of resistor heating in direct contact with the tested sample. The thermal conductivity is evaluated by periodically recording the sampled temperature as function of the time. Built-in menu system on color graphic display and the alphanumeric keypad provides a good utilization of the device. The measurement reproducibility of the device was 3 % + 0.001 W/ m. K. the measurement was done at ambient temperature (20 °C) and 10 %RH. The thermal conductivity of the Starch-Beet Pulp was measured on three samples for each formula [19]. The heat capacity of the S/BP composite was measured using a C80 Calvet calorimeter from Setaram Instrumentation. In the calorimetric detector, the sample and the cell reference were completely surrounded by an array of thermocouple detectors allowing the heat transfer measurement including radiation, convection, and conduction.

3 Results and discussion

3.1 Density and porosity

The bulk density was measured for BP before and after extrusion. Table 1 shows the bulk and the absolute density of sugar beet pulp. In both cases the extruded BP appears denser, probably due to the strong compression during the extrusion process. During oven drying of the samples, microscopic cracks appear on the composite surface due to extensive shrinkage.

Table 1. Bulk and absolute density of different aggregates

Aggregates	ρ_{bulk} (kg/m ³)	ρ_{abs} (kg/m ³)	Porosity (%)
Fresh beet pulp	134 ± 6.7	911.61 ± 45.6	85.30
Extruded beet pulp	194 ± 7.9	1073.38 ± 53.7	81.93
Hemp shive 5 mm	135 ± 6.75	1271 ± 63.5	91.35
Hemp shive 20 mm	110 ± 5.5	1266 ± 63.3	89.34

As both the beet pulp and starch are highly hydrophilic, water gradients inside and anisotropy of the drying within the composite sample probably cause irregular shrinkage of the composite. Large dimensional variations were observed after classic oven drying at 100 °C, the sample underwent a (33 %) irregular retraction, which caused large millimeter size cracks. Drying under vacuum at 40 °C resulted in much more regular shapes, the composite retracted, there were no macroscopic cracks observed and the surface was flatter. When dried under the freeze dryer, the BP-starch composites release water more regularly, and consequently retract less, which results in rigid and light composite blocks. The blocks present a uniform volume shrinkage (8 %). Therefore, freeze drying provided the best results (see Figure 4).

Figure 8 shows the variation in the density of the completely dried composite material in climatic room at 50 °C and 10 %RH, as a function of the S/BP weight ratio. It can be observed that the composite material density increases linearly with S/BP weight ratio. The bulk density increases from 271.4 kg/m³ to 360 kg/m³, noting that the average density of the Cement- Beet Pulp concrete ranged between 570 kg/m³ and 770 kg/m³ [16]. The bulk density of the samples can be expressed as function as S/BP weight ratio between 0,1 and 0.4 by equation (5):

$$\rho = 295.42 \times \left(\frac{S}{BP}\right) + 242.8 \quad (5)$$

Results of density and porosity are shown in Figure 8. The absolute and bulk density increase with S/BP weight ratio. However, the porosity of the composite decreases logarithmically. Therefore, the sample having the lowest amount of starch (S/BP=0.1), has the higher porosity (79.75 %) and the lowest absolute density ($\rho_{\text{abs}} = 1222 \text{ kg/m}^3$). The total porosity was between 70.60% and 79.75 % and decreasing the S/BP weight ratio seems to increase the total porosity. The starch influence is consistent with results obtained by Rahim *et al.* [20] and Bourdot *et al* [7]. The presence of starch gel increases the bulk density and decreases the total porosity by filling the inter-particle space between pulp particles and filling or sealing the pores. Therefore, the composite would contain more or less accessible closed and open pores.

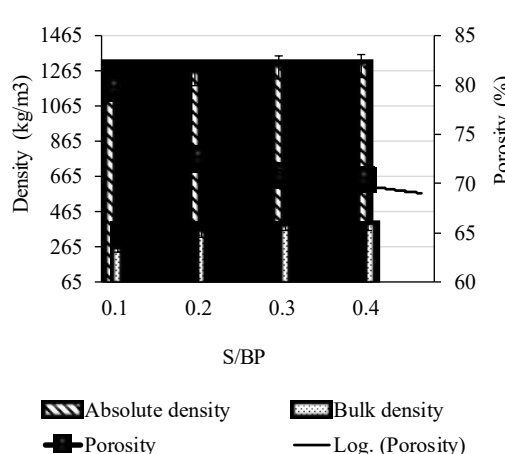


Fig. 8. The density and the porosity of BP composite

3.2 Mechanical properties

All mechanical properties shown in this section: tensile strength, compressive strength were measured on four S/BP weight ratio between 0.1 and 0.4. The compression test led to the failure of the sample. The compressive strength variation of the aforementioned samples is shown in Figure 9. the compression led to the failure of the samples. At 10 % of the strain of the sample for the samples of 10 x 10 x 10 cm with S/BP = 0.1, 0.2, 0.3 and 0.4, the corresponding stress is 0.20, 0.23, 0.24 and 0.32 MPa respectively. It is useful to indicate that the Starch-beet pulp composite is deformable and compressible. However, the sample can be crushed with load greater than load limit. The compressive strength was obtained for longitudinal strain ϵ between 22 % and 27 %. The compressive strength of the Starch- Beet Pulp increased from 0.37 MPa to 0.52 MPa. For the Starch-Hemp composites, the average compressive strength is ranged between 0.40 and 0.63 MPa [7–9]. It can be seen that the compressive strength of the Starch-Beet Pulp is comparable. It seems to indicate that the compressive strength of the Starch-Beet Pulp increases with increasing the starch amount. However, The influence of the variation in the density on the compressive strength of the Starch-Hemp composites is not significant [7–9].

The typical tensile stress-displacement curves for different S/BP weight ratio are represented in Figure 10 a. Figure 10 b shows that the tensile strength increases when the starch content increases. The average tensile strength

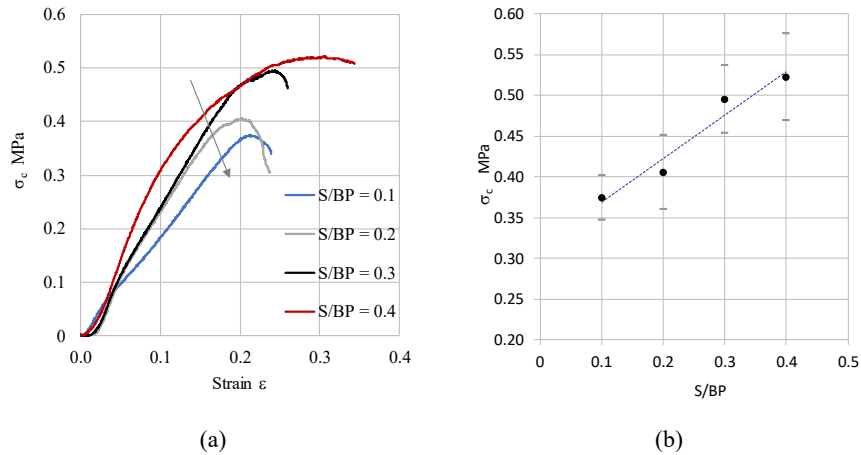


Fig. 9. (a) Compressive strain-stress curves for different S/BP weight ratios, (b) Compressive strength-S/BP weight ratio curves

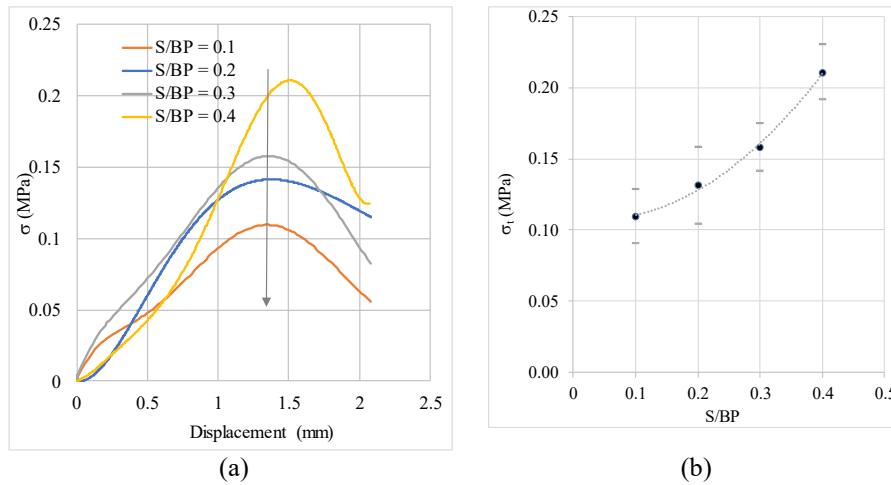


Figure 10. (a) Tensile stress-displacement curves for different S/BP weight ratios, (b) Flexural strength-S/BP weight ratio curves.

Table 2. Thermal properties of starch-beet pulp composites at 23 °C.

Mass ratio (S/BP)	λ (W. m ⁻¹ . K ⁻¹)	a (m ² .s ⁻¹)	b (J.K ⁻¹ . m-2. s-1/2)
0.1	0.069 ± 0.0006	1.76 ± 0.058E-07	165.5 ± 3.6
0.2	0.071 ± 0.0005	1.66 ± 0.0713E-07	175.2 ± 3.8
0.3	0.072 ± 0.0003	1.50 ± 0.052E-07	186.8 ± 3.5
0.4	0.075 ± 0.0002	1.47 ± 0.042E-07	197.5 ± 5.7

increases from 0.11 MPa to 0.21 MPa. These values are greater than those measured on the Starch-Hemp composites 0.08 - 0.11 MPa [7]. These results confirm (see section 3.1) that when the particles used is finer (Beet Pulp), the beet pulps are better coated by the starch binder during manufacturing of the material. The composite materials show better the tensile strength [23]. The tensile strength of the Starch-Beet Pulp composite (σ_t) can be expressed as function as the S/BP mass ratio by the Equation (6).

$$\sigma_t = 0.79 \times \left(\frac{S}{BP}\right)^2 - 0.06 \times \left(\frac{S}{BP}\right) + 0.11 \quad (6)$$

3.3 Thermal conductivity

Figure 11 shows the evolution of the thermal conductivity λ versus the S/BP weight ratios. The thermal conductivity of the Starch-Beet Pulp increases linearly from 0.0695

W/m. K to 0.0757 W/m. K when the S/BP weight ratio increases from 0.1 to 0.4.

Increasing the starch amount (S/BP), the composite porosity decreases and increases consequently the thermal conductivity of the composite material. According to RILEM classification, the Starch-Beet Pulp having thermal conductivity lower than 0.3 W/m. K, can be used as thermal insulation material. This value range of thermal conductivity is close to values for hemp-starch composites measured by authors [7].

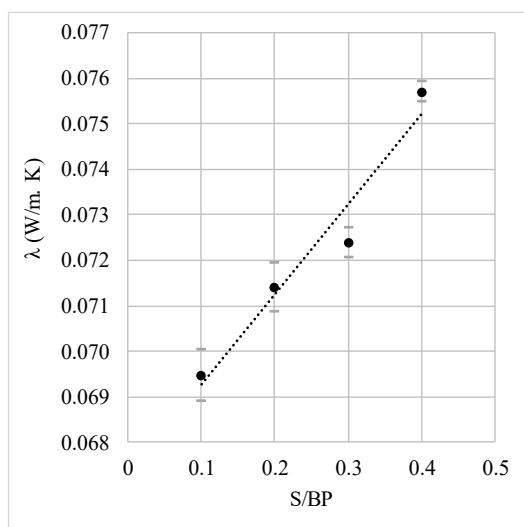


Fig. 11. Variation of the thermal conductivity as function of the S/BP weight ratio

Table 2 shows the thermal properties of starch-beet pulp composites for different mass ratio S/BP. The results show that the thermal conductivity λ increases linearly from 0.069 W. m-1. K-1 to 0.075 W. m-1. K-1 when the mass ratio S/BP increases from 0.1 to 0.4. the thermal conductivity can be expressed as function of the mass ratio S/BP according the equation (7). The increase of starch amount in S/BP composite decreases the composite porosity by filling the micropores, therefore the thermal conductivity increases.

The thermal diffusivity a is a physical quantity that characterizes the ability of a continuous material to transmit a temperature signal from one point to another of this material. It is calculated according to the equation (8) where ρ_{app} is the apparent density (kg.m^{-3}) and C_p is the heat capacity ($\text{J.K}^{-1}.\text{kg}^{-1}$). The thermal effusivity ($\text{J.K}^{-1}.\text{m}^{-2}.\text{s}^{-1/2}$) b represents the ability of the composite to exchange thermal energy with its environment. The thermal effusivity is given by the equation (9). The thermal effusivity and diffusivity is strongly related to the binder content (starch). Increasing the binder content in the composite promotes the thermal effusivity and decreases the thermal diffusivity. Thus, the starch content increases the composite ability to store heat. Thermal conductivity of starch-beet pulp composite is comparable to hemp-starch composite (between 0.063 W. m-1. K-1 and 0.100

W. m-1. K-1) [2,7]. But it is greater than others composite such as hemp-clay and cork concrete [8,11]. Thus, leads to a better thermal inertia compared to hemp-starch composite [2,7,8].

$$\lambda = 0.0197 \times \frac{S}{BP} + 0.0673 \quad (7)$$

$$a = \frac{\lambda}{\rho_{app} \times C_p} \quad (8)$$

$$b = \sqrt{\lambda \times \rho_{app} \times C_p} \quad (9)$$

4 Conclusions

In this paper, the physical properties of the extruded sugar beet pulp were studied (morphology, porosity, densities and particles size). Several methods and designs were used to elaborate and dry the BP-Starch composites. One of the difficulties in designing this agro-composite was the competition observed between the pulp and the binder for water absorption. To overcome this problem, the amount of water was decreased when mixing the two components, using the wet extruded pulp to swell the added dry native starch in an autoclave.

The properties of a new agro-composite based on BP and potato starch are presented. Different formulations were studied with BP-starch ratios varying between 0.1 and 0.4. The bulk density of the Starch-Beet Pulp composite depends of the S/BP weight ratio. It increases when the amount of the potato starch increases. The results show that the Starch- Beet Pulp is heavier than Starch-Hemp composites which is varying generally between 160 kg/m^3 and 180 kg/m^3 However, it is much lighter than Cement- Beet Pulp concrete which ranged between 570 kg/m^3 and 770 kg/m^3 .

The average of the tensile strength evolved from 0.11 MPa to 0.21 MPa when the S/BP weight ratio increased from 0.1 to 0.4. These values were greater than those measured on the Starch- Hemp composites 0.03 MPa 0.13 MPa and were comparable with those obtained with the Starch- Hemp composites. The difference can be explained by the better adhesion that the finer particles used (Beet Pulp) provide to the composites and consequently increased elasticity.

Regarding the thermal performance, the results shows that the Starch- Beet Pulp composite have a low thermal conductivity to around 0.070 W/m. K. The thermal conductivity increases when the S/BP weight ratio increases. Finally, the BP-starch composite S/BP 0.4 can be considered as the optimal composition in terms of mechanical properties. However, the S/BP 0.1 presents the optimal composition for thermal properties.

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