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Thermal stability and mechanical behavior of technical bamboo fibers/bio-based polyamide composites

Louise Lods¹ · Tutea Richmond¹ · Jany Dandurand¹ · Eric Dantras¹  · Colette Lacabanne¹ · Jean-Michel Durand² · Edouard Sherwood² · Gilles Hochstetter³ · Philippe Pontoins⁴

Abstract

The objective of this research is to tailor composites with continuous bamboo fibers as reinforcement of a bio-based polyamide (RilsanXD). Using mild chemical and mechanical treatment, technical bamboo fibers were extracted from *Phyllostachys viridiglaucescens*. They have an average diameter of 397 μm . Their average tensile strength is 323 MPa, and their average Young modulus is 26 GPa. Single ply bamboo fibers/polyamide composites were processed. Gravimetric analyses show that polyamide allows mild processing conditions at 200 °C, therefore permitting to make composites without degrading the fibrous reinforcement. Composites composition was determined from peak analyses of the mass derivative curve associated with bamboo situated at 330 °C. Composites with 60 m % of bamboo fibers were prepared and analyzed. SEM images observation of cryo-cuts shows the absence of voids between technical fibers and polyamide matrix. The cohesion in shear was tested by dynamic mechanical analyses. Comparative data recorded on the bio-based polyamide and the 60/40 bamboo fibers/polyamide composite show a significant improvement of the shear glassy modulus which is multiplied by 1.6 at 20 °C. This result is consistent with the continuity of matter between bamboo fibers and polyamide observed by SEM. This effect may be due to a dense network of static hydrogen bonds.

Keywords Thermogravimetric analysis · Dynamic mechanical analysis · Bamboo fibers/polyamide composites · Technical bamboo fibers · Glassy modulus · Rubbery modulus

Introduction

In recent years, the use of natural fibers as reinforcement in polymer-based composites has drawn much interest. The growing awareness of environmental problems, especially the depletion of fossil fuels and climate change, makes natural fibers emerging.

Glass and carbon are the most common man-made fibers to reinforce polymer composites with a great interest due

to their good mechanical properties. Nevertheless, they are non-renewable materials and their production requires high energy consumption. Natural plant fibers such as jute, coir, flax, hemp, bamboo and sisal have many advantages over inorganic fillers and petroleum-based synthetic fibers [1–5]. These natural fibers are used due to their bio-based character, high specific mechanical properties [6], low density, sustainability, good thermal insulation, biodegradability, low price and durability. As a viable green alternative for conventional synthetic fibers, especially glass fibers, natural plant fibers are gaining popularity in automotive [7, 8], aircraft and structural construction applications [9]. Several studies involving life cycle assessment (LCA) conclude that natural fibers are likely to be environmentally superior to synthetic fibers in each phase of the entire life cycle of the composite materials.

Among all the vegetal fibers, bamboo is an attractive solution due to its rapid growth rate and its availability [10]. The total bamboo forest area in the world reaches 22 million hectares. Moreover, bamboo is one of the fastest renewable

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plants with a maturity cycle of 3–4 years. Bamboo constitutes a group of perennial evergreens in the grass family Poaceae, subfamily Bambusoideae. More than 1400 bamboo species from 115 genera are found in diverse climate and world regions [11]. The tribes of the woody bamboos, Bambuseae and Arundinaria, and in particular the «giant bamboos» are of special interest for composite applications. Bamboo genera that have been identified as a potential source for fibers extraction due to their anatomical characteristics and their common use in traditional construction are: Bambusa, Dendrocalamus [12, 13], Gigantochloa [14], Guadua [15] and Phyllostachys.

An abundant literature is available on bamboo fibers for reinforcing polymeric matrices [16–18]. First articles were focused on thermoset matrices like polyester [19–21] or epoxy [22–24] with low processing temperature compatible with the thermal stability of natural fibers. Thermoplastics like PLA [25–27], PVC and polyamides [28] are also used as matrix.

The most common polyamides—polyamide 6 6 and polyamide 6—require processing temperatures that are not compatible with the thermal stability of natural fibers. Moreover, their relatively high water sensitivity is an inconvenience to tailor green composites. Indeed, the sorption of water molecules leads to a plasticization of the glass transition and the decrease of mechanical properties. By increasing the length of aliphatic sequences of polyamides, the melting temperature decreases and the processing temperature becomes compatible with natural fibers. Moreover, the water uptake is limited. For improving the mechanical modulus, aromatic rings may be inserted in the hydrocarbon sequence. Following those lines, PA meta-xylylene diamine 10 (RilsanXD) was chosen as matrix of continuous bamboo fibers composites.

In this work, composites were prepared from continuous bamboo fibers reinforced RilsanXD [29, 30]. This polyamide is synthesized from castor oil extracted from the seeds of *Ricinus communis* which is a tropical plant. Therefore, this thermoplastic is a “green” and bio-based polymer. Moreover, this thermoplastic matrix composite allows mechanical downcycling (recycling).

This article presents the tailoring and fabrication of bamboo fibers reinforced composites. Particular challenges must be overcome in order to implement the bamboo fibers composites in technical applications. Key parameters, i.e., the thermal degradation of fibers, the optimum thermal processing parameters and the variability in fibers mechanical properties, are investigated. The first step deals with the characterization of the extracted technical fibers; their thermal and mechanical behavior is studied by thermogravimetric analysis and tensile test. Then, the investigations focus mainly on the matrix, the processing of composites and the increase of mechanical properties of composite due to fibers.

Experimental

Materials

Continuous bamboo fibers

The selected bamboo species for this study was *Phyllostachys viridiglaucescens*. The habitat of *Phyllostachys* species is a warm-temperate climate. They are also known to have a good hardness against the cold, which makes them suitable to grow in Europe [31]. Bamboo culms were harvested in the southwest of France (trade by Cobratex/France) with an annual temperature of 16 °C, an annual average precipitation of 800 mm and a relative humidity of 80%. All bamboo culms studied are 3 years old because the research on fibers chemical composition showed that the lignin content is still changing during the first 3 years of growth, though this evolution does not modify the mechanical properties [32]. Technical fibers, which will be referred as fibers in this article, were extracted from rewetted bamboo culms using a novel hybrid process between chemical and mechanical approach (neither high temperature nor steam explosion were used during the extraction) [33, 34]. This process makes it possible to obtain fibers from sticks (15 cm longitudinal \times 0.5 cm tangential \times 2–3 cm radial) using chemical treatment with an alkaline solution at 1% NaOH [35–39] and mechanical treatment (by rolling and brooming bamboo strip with a rolling machine). The maximum length of the extracted fibers could be the internode length. For a 3-year-old bamboo, the internode length is 25–30 cm. Figure 1 shows extracted bamboo fibers using hybrid method.

Bio-based polyamide matrix

Polyamide meta-xylylene diamine 10 is a new semicrystalline bio-based thermoplastic synthesized by Arkema (France) under the trade name RilsanXD. The chemical structure of this material is recalled on Fig. 2. Its water uptake at room temperature for a humidity rate of 50% is $1.3 \pm 0.2\%$ [40]. Due to its low melting point at 189 °C, it is well suited for constituting the matrix of continuous bamboo fibers composites. It was provided as pellets for the processing of composites.

It is important to note that DSC studies show that the crystallinity of polyamide is not modified in composites.

Methods

Scanning electron microscopy

Bamboo fibers and bamboo/polyamide composites were observed by scanning electron microscopy (SEM).

Fig. 1 Extracted bamboo fibers

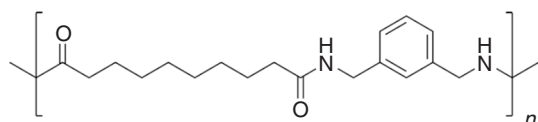


Fig. 2 RilsanXD chemical structure

Composite samples were cryo-fractured by breaking frozen composites in liquid nitrogen. The samples were coated using platinum and observed with a JSM 7800F Prime (JEOL, Japan). This apparatus is equipped with a field-emission electron gun, at a voltage of 5 kV and a secondary electron detector.

Thermogravimetric analysis and gravimetric analysis

Thermogravimetric analysis (TG) and gravimetric analysis (GA) were performed using an TGA Q50 from TA Instruments; mass sensitivity was 0.1 μg ; and temperature sensitivity was 0.1 $^{\circ}\text{C}$. Sample mass evolution (initially 5–10 mg) was measured for a linear increase of temperature under inert gas (nitrogen) or oxidant atmosphere (air). For TG, the temperature rate was 10 $^{\circ}\text{C min}^{-1}$.

In GA, mass variation was measured versus time at a given temperature under air atmosphere. Isotherms were reached with a heating ramp of 20 $^{\circ}\text{C min}^{-1}$.

Tensile test

Before tensile testing, fibers were visually selected in order to verify the absence of major damages and conditioned at relative humidity and room temperature. Fibers diameter was determined by optical microscopy; the measurement was taken with 9 points giving an average diameter for each fiber. In order to determine the mechanical properties of extracted fibers, tensile tests were performed using a paper frame [41]. The fiber was glued on the paper frame (Fig. 3) with a gauge length of 25 mm. The frame offers clamping surfaces with sufficient friction to transfer load without damaging the fiber; sliding in the clamp was avoided; and fiber alignment in the grips was insured. The paper frame was clamped in

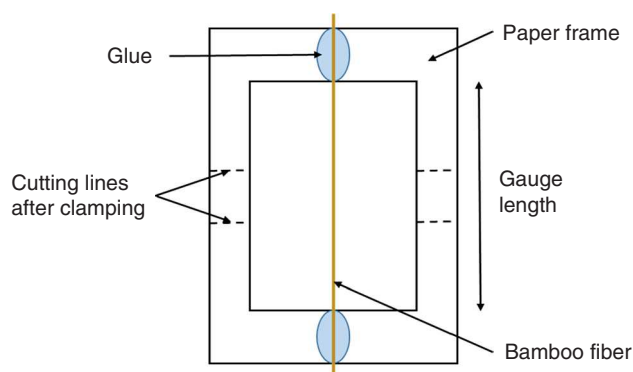


Fig. 3 Schematic representation of paper frame for the single-bamboo fiber tensile test

the machine at top and bottom, and was then cut carefully. Tensile tests were performed with a Criterion Model 43 (MTS, USA) equipped with a 2.5 kN capacity load cell. Samples were analyzed at a tensile speed of 1 mm min^{-1} . The test was performed on 44 samples. The Young's modulus was calculated as the slope of the stress–strain curve in the linear strain interval.

Dynamic mechanical analysis

The mechanical behavior was determined by DMA using an Advanced Rheometric Expansion System setup (Rheometric Scientific, USA). Measurements were performed on parallelepipedic samples using torsional mode in the temperature range from -130 to 150 $^{\circ}\text{C}$, with a heating rate of 3 $^{\circ}\text{C min}^{-1}$, under nitrogen flow, at an angular frequency of 1 rad s^{-1} with a dynamic shear strain of 0.1%. After the first scan, samples were immediately cooled at the same rate and the second scan was always recorded with the same previous condition.

Complex shear modulus G^* is defined by the following equation:

$$G_{\omega}^*(T) = G'_{\omega}(T) + i \cdot G''_{\omega}(T)$$

where $G'_\omega(T)$ is the storage modulus and $G''_\omega(T)$ is the loss modulus.

Since composites are isotropic, the shear modulus G and the elongational modulus E are linked through the ν Poisson's coefficient:

$$G = \frac{E}{2(1 + \nu)}$$

Tsai model [42] predicts the evolution of the shear modulus of a heterogeneous material like composite. Composite modulus analysis should take into account the fibers contiguity. We define the degree of contiguity C as $C=0$ corresponds to a non-contiguity (isolated fibers) and $C=1$ corresponds to a perfect contiguity (all fibers are in contact). From an analytical point of view, a linear combination of a solution where all fibers are isolated from each other ($C=0$) and a solution for where all fibers are in contact ($C=1$) gives a correct modulus. In this case, Tsai proposes for storage modulus:

$$G = (1 - C)G_m \frac{2G_f - (G_f - G_m)V_m}{2G_m + (G_f - G_m)V_m} + C \cdot G_f \frac{(G_f + G_m) - (G_f - G_m)V_m}{(G_f + G_m) + (G_f - G_m)V_m}$$

where G is the composite modulus, G_m is the matrix modulus, G_f is the fibers modulus, and V_m is the matrix ratio (vol.%).

Processing

Before processing of composites, the thermal stability of bamboo fibers was determined by thermogravimetric analysis (TG). Dynamic tests were performed from room temperature to 1000 °C, at 10 °C min⁻¹, under a synthetic air flow. Mass loss and its derivative (DTG) are reported in Fig. 4. First stage of mass loss (6.7%) before 100 °C is due to water desorption. Following moisture evaporation stage, complex degradation occurs from 200 to 550 °C with a rapid decrease of mass. This thermal event is determined as active pyrolysis stage which may correspond to the decomposition of hemicelluloses, cellulose and lignin components of bamboo [43, 44]. The double DTG peak around 300 °C displayed hemicelluloses decomposition at first (200–315 °C) and decomposition by unzipping or cellulose trans-glycosylation in the range 315–400 °C. Lignin components decomposed slowly over the entire temperature range, which typically started at 160–530 °C [45, 46] with a DTG peak around 430 °C. Residue is about 0–1% (total decomposition). The maximal processing temperature is set before 200 °C: the beginning of fibers components degradation. The degradation and the

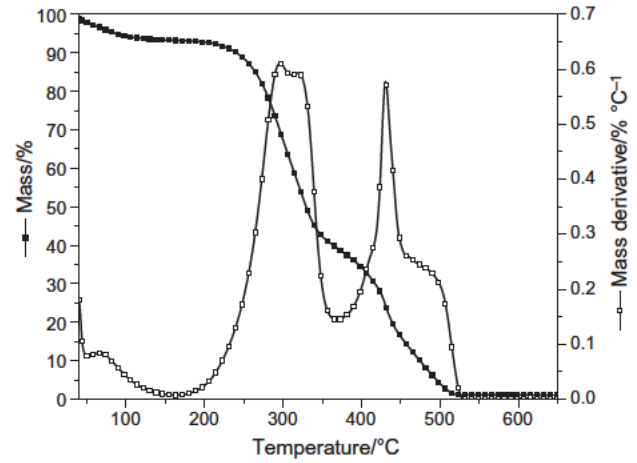


Fig. 4 TG and DTG curves of bamboo fibers under air atmosphere

decomposition can be distinguished by performing TG under N_2 . Indeed, under nitrogen only the degradation is observable since the entire decomposition requires oxygen.

Moreover, a gravimetric analysis (isothermal test) was performed on PA films for 100 min under synthetic air flow at 10 different isotherms between 200 and 400 °C. These films were prepared using a compression molding machine at 220 °C and were stored at room conditions (humidity and temperature). Figure 5 presents the evolution of the mass for polyamide. Each sample presents a mass loss of 1.3% at the beginning of the run. This decrease is associated with the water vaporization. The mass is constant at 200 °C and decrease slowly at 300 °C. Then, at 310 °C after 85 min, an increase of the mass loss is brought to the fore. This loss

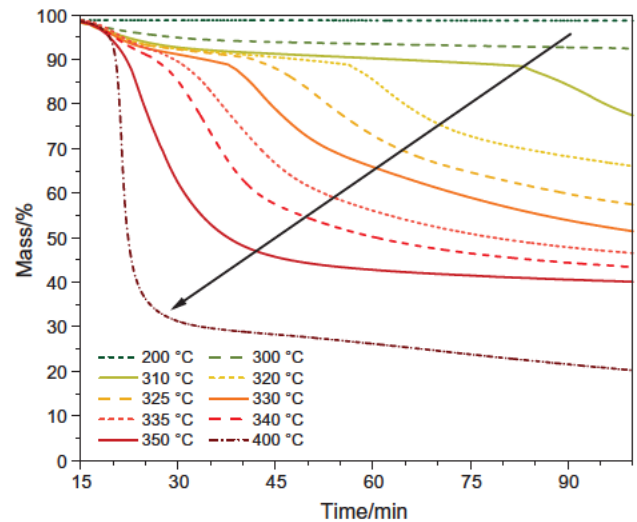


Fig. 5 Gravimetric analysis of polymer matrix under air atmosphere for 10 isotherms (The apparatus temperature takes time to stabilize. This explains why X axis begins to 15 min)

is attributed to the thermal degradation of the polyamide matrix. The higher isothermal temperature is; the sooner the degradation occurs. At 400 °C, the degradation takes place as soon as the temperature is reached.

Thus, by processing the composites for 15 min at 200 °C, the matrix is not degraded. Those parameters allow the melting of the matrix and a satisfactory flow between fibers.

Processing of composites

Polyamide pellets were hot pressed at 220 °C for 15 min in order to obtain a homogeneous film that was cut into rectangular sheets of 100 × 10 mm. Then, the fibers were disposed as a unidirectional layer between two polyamide films as shown on Fig. 6. The composite was pressed at 200 °C for 15 min [47] (final composite thickness ≈ 0.5 mm). Finally, the samples were naturally cooled (around 20 °C min⁻¹) and stored at room temperature for 24 h before any analyses.

Samples are labeled: BF for bamboo fibers and PA for the polyamide RilsanXD. Composites are named with the mass percentage of bamboo/polyamide.

Results and discussion

Bamboo fibers

SEM observation in Fig. 7 is representative of fibers extracted in the laboratory. Microscopy shows a technical fiber and its constitutive elementary fibers. Some parenchyma cells are still visible at the bottom of the image; however, fibers seem quite bare and so favorable to a good

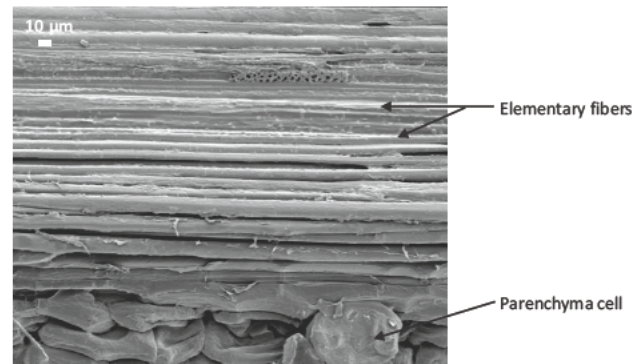


Fig. 7 SEM image of technical bamboo fiber

interface with the matrix. In addition, fibers are not damaged by the treatment, and therefore, their mechanical properties are maintained.

In order to determine mechanical behavior of extracted technical fibers, tensile tests were performed on 44 samples. Tensile strength and Young's modulus were measured as a function of fiber diameter.

The average diameter is 397 μm with a minimum of 239 μm and a maximum of 743 μm. Diameter variability is due to the extraction process. The variation of tensile strength as a function of the fibers diameter is reported on Fig. 8a. The average tensile strength is 323 MPa., and this value is comparable to the one reported in literature, e.g., Liu [17] that indicates a bamboo tensile strength between 140 and 800 MPa. The tensile strength minima in this work are 44 MPa and the maxima 742 MPa. Maximum value is obtained for smaller diameters in the range of 300–350 μm

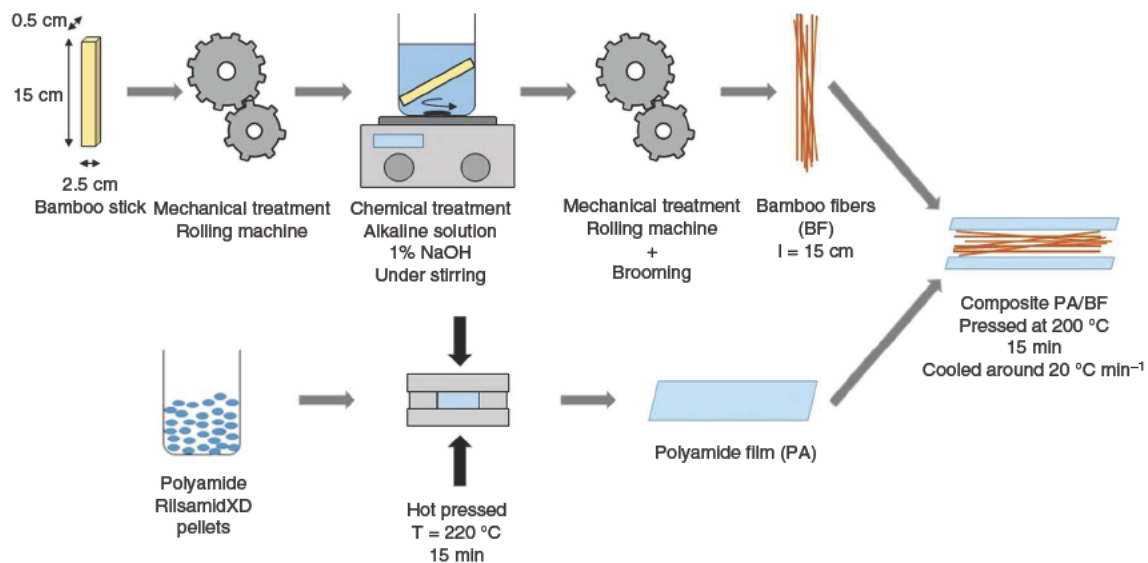


Fig. 6 Schematic representation of bamboo fibers/polyamide composites processing

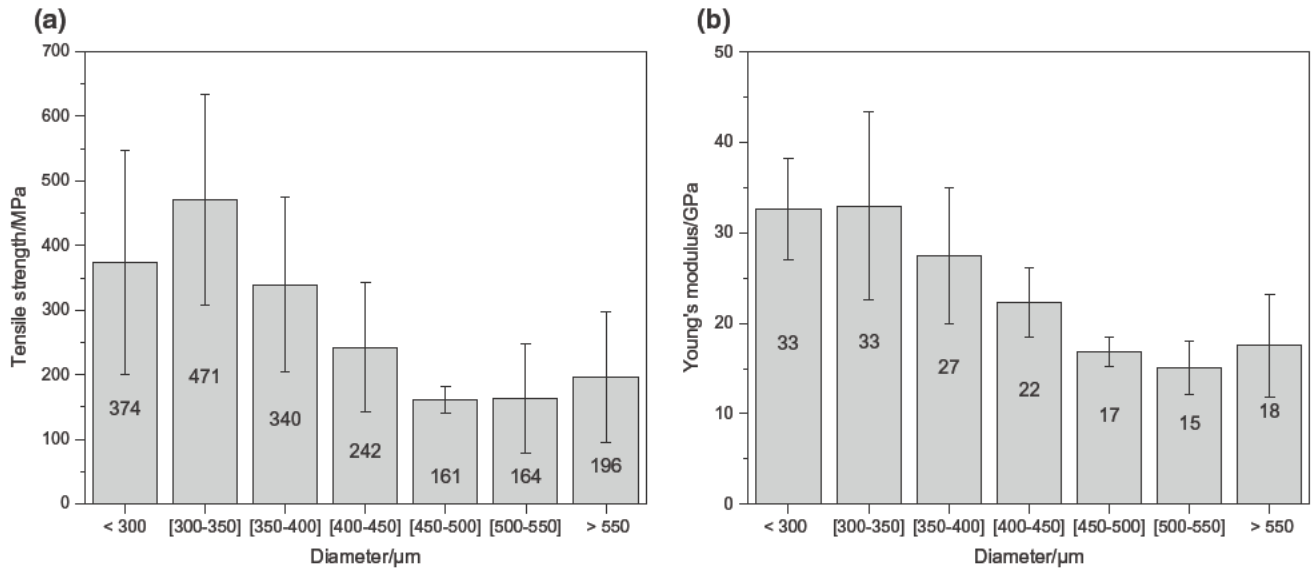


Fig. 8 **a** Plot of tensile strength versus technical fiber diameter, **b** Plot of Young's modulus versus technical fiber diameter

and minimum value for largest diameters. This divergence can be attributed to the non-uniform diameter of bamboo fibers or to the presence of weak points in fiber. Moreover, the presence of parenchyma cells and/or induced defects due to extraction can explain experimental results.

The correlation between the Young's modulus and the fiber diameter is assessed in Fig. 8b. Average Young's modulus is 26 GPa with a minimum of 10 GPa and a maximum of 52 GPa. Just like for tensile strength, Young's modulus is higher for small diameter fibers. It can be observed that the spread in tensile strength is higher than the spread in Young modulus. This behavior is consistent with the fact that strength is defect sensitive, whereas the modulus is governed by the intrinsic structure [48].

Bamboo fibers/polyamide composites

Extracted and characterized fibers were used to prepare composites. Composites with different fibers/matrix compositions have been implemented according to the process presented earlier.

Morphology of composites

Figure 9 presents the cryo-cuts of a 60/40 BF/PA composite observed by SEM. The SEM observation shows continuity between polyamide and bamboo fibers which means there is a good interfacial adhesion between matrix and fiber. This result is consistent with DMA analysis and refers the existence of hydrogen bonds between polyamide matrix and fibers. These bonds are inherent to the material and may be

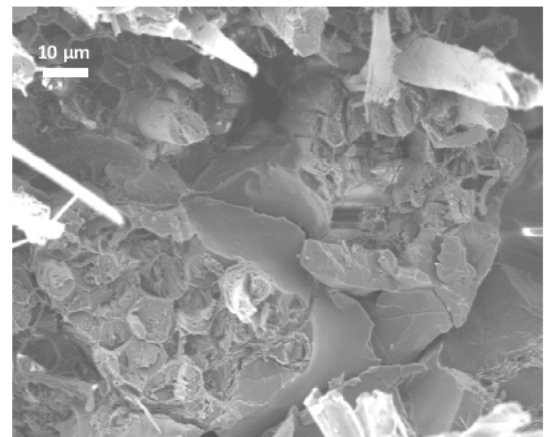


Fig. 9 SEM image of the interface in BF/PA composite

sufficient to promote interfacial adhesion and create matter continuity.

Composites analysis

Samples were characterized by TG under nitrogen atmosphere. Figure 10 shows the DTG between 100 and 900 °C for 7 different ratios between 0 m % fiber and 100 m % fibers. These DTG curves carried at 10 °C min⁻¹ exhibits 2 peaks. The highest is at 400 °C, for pristine polymer: the magnitude of this peak is maximum. For 100 m % fibers, there is only a residue. Accordingly, this peak results mainly from the matrix degradation.

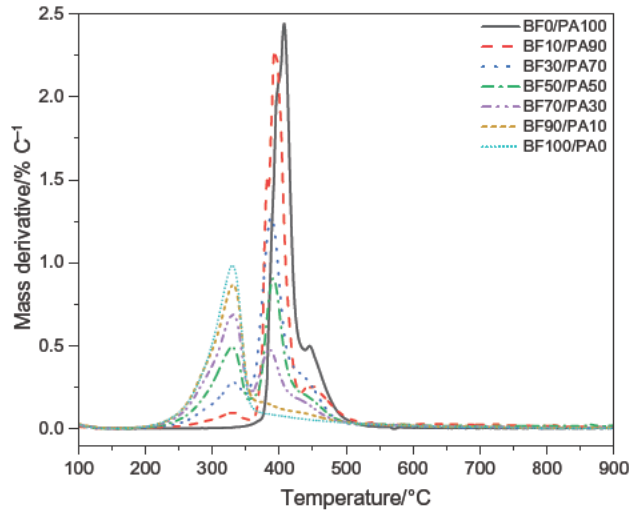


Fig. 10 DTG curves of composites recorded under nitrogen atmosphere at 10 °C min⁻¹

Moreover, the pure matrix peak initially at 408 °C shifts at lower temperature (390 °C) with the fibers introduction in composites. These values suggest two assumptions: either the peak shifts because of fibers/matrix interactions or fibers addition degrades the thermal properties of the matrix.

The peak magnitude at 330 °C zoomed in Fig. 11a increases linearly with the fibers ratio introduced in the composite. With 0% fibers the peak is missing, and for 100% fibers the maximum of the mass derivative is equal to 1. Therefore, it is only dependent on the fibers content. This

result is due to the fact that polyamide degradation occurs only after 300 °C.

Maximum value of the peak at 330 °C on a DTG at 10 °C min⁻¹ could be a reliable way of determining the ratio of filler in fibers/polyamide composite without having to do any calculations.

In order to verify the proportionality and the correlation coefficient, an abacus was drawn. Figure 11b presents the DTG peak height for 330 °C function of the calculated fibers proportion in the composite. Linear fit of these points gives a Person's correlation coefficient of 0.99977 with an *R*-Square of 0.99944. This method is therefore reliable to go back to the mass percentage of fibers introduced into composites simply with the maximum value of the peak at 330 °C.

Dynamic mechanical properties Bamboo fibers influence on mechanical properties of polyamide has been studied by dynamic mechanical analysis. 60/40 bamboo fibers/polyamide single ply composites were compared to the polyamide matrix (Fig. 12). DMA curves correspond to the second scan in order to erase the thermal history inherent to the processing of composites. The storage modulus *G'* was indicated by filled symbols and the loss modulus *G''* by open symbols.

Shear storage modulus On the storage modulus *G'*, one major step is observed around 55 °C: it reflects the transition from the glassy plateau to the rubbery plateau through the viscoelastic transition of polyamide. Upon the bamboo fibers introduction, an increase of the storage modulus *G'* is observed for both the vitreous and the rubbery states.

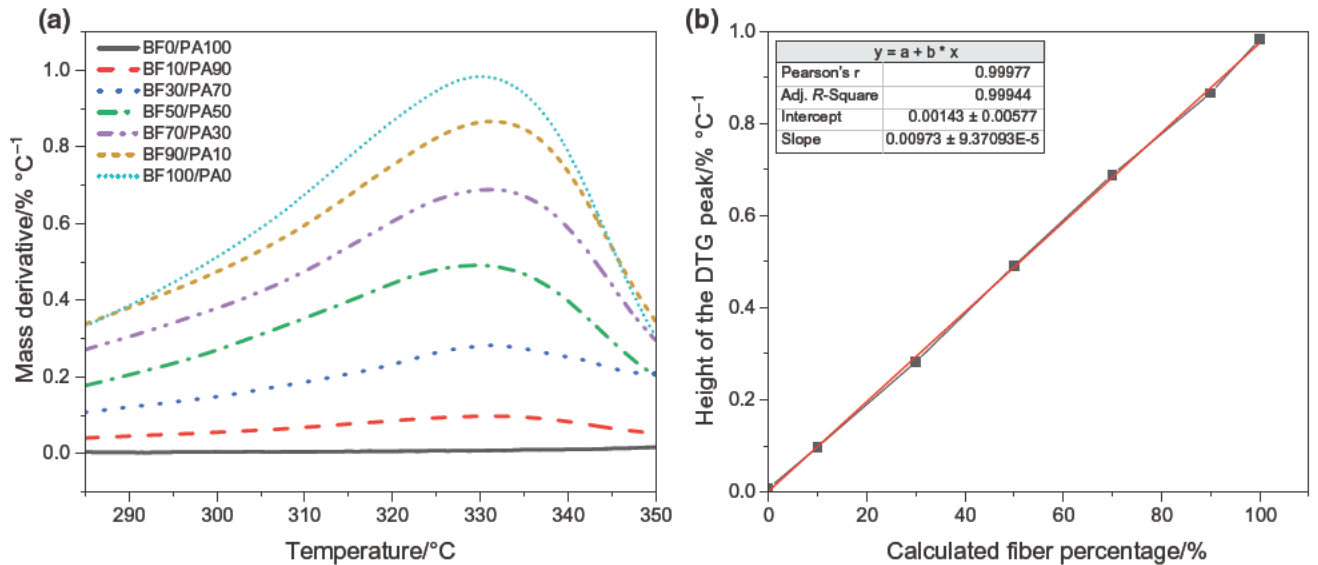
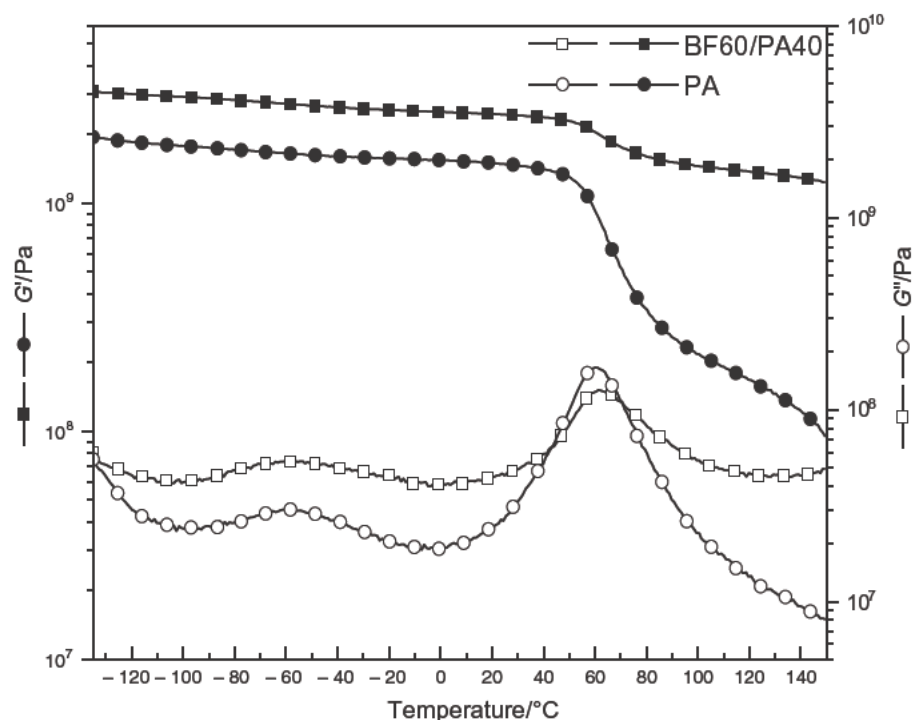


Fig. 11 a Zoom on the DTG curves between 285 and 350 °C, b DTG peak magnitude at 330 °C as a function of the calculated fibers percentage in the composite

Fig. 12 DMA curves of polyamide and bamboo fibers/polyamide composites



In the *glassy state*, this increase is associated with the transfer of stresses between fibers and matrix due to the existence of static hydrogen bonds between hydrophilic filler and amide groups of the matrix. Knowing the G'_m modulus of the matrix ($G'_m = 1.5$ GPa at $T = 20$ °C) and the composite one ($G' = 2.46$ GPa at $T = 20$ °C) as well as the mass percentage of fibers in the composite (60 m %), we can evaluate the shear modulus G'_f of bamboo fibers using the Tsai model. The appropriate value of C for an actual composite was empirically determined. For example, for glass–epoxy composite, $C = 0.2$ was found to provide the best agreement with experimental data [49]. The value of C being generally between 0 and 0.4, it is possible to determine bamboo fibers modulus for these values. We have $4.2 < G'_f < 4.8$ GPa. These values sound consistent with chemical composition of bamboo.

In the *rubbery state*, polyamide and bamboo fibers/polyamide composite have different behaviors: the first one exhibits the flow of a linear polymer while the composite behaves as a network. In composites, bamboo fibers act like additional topological nodes. The viscoelastic step corresponds to the transformation of static hydrogen bonds into dynamic hydrogen bonds. By analogy with glassy modulus, the rubbery moduli of the polyamide and the composite at 80 °C, i.e., before the flow, have been compared. It is interesting to note that the contribution of the fibers remains of the same order of magnitude. The point that needs to be emphasized is the high value of the rubbery modulus for composite: 1.6 GPa.

Shear loss modulus The loss modulus G'' (open symbols) shows three relaxations. At lowest temperatures, part of the γ relaxation is observed. It was reported for polymers with aliphatic sequences involving more than 4 CH_2 . This relaxation is associated with crankshaft motions of ethylene sequences in polyamide chains. Peak at -60 °C, designated as the β relaxation, is due to the mobility of amide/amide interactions. This mode is more resolved in polyamide than in composite. The temperature of the peak is not shifted upon the introduction of bamboo fibers. Moreover, β relaxation magnitude is the same with or without fibers: it can be explained by a similar water uptake for the two samples. The widening of the relaxation indicates a higher heterogeneity of the glassy state upon introduction of bamboo fibers. For polyamide, α relaxation is situated at $T_\alpha = 60$ °C. For composite, this value is slightly shifted till $T_\alpha = 62$ °C upon fibers introduction. As it can be expected, the magnitude of the α peak is lower for the composite than for the matrix.

Conclusions

Using mild chemical and mechanical treatment, bamboo fibers were extracted from *Phyllostachys viridiglaucescens* grown in southwest of France giving rise to a sustainable source of filler. They have an average diameter of 397 μm . Their average tensile strength is 323 MPa, and their average Young modulus is 26 GPa. Maxima values of these tensile properties are obtained for diameters in the range of

300–350 μm . Such continuous bamboo fibers were used as reinforcement of a bio-based polyamide (RilsanXD). Single ply bamboo fibers/polyamide sustainable composites were processed. The composition of composites was determined from the DTG analyses of the peak associated with bamboo situated at 330 $^{\circ}\text{C}$. Composites with 60 m % of bamboo fibers were prepared and analyzed. The observation of SEM cryo-cuts images shows the absence of voids between technical fibers and polyamide matrix which suggests interesting mechanical properties. The cohesion in shear was tested by dynamic mechanical analyses. Comparative data recorded on bio-based polyamide and 60/40 bamboo fibers/polyamide composite highlights a significant improvement of the shear glassy modulus which is multiplied by 1.6 at 20 $^{\circ}\text{C}$. This result seems consistent with SEM images and may be due to a dense network of static hydrogen bonds.

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