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► **To cite this version:**

Boussad Abbès, Catherine Lacoste, Christophe Bliard, Chadi Maalouf, Florica Simescu-Lazar, et al.. Novel Extruded Starch-Beet Pulp Composites for Packaging Foams. *Materials*, 2020, 13 (7), pp.1571-10.3390/ma13071571 . hal-02882047

**HAL Id: hal-02882047**

**<https://hal.science/hal-02882047>**

Submitted on 19 Nov 2020

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# Novel Extruded Starch-Beet Pulp Composites for Packaging Foams

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Received: date; Accepted: date; Published: date

**Abstract:** This article deals with the elaboration and the characterization of a novel biobased composite made of beet pulp and potato starch for packaging applications as cushion foams. The influence of the starch amount on composite characteristics was studied. A twin-screw extruder was used to elaborate composite foams. SEM observations of these materials were conducted, and thermomechanical properties were studied in terms of thermal transitions (TGA, DSC) and viscoelastic properties (DMA). The influence of relative humidity content on viscoelastic properties was studied as a function of frequency. Test results show that the composite structures are homogeneously mixed. The sponge-like structure of the beet-pulp disappears indicating a good compatibility between the two mixed constituents. The DSC curve of starch-beet pulp foam in shows a single thermal transition at 153.6°C indicating the thermal homogeneity of the obtained composite material. The density value of starch-beet pulp foam is higher than conventional foams, but this can be optimized by adjusting the technological parameters of the extruder. The viscoelastic properties of the developed materials depend on the relative humidity.

**Keywords:** starch; beet pulp; extrusion; biobased composite

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## 1. Introduction

The packaging industry is looking for materials with specific properties (strength, lightness, impact protection, etc.) that can be easily disposed of after use while being environmentally friendly. At the same time, consumers have developed an environmental conscience and have now become aware of the need to use biodegradable packaging [1]. Packaging has thus become a societal issue. Research teams embraced this trend and numerous studies related to biopolymers have been conducted [2] to reduce the carbon footprint and pollution risks caused by using conventional polymers [3-6]. The NF-EN-13432 standard defines requirements for qualifying packaging as "biodegradable and recoverable in industrial composting" [7,8].

With this in mind, we propose an original combination of starch and beet pulp to create loose fill wedging material for packaging. Today, these chips are mainly made of expanded polystyrene whose cellular structure (foam) has a low density and interesting impact resistance and thermal insulation characteristics [2]. These packages generate large quantities of waste; they are generally disposed of quickly and are expected to take several years or even centuries to be degraded. Expanded starch (from different sources: potato, rice, manioc, wheat, corn...) has already been studied in all its forms [9,10] and each of these starches has its own characteristics: viscosity, molecular mass profile and amylose / amylopectin ratio [11,12]. Potatoes as well as cassava starch

has been shown to generate foams with lower densities than those from cereals such and corn [13]. The production of a biopolymer composite based on a starch-beet pulp mix has already been successfully described [14]. A similar composition is studied here for a new application as material for cushioning goods during the transport of parcels.

The sugar manufacturing process, derived from sugar beet, generates large quantities of solid pulp by-product at the end of the sugar extraction process. Dried beet pulp pellets are mainly used for animal feed or biogas generation [15-17]. The extruded pulp is mainly composed of pectin, cellulose and hemicellulose [18].

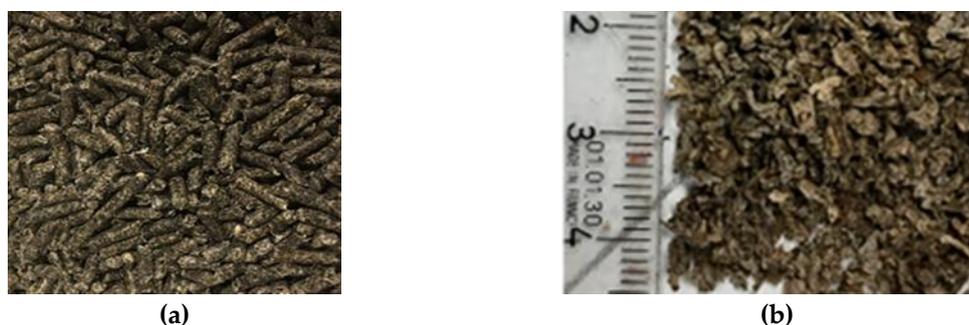
After having presented the specific characteristics of the two components used, the formulation chosen to produce these starch-beet pulp foams will be specified as well as the methods for obtaining the material with a study of the properties of the starch-beets composite. The physical and mechanical characteristics of the obtained composite will then be presented.

## 2. Materials and Methods

### 2.1. Materials

#### 2.1.1. Extruded Beet Pulp

The extruded beet pulp (BP) pellets were provided by Cristal Union factory (Pomacle, France) with 8–10 mm diameter and 18% humidity (Figure 1) during the 2017- 2018 sugar beet campaign. To ensure a proper conservation, the pellets were kept in the laboratory at  $-20^{\circ}\text{C}$  until use. Before use, the extruded BP were immersed in 3.5 times their weight of water for two hours to ensure a complete swelling and then dried at  $50^{\circ}\text{C}$  for two days in a pulsed air tower.



**Figure 1.** (a) Extruded beet pulp pellets and (b) Dried extruded beet pulp.

#### 2.1.2. Potato Starch

Potato starch (15% humidity) was purchased from Roquette (Lestrem, France). Potato starch was chosen among other botanical sources because of its high polymerization degree and high viscous properties providing a good mechanical property for the Starch-Beet Pulp (SBP) composite. In SBP composite starch acts as a binder to stick the particles together and ensure the transmission of shear forces between the fibers.

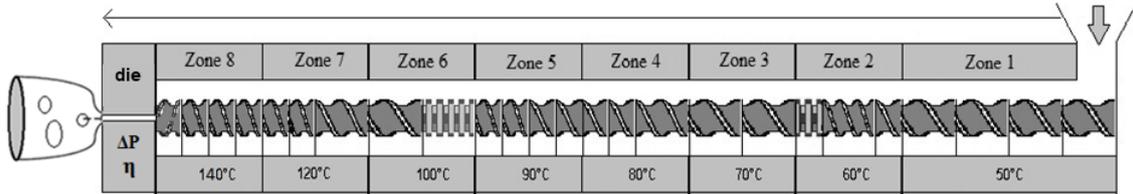
#### 2.1.3. Composite formulation

Potato starch and potato SBP composite foams were prepared as follows. To 10 parts of dried swollen pulp obtained from extruded beet pulp was added 3 parts of water and 10 parts of a 7/5 starch-glycerol mix previously dried for 2 hours at  $120^{\circ}\text{C}$  in an oven. The obtained admixture was mechanically stirred to homogenize the distribution of the different constituents in the SBP blend.

### 2.2. Methods

### 2.2.1. Extrusion setup

The SBP mix were compounded using a Clextral BC21 co-rotating twin-screw extruder machine, with a mixing and a suited transport screw elements configuration (Figure 2). The barrel temperature was set from 70°C to above 120°C, and the compound was extruded through a ribbon die geometry. After extrusion, the ribbons were cut into 15 cm length and 2 cm width wise strips, identified and conditioned for one week at 23±2°C and 50±10% relative humidity according to NF EN ISO 7214 norm prior to characterization.



**Figure 2.** Co-rotating twin-screw extruder machine with a mixing and a suited transport screw elements configuration.

### 2.2.2. Foam density

The bulk density was calculated as the mass of an extruded SBP (ESBP) ribbon divided by its geometric external measured volume. The absolute density was determined using the Bourdot pycnometer method [19] particularly well adapted for structurally complex multi-hollow fibrous biological material. In this method, the pycnometer flask is filled with a known mass of dried pulp (approx. 20 g) and half of its volume with cyclohexane, which is a non-polar solvent and does not affect the composition and mass of the pulp. The system is subjected to six cycles of boiling (30 min) and cooling (10 min); during these cycles, air escape from the pulp cells and allowing cyclohexane to occupy the pore spaces; and during the sixth cycle, the system is kept under an argon atmosphere to avoid the reentry of humidity. At the end of the cycles, the pycnometer is filled up with cyclohexane at room temperature (20°C) and plugged with the stopper. The system (approx. 150 g) is then weighed with an accuracy of 10<sup>-3</sup> g. The absolute density is calculated using the following equation:

$$\rho_{abs} = \frac{m_1 \times \rho_{cyc}}{m_1 - (m_2 - m_3)} \quad (1)$$

where  $\rho_{abs}$  is the absolute density (kg·m<sup>-3</sup>),  $\rho_{cyc}$  is the density of cyclohexane (kg·m<sup>-3</sup>),  $m_1$  is the dry mass of SBP aggregates,  $m_2$  is the mass of the pycnometer filled with cyclohexane and saturated aggregates, and  $m_3$  is the mass of pycnometer and cyclohexane.

### 2.2.3. Microscopy Observations

Scanning electron microscopy (SEM) observations were carried out on a 15kV JEOL/JSM 6460LA. All samples were dried at 60 °C during 48 h in a vacuum desiccator before being metallized.

### 2.2.4. Thermogravimetric Analysis (TGA) Procedure

Experiments were conducted using a TGA (NETZSCH - TG 209 F3 Tarsus) in order to assess the thermal stability and the moisture content of potato starch and starch-beet pulp composite foams. The measurements were carried out on 10–15 mg samples put in an aluminum pan and heated from 25 °C to 600 °C with a heating rate of 10°C/min in a nitrogen atmosphere.

### 2.2.5. Differential Scanning Calorimetry (DSC) Procedure

The thermal characteristics of potato starch and starch-beet pulp composite foams were determined by using a DSC (NETZSCH – DSC 204 F1 Phoenix), after 48 hours conditioning at a

temperature of 23°C and 65 % relative humidity. Aluminum sealed pans were used to test in triplicate samples of 5–40 mg. The heat flow was measured as function of the temperature and time. The samples were heated from -80°C to 200°C at a heating rate of 20°C/min. An empty DSC pan was used as a reference to balance the heat capacity of the sample pan. The results were analyzed with Proteus V4.8.5 NETZSCH software.

#### 2.2.6. Dynamic Mechanical Analysis (DMA) Procedure

Dynamic Mechanical Analysis was carried out in a DMA Q800 (TA Instruments), with DMA-RH accessory for humidity and temperature control. This allows to determine the mechanical properties of the samples under controlled humidity and temperature conditions.

To compare the viscoelastic properties of the different foams, creep/recovery tests were performed using the compression clamp kit which is suitable for low to moderate modulus materials such as foams. In this mode, the sample is placed on a fixed flat surface of 40 mm diameter and an oscillating plate applies force. The samples were cut into 1.22 cm<sup>2</sup> circular slices of 5 mm thickness. The samples were preloaded with a force of 0.01 N and then assigned a constant stress of 10 kPa for 5 min at a constant temperature of 23±2°C and variable relative humidity conditions ranging from 30 to 52%. After 5 min, the stress was removed, and the samples undergo 15 min recovery. The creep compliance of the tested materials was measured.

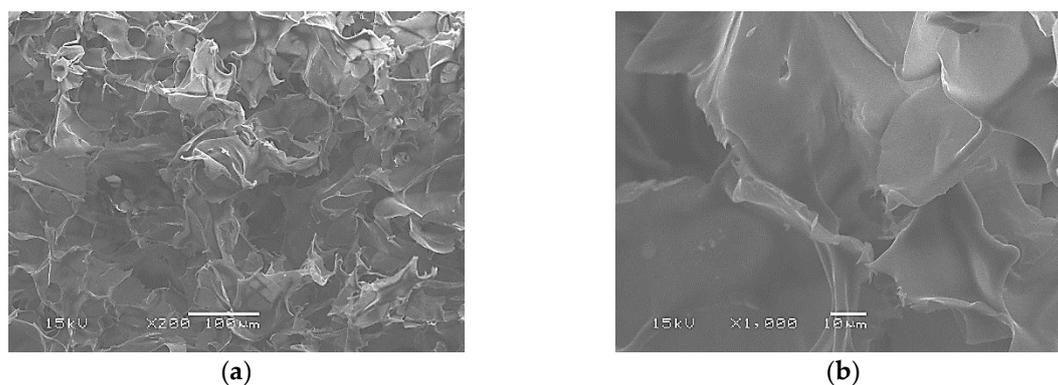
To assess the viscoelastic properties as a function of frequency, we have also performed tests in the multi-frequency mode (0.1 Hz - 100 Hz), with a constant oscillation stress amplitude of 10 kPa at a constant temperature of 23±2°C and variable relative humidity conditions ranging from 13 to 54%. The storage modulus and loss modulus of the materials were measured.

### 3. Results and discussion

#### 3.1. Microscopic Structure of the Extruded Foams

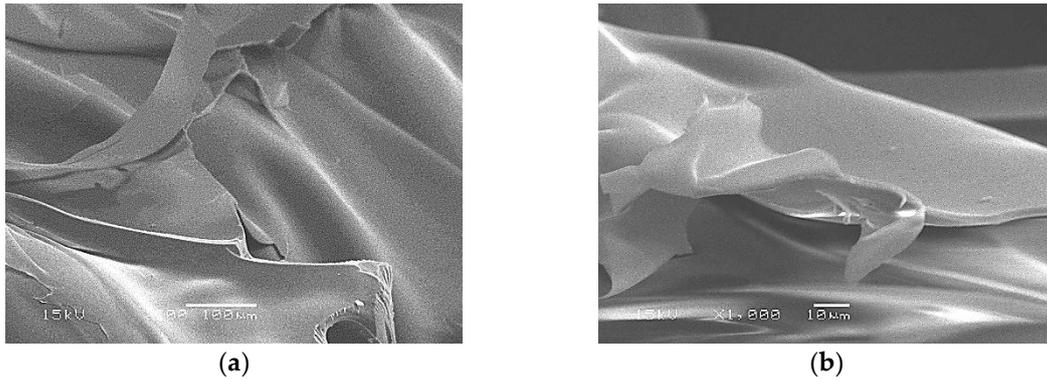
The following SEM images allow to visualize the microscopic structure of the raw materials and their physical transformation after the extrusion process.

Figure 3 shows the dry lyophilized starch raw material with two different magnifications. These SEM micrographs are characteristic of a foamed material with a homogeneous distribution of the cells. The cells size ranges from 50 to 100 micrometers.



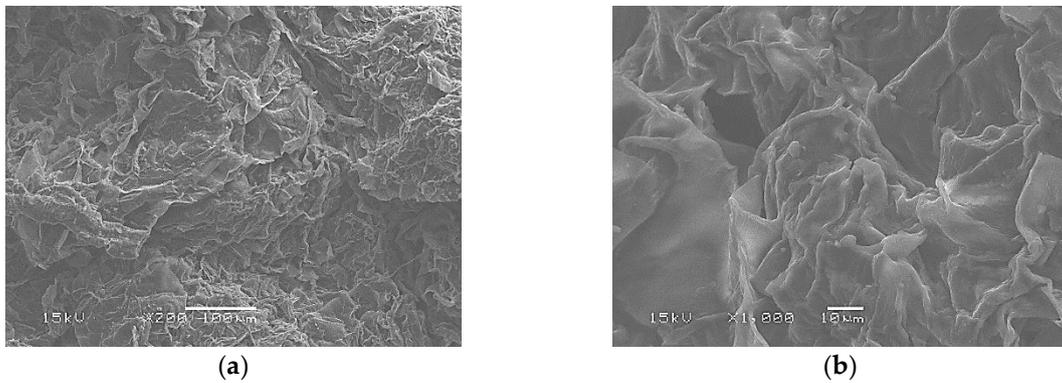
**Figure 3.** Dry lyophilized starch raw material SEM micrographs: (a) ×200; (b) ×1000.

After plasticization of starch, its cell structure observed in Figure 3 disappears leaving a corrugated sheet structure as shown in Figure 4. This corrugated sheet structure is characteristic of a plasticization obtained by the mechanical action of the extrusion screw in the presence of water and glycerol.



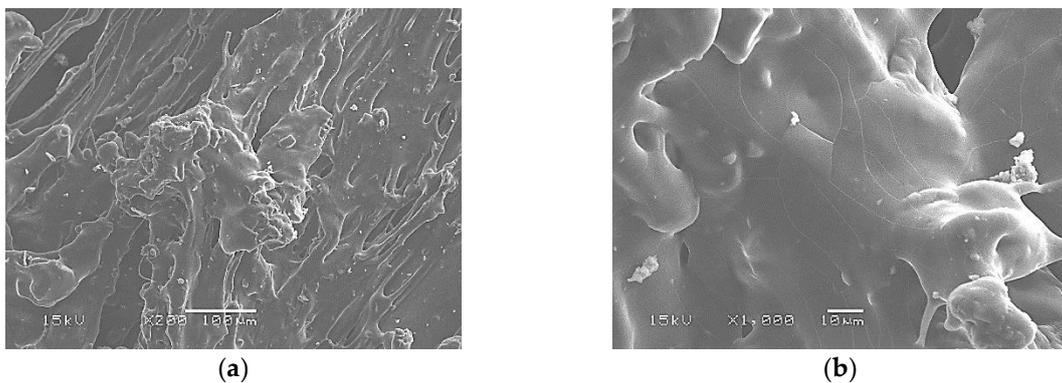
**Figure 4.** Plasticized starch SEM micrographs: (a)  $\times 200$ ; (b)  $\times 1000$ .

Figure 5 shows the native homogeneous sponge-like structure of beet pulp. This structure results from the destructuring undergone by the beet during the industrial sugar extraction. The vacuoles size ranges from 25 to 50 micrometers. The typical cellulose rectilinear fibrous structure is not observed in the micrographs.



**Figure 5.** Beet pulp raw material scanning electron micrographs: (a)  $\times 200$ ; (b)  $\times 1000$ .

Figure 6 shows the potato starch-beet pulp composite foam structure, where the previous structures are homogeneously mixed. The sponge-like structure of the beet-pulp disappear indicating the good compatibility between the two mixed constituents.



**Figure 6.** Potato starch-beet pulp composite foam SEM micrographs: (a)  $\times 200$ ; (b)  $\times 1000$ .

### 3.2. Density Analysis

Table 1 gives the bulk and absolute density values for extruded starch beet pulp foam (ESBP) compared to the ones of native starch, and beet pulp. The density value of starch-beet pulp foam is still higher than conventional foams. This poor value is the result of preliminary tests on the

feasibility of extrusion with no special focus on the foam characteristics. The density of the obtained foam could be largely optimized by adjusting the technological parameters at the exit of the extrusion die since the mixture tended to collapse. A physical or a chemical agent such as an expanding gas (CO<sub>2</sub> for example) can also be added in the extrusion process to improve the foam expansion and decrease the density value.

**Table 1.** Material densities.

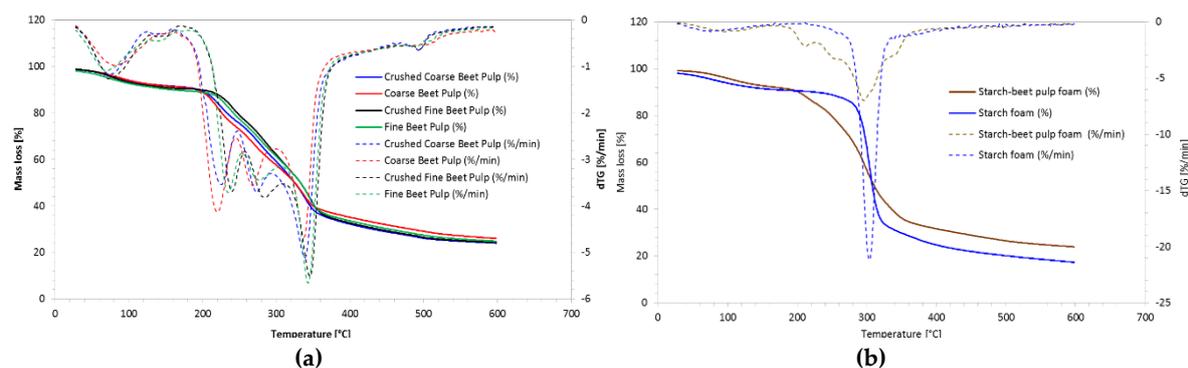
ESBP foam bulk density (g/cm <sup>3</sup> )	Raw Starch bulk density (g/cm <sup>3</sup> )	BP bulk density <sup>a</sup> (g/cm <sup>3</sup> )
0.686 +/- 0.05	0.510 +/- 0.05	0.194 +/- 0.007
SBP foam abs. density (g/cm <sup>3</sup> )	Raw Starch abs. density (g/cm <sup>3</sup> )	BP abs. density <sup>a</sup> (g/cm <sup>3</sup> )
1.470 +/- 0.005	1.510 +/- 0.005	1.073 +/- 0.005

<sup>a</sup>Ref. [14]

### 3.3. Thermogravimetric Analysis (TGA) Results

TGA thermograms of beet pulps are given in Figure 6(a) showing three consecutive thermal zones corresponding to water loss (100°C – 200°C), followed by the substrate decomposition (200°C – 350°C) and finally the material carbonization (> 350°). The water loss of the beet pulp is about 10% at 100°C. The beet pulp undergoes several decompositions between 200°C and 350°C due to the fragility of its constituents such as the pectin and hemicellulosic fractions. The native beet pulp loses mass earlier because it has not undergone any preliminary degradation by mechanical energy. This is clearly shown by the temperature shift of about 20°C observed between the mass loss curves and confirmed by derivative peaks shift (dotted lines).

Figure 6(b) shows a typical TGA curve obtained for starch foam with slow water loss between 100°C and 200°C, followed by sudden decomposition at 300°C and finally the material carbonization (> 300°). The TGA curve obtained for the composite starch-beet pulp foam corresponds to the superposition of the beet pulp and starch foam thermograms without mutual influence between the two materials.



**Figure 6.** TGA thermograms: (a) beet pulps – (b) starch and starch-beet pulp foams.

### 3.4. Differential Scanning Calorimetry (DSC) Results

Figures 7 shows the DSC curves of beet pulp, starch and the composite resulting from their mixture. The peak temperatures corresponding to the thermal transition of beet pulp and starch are approximately the same: 123.5°C for starch and 125.6°C for beet pulp. The DSC curve of starch-beet pulp foam in Figure 7(b) shows a single thermal transition at 153.6°C indicating the thermal homogeneity of the obtained composite material.

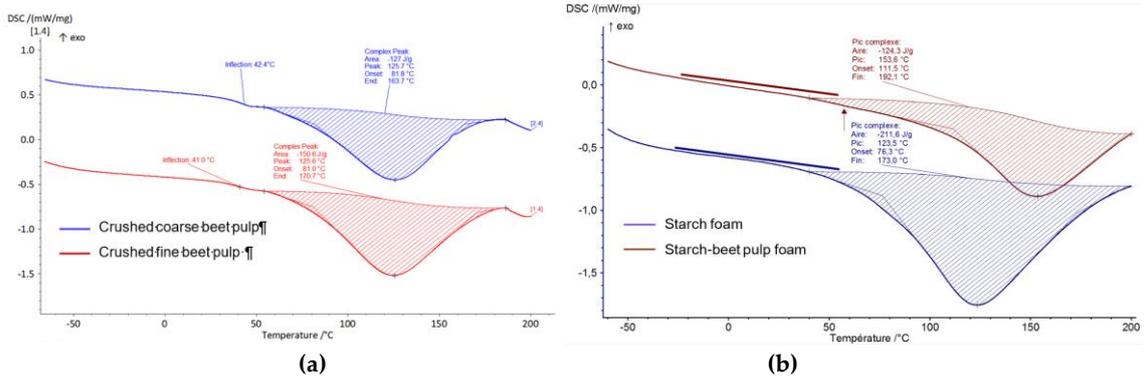


Figure 7. DSC thermograms: (a) beet pulp – (b) starch and starch-beet pulp foams.

### 3.5. Dynamic Mechanical Analysis (DMA) Results

#### 3.5.1. Storage and loss moduli

Figure 8 show the storage ( $G'$ ) and loss ( $G''$ ) moduli of starch-beet pulp foams at different RH conditions. The storage modulus represents the energy stored in the elastic structure of the sample. It is higher than the loss modulus the material can be regarded as mainly elastic. The loss modulus represents the viscous part, or the amount of energy dissipated in the sample. We can notice that the effect of relative humidity of the composite material on the viscoelastic properties is small. The ratio of the moduli ( $G''/G'$ ) is defined as  $\tan(\delta)$ , and indicates the relative degree of energy dissipation or damping of the material. As shown in Figure 9, this ratio varies between 0.1 and 0.2 in the range of the tested RH.

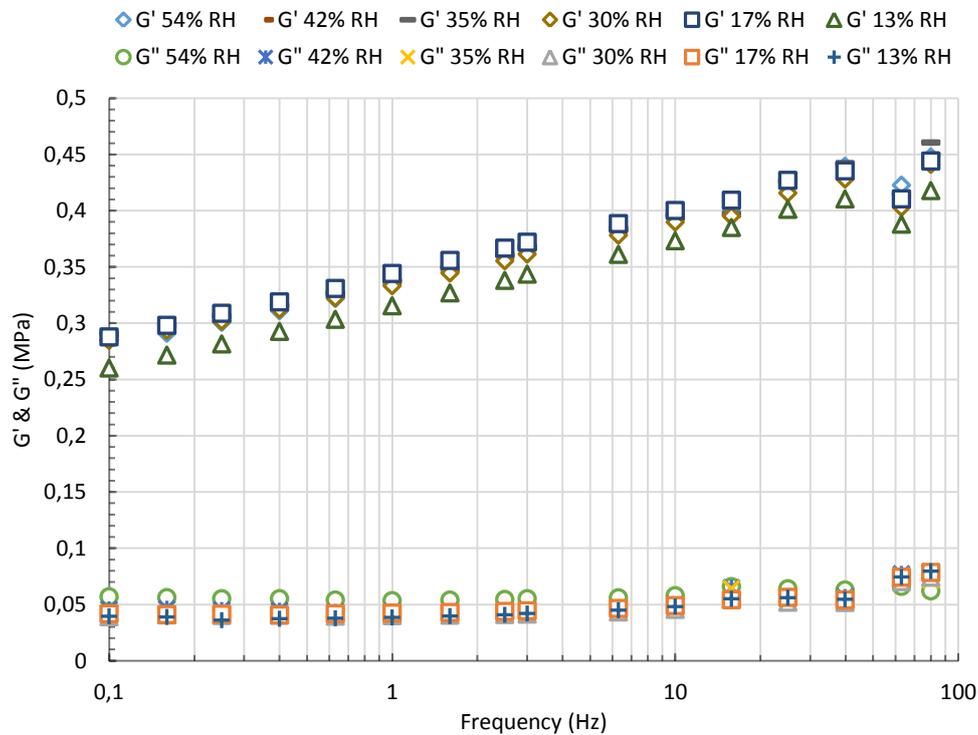


Figure 8. Storage modulus ( $G'$ ) and Loss modulus ( $G''$ ) of starch-beet pulp foams at different RH conditions.

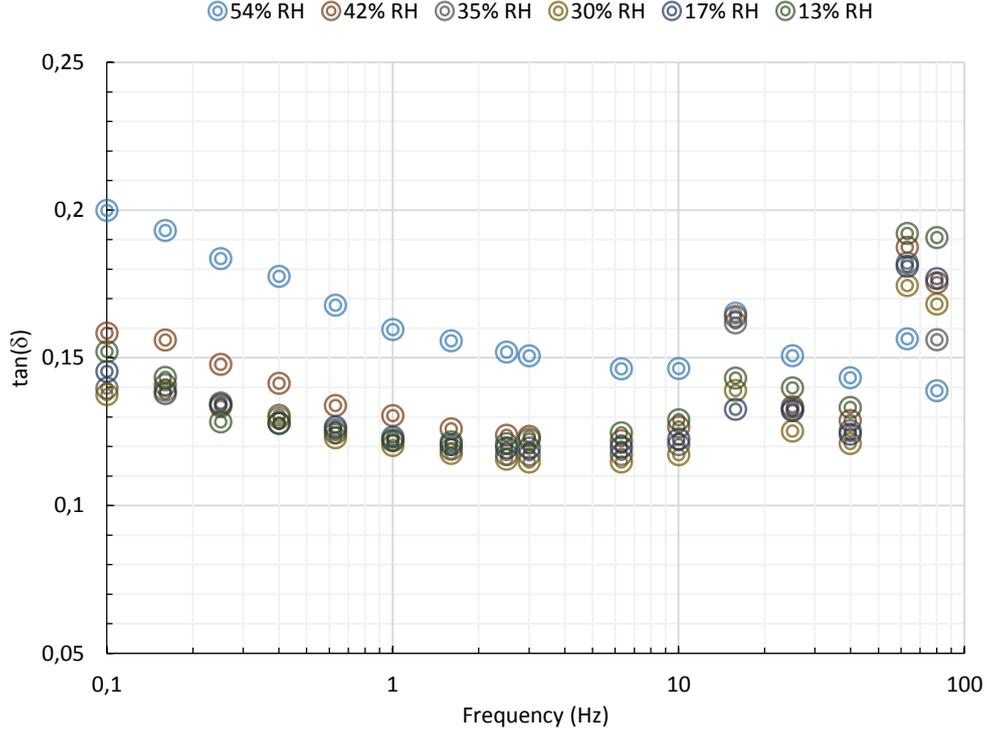


Figure 9.  $\tan(\delta)$  of starch-beet pulp foams at different RH conditions.

### 3.5.2. Creep-Recovery Response

Figure 10 shows the creep-recovery curves of starch-beet pulp foams at different RH conditions. When the relative humidity increases, the composite foam is stiffer allowing less deformation under stress. The creep-recovery response of the foams can be modelled using the viscoelastic generalized Kelvin-Voigt model given by the following equations for creep ( $\varepsilon_c(t)$ ) and recovery ( $\varepsilon_r(t)$ ) respectively:

$$\varepsilon_c(t) = \sigma_0 \left( J_0 + \frac{t}{\eta_0} + \sum_{i=1}^n J_i (1 - e^{-t/\theta_i}) \right) \quad (2)$$

$$\varepsilon_r(t) = \sigma_0 \left( J_0 - \sum_{i=1}^n J_i e^{-t/\theta_i} (1 - e^{\tau/\theta_i}) \right), \quad t > \tau \quad (3)$$

where  $t$  is the time,  $\sigma_0$  is the creep stress,  $\eta_0$  is the viscosity,  $J_i$  are the compliances,  $\theta_i$  are the retardation times and  $\tau$  is the creep loading time.

Table 1. Generalized Kelvin-Voigt model parameters.

RH (%)	$J_0$ (MPa <sup>-1</sup> )	$\eta_0$ (MPa.min)	$J_1$ (MPa <sup>-1</sup> )	$\theta_1$ (min)	$J_2$ (MPa <sup>-1</sup> )	$\theta_2$ (min)
30	2499.4	1.04	3671.6	0.00076	1366.9	1.085
41	2299.0	1.05	3751.6	0.00075	1115.4	0.732
52	1200.0	1.07	3881.4	0.00075	1291.0	0.674

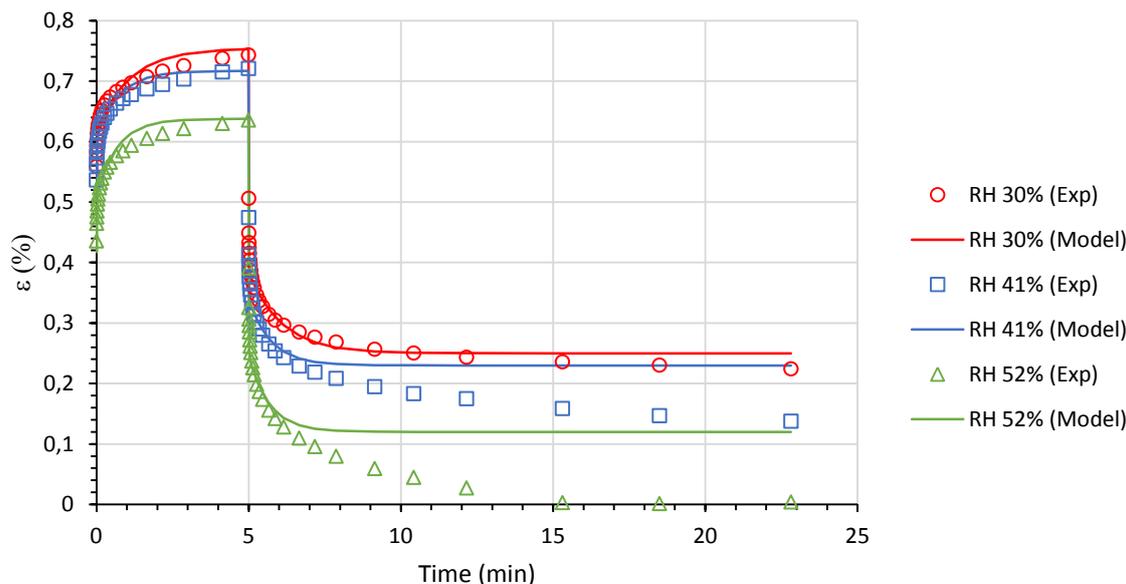


Figure 10. Creep-recovery curves of starch-beet pulp foams at different RH conditions.

#### 4. Conclusions

The extrusion process of a BP / plasticized starch compound produces a homogeneous foamy material. Microscopic analysis shows a good compatibility of the two substrates. Density measurements show that the bulk values for extruded starch beet pulp foam are still higher than conventional foams. These results were obtained with a non-optimized process and can be improved by adjusting the technological parameters at the exit of the extrusion die since the mixture tended to collapse after extrusion. A physical or a chemical agent such as an expanding gas (CO<sub>2</sub> for example) can also be added in the extrusion process to improve the foam expansion and decrease the value of the density. Thermogravimetric data correspond to the addition of the thermogravimetric characteristics of the two components while DSC shows a shift of the endotherm towards a higher temperature as compared to the one observed for each component. Dynamic Mechanical Analysis show a mild effect of the relative humidity on the ESBP mechanical properties.

**Author Contributions:** B.A., G.P., C.B. and C.M. discussed the concept of the novel composite. F.B. realized the bibliographic review and paper editing. B.A. wrote the original draft. C.L., C.B. and C.M. elaborated the composite material. C.L. and F.S.-L. conducted the experimental observations and measurements (DSC, TGA, DMA, SEM). All authors interpreted and discussed the experimental results and data. All authors of the article provided substantial comments.

**Funding:** This research received no external funding.

**Conflicts of Interest:** The authors declare no conflict of interest.

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