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Physical structure and mechanical properties of polyamide/bamboo composites

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Colette Lacabanne¹

Abstract The main objective of this work is to process innovative bamboo flour (BF)-reinforced polymer composites. In this context, polyamide 11 (PA 11) is used as technical matrix. Moreover, BF is treated with tetraethyl orthosilicate (TEOS) playing the role of coupling agent. SEM observations show no influence of TEOS on the affinity. The composites were analysed by DSC and DMA, in comparison with neat PA 11. DSC analyses of PA 11/BF highlight that there is no significant modification of the percentage of crystallinity upon introduction of BF whatever the treatment is. Concerning the amorphous phase, only a slight shift of the glass transition of PA 11 from 35 °C (PA 11) to 38 °C (PA 11/BF composites) is recorded. This shift can be explained by physical bonds at the interface PA 11/BF. DMA analyses allow us to explore the role of BF fillers onto the properties of PA 11/BF composites. The first observation is a slight improvement of the shear modulus G' when the concentration in TEOS increases. The β relaxation at -80 °C is associated with the mobility of the complexes free amide groups/water molecules. There is no shift of the peak due to the presence of BF fillers. The magnitude and width of the relaxation increase with BF and also with TEOS treatment. These observations highlight the increase of hydrogen-bonded

water in various sites of the vitreous state. The α relaxation is associated with the anelastic mobility liberated at the glass transition. For PA 11/BF composites, it is constituted by two components: the lower-temperature one due to neat polyamide and a higher-temperature one associated with PA 11/BF amorphous domains with a lower thermal conductivity.

Keywords Polyamide/bamboo composite · Dynamic mechanical analysis · DSC · Bamboo flour · Viscoelasticity · Glass transition · Coupling agent

Introduction

In recent years, natural fibres gained interests to be used as fillers reinforcing polymer composites and more precisely, the vegetable fibres, such as wood, flax or hemp [1–5]. These fillers have good mechanical properties, and due to their low density, they are an interesting renewable green solution to conventional fibres such as glass fibres [6]. Among all the vegetable fibres, bamboo seems to be one of the most interesting solutions [7–9]. Bamboo is in abundance in all the continents, and its quick growth leads to continuous provision which is an important asset for industrial applications. The main drawback is its thermal stability which limits the process temperature and consequently the choice of the matrix.

The thermoset matrices have low process temperature compatible with the thermal stability of the natural fibres allowing to preserve them during the processing [10]. The polyepoxy (EP) was highly investigated [11–14]. Jain et al. [15] have shown the improvement of the mechanical properties due to the use of bamboo mats. Moreover, Kushwaha et al. [16] have increased the properties of

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bamboo/EP composites by treating the bamboo with solution of different concentrations in NaOH and they found an optimum for 5% NaOH. Furthermore, the water uptake decreased due to the treatment with soda, but with silanes as well [17]. Other studies analysed bamboo-reinforced phenolic composites. Das et al. [18, 19] obtained better mechanical properties and less water uptake for a treatment up to 20% NaOH. The drawback of the thermosets is they are not recyclable. Further studies have been done on bamboo fibres-reinforced thermoplastic composites, and more precisely the polyolefins such as polypropylene (PP). One of the main challenges is to promote a good adhesion between the hydrophilic fillers and the hydrophobic matrix [20, 21]. The use of copolymers is one route to enhance the compatibility [22, 23]. These studies have shown the use of maleic anhydride polypropylene (MAPP) as coupling agent, in addition to improving the adhesion and the mechanical properties, leads to the formation of transcrystallinity at the interface: some spherulites grew along the fibres. The fibres may act as nucleation sites. Another way to improve the compatibility is to modify the surface of the bamboo with silanes [24]. Lee et al. [25] reported an increase of the mechanical properties with the addition of silane in bamboo/PP composites. For PVC/bamboo flour composites, interfacial adhesion has been promoted by coupling agents [26]. Hydrothermal pretreatment of bamboo particles has also given interesting data [27]. In order to process fully bio-based composites, polylactic acid (PLA) was used as matrix [28, 29]. Sukmawan et al. [30] studied the steam-exploded bamboo treated with an alkali solution in order to promote the compatibility with PLA. Their results showed PLA/bamboo composites have mechanical properties close to the ones of glass fibres-reinforced composites. Lee et al. [31] improved the properties of bamboo/PLA composites and bambou/polybutylene succinate (PBS) composites using lysine-diisocyanate (LDI) as coupling agent. LDI improves the adhesion increasing the mechanical properties and also decreasing the water uptake, just like silanes in EP.

In this work, another bio-based renewable thermoplastic, polyamide 11 (PA 11), was investigated. This semicrystalline polymer, with a melting point around 189 °C, has higher mechanical properties than the matrices previously investigated. PA 11 was reinforced with Vietnamese bamboo flour (BF). A thermogravimetric study was performed in order to check the integrity of BF after the processing cycle of PA 11. In order to optimize the matrix/filler compatibility, BF was treated with tetraethyl orthosilicate (TEOS). The PA 11/BF interface, with and without coupling agent, was observed by scanning electron microscopy. The physical structure of the matrix of composites was analysed by differential scanning calorimetry. Finally,

the mechanical properties of the composites were studied, in shear, by dynamic mechanical analysis.

Experimental

Materials

The *dendrocalamus barbatus* bamboo flour (BF) was purchased from VNDD Ltd (Vietnam). The final grains of 150 µm were selected, thanks to a 100-mesh sieve. The BF was dried in an oven for 5 h at 100 °C to removed humidity. The BF was added into an ethanol solution containing tetraethyl orthosilicate (TEOS) in which the pH was adjusted between 8 and 9 using an ammonia solution. TEOS with 0.934 g cm⁻³ was provided by Daejung Company (Korea). Different mass ratios of TEOS were selected: $m_{\text{TEOS}}/m_{\text{BF}} = 5$ and 10 mass%. Distilled water was added after 30 min, at a temperature of 50 °C under magnetic stirring for 2 h. Finally, the suspension was filtered and washed by alcohol before to be dried in a vacuum oven at 80 °C for 8 h.

PA 11 was purchased in fine powder about 30 µm from Arkema (France). PA 11 is a technical thermoplastic with a glass transition between 30 and 40 °C and a melting temperature about 189 °C.

Thermal stability

Before the processing of the composites, the thermal stability of untreated and treated BF was studied by thermogravimetric analysis using a TG Q50 (TA Instruments, USA). The samples were stored at room conditions (humidity and temperature). An isothermal test was performed at 200 °C during 60 min, under a synthetic air atmosphere. Figure 1 presents the evolution of the mass for untreated

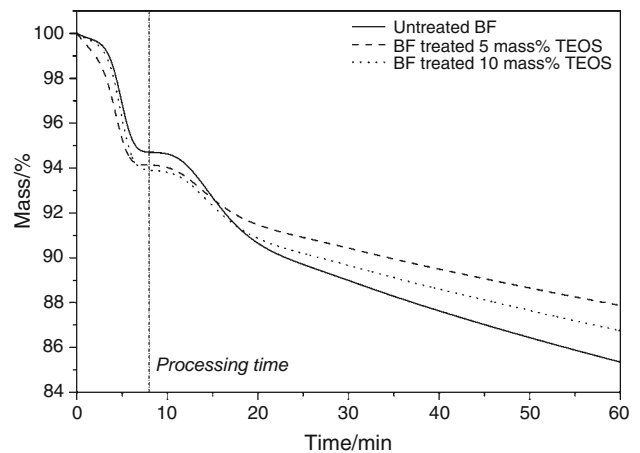


Fig. 1 Evolution of the mass of untreated BF, treated with 5 mass% of TEOS and treated with 10 mass%, at 200 °C for 60 min

and treated BF. The results have the same behaviour, independent of the concentration in TEOS. For each sample, there is a mass loss of about 6% until 7 min. This decrease is associated with the water vaporization [32, 33]. Then the mass is constant before decrease after 10 min at 200 °C, and then the decrease slows. This loss is associated with the thermal degradation of the bamboo flour. According to the results, the duration of 8 min has been chosen for the processing of PA 11/BF composites in order to preserve the natural fillers, regardless of the treatment.

Processing of composites

Untreated and treated BF and PA 11 powders were dispersed into an ethanol solution at a ratio $m_{BF}/m_{PA\ 11} = 40$ mass%. The mixture was stirred by ultrasounds for 2 min. Then, it was dried using a rotary vacuum evaporator at 80 °C for 30 min, in order to totally remove ethanol. Finally, the mixture was pressed into rectangular specimens using hot pressing at 198 °C, under a pressure of 10 MPa for 8 min. The sample designations and their proportions are indexed in the Table 1. CA0 designates composites reinforced by untreated BF, and CA5 and CA10 designate composites reinforced by BF, respectively, treated with 5 and 10 mass% of coupling agent (CA), here TEOS.

Methods

Scanning electron microscopy

Scanning electron microscopy (SEM) was used for the observation of the interface between the matrix and the fillers. The cryo-fractured surfaces were obtained by breaking composites frozen in liquid nitrogen, and then the samples were coated using platinum. The interfaces were observed by using a JSM 6700F (JEOL, Japan), equipped with field emission gun electron, at a voltage of 5 kV. It is equipped with a secondary electron detector.

Differential scanning calorimetry

The physical structure of the PA 11/BF composites were analysed using a Diamond DSC (PerkinElmer, USA) from 0 to 200 °C at 10 °C min⁻¹ under helium atmosphere with

Table 1 Mass ratio of PA 11, BF and TEOS for the composites

Samples	PA 11/mass%	BF/mass%	TEOS/BF ratio/mass%
PA 11 (reference)	100	0	–
CA0	60	40	0
CA5	60	40	5
CA10	60	40	10

a rate flow of 150,000 Pa. The samples (around 12 mg) were placed in closed aluminium pans. The apparatus was calibrated in temperature using stain and indium standards.

Dynamic mechanical analysis

The thermal behaviour of the composites was determined by dynamic mechanical analyses (DMA) using an Advanced Rheometric Expansion System set-up (Rheometric Scientific, USA). Rectangular samples of dimension 50 × 10 × 0.65 mm were used for the trials. The measurements were taken from –130 to 150 °C, with a heating rate of 3 °C min⁻¹, under nitrogen flow, at a frequency of 1 rad s⁻¹, with a dynamic strain of 0.1%.

Results and discussion

Morphology analysis

Figure 2 presents the cryo-cuts of the composites observed by SEM, and the interface matrix/filler is illustrated by the white dashed line. In Fig. 2a), there is a continuity between the PA 11 and untreated BF. This continuity means there is a good interfacial adhesion between the matrix and the filler. Previous studies highlighted that TEOS creates interfacial adhesion in apolar matrices. In such cases, there is a critical ratio above which the adhesion between the matrix and the fillers is damaged [34]. Wu et al. [21] determined the limits in coupling agent from 2 to 8 mass%. With a polar matrix such as PA 11, the maximum coupling agent content that can be used without damage is higher.

It is worth noting that the same morphologies are observed for the composites reinforced by the untreated BF. This means that the hydrogen bonding inherent to the PA 11 matrix is sufficient to promote interfacial adhesion and to create matter continuity.

Physical structure

The crystallinity of each material had been determined according to the following equation:

$$\chi_c(\%) = \frac{\Delta H_m}{\Delta H_\infty} \times 100$$

where χ_c is the crystallinity, ΔH_m is the measured melting enthalpy, and ΔH_∞ is the theoretical melting enthalpy of 100% crystalline PA 11. The value used here is 244 J g⁻¹ [35]. The curves of PA 11, CA0, CA5 and CA10 are shown in Fig. 3: on the left hand side, the lower-temperature zone shows the glass transition; on the right hand side, the higher-temperature zone shows the melting.

All the thermal parameters are indexed in Table 2.

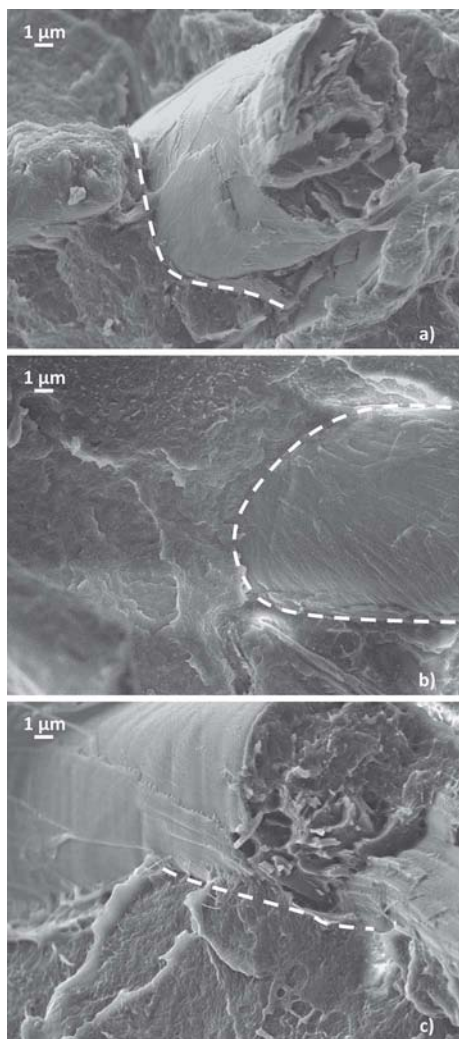


Fig. 2 SEM observations of the interface between BF and PA 11 for **a** CA5, **b** CA10 and **c** CA0 composites. *Dashed lines* are guidelines for the interface

The glass transition of PA 11 is 35 °C. An endothermic phenomenon is superimposed on the glass transition step: this event is characteristic of physical ageing. For composites, the glass transition is slightly shifted to 37 °C and physical ageing is still present. This behaviour can be explained by an increase of the density of hydrogen bonds due to BF. The melting point of PA 11 is 188 °C and exhibits a shoulder at 180 °C. It is explained by the existence of crystallites with different morphologies and different sizes [36, 37]. For the composites, the melting points are not modified, neither the introduction of BF nor its treatment with TEOS. The melting enthalpies were measured from 170 to 195 °C, integrating the main peak and the small one. Moreover, the crystallinity for each composite is 22% which is close to the crystallinity of PA 11: 21%. The BF has no significant influence on the crystallinity of PA 11.

In the literature, the introduction of treated natural fillers may increase the crystallinity of the matrix [38, 39]. In such studies, the fillers were treated by grafted polymers. Other works highlighted a decrease of the crystallinity due to a modification of the kinetics of crystallization [40, 41]. In this work, the coupling agent is a silane which induces no modification of the crystalline phase. In our previous work, TEOS had no significant influence on cross-linked polyethylene crystallinity (XLPE) [42].

Mechanical behaviour

Figures 4 and 5 show, respectively, the shear conservative modulus G' and the shear loss modulus G'' of PA 11 and the composites.

In Fig. 4, two relaxations are observable. The step at -80 °C is associated with the β relaxation which is the rotation of the free amides groups [43, 44]. Around 35 °C, the α relaxation is associated with the mechanical manifestation of the glass transition. The temperature of the α relaxation is consistent with DSC data. Figure 4 highlights an improvement of glassy modulus due to the introduction of treated BF. In the case of CA0, there is also an enhancement of the modulus. This increase may be explained by the formation of hydrogen bonds between the hydrophilic filler and the polar matrix. Liu et al. [45] have highlighted an increase of the mechanical properties due to the introduction of untreated BF in HDPE.

The rubbery plateau of CA0, CA5 and CA10 reaches the same value, which means that TEOS has practically no influence on the rubbery phase. Contrarily, the presence of the BF filler on the rubbery PA 11 matrix is clear: BF might act as topological nodes.

In Fig. 5, a part of the γ relaxation is observed at the lowest temperatures. This relaxation is associated with the well-known crankshaft motion of the ethylene sequences in the PA 11 chains [43]. The previous α and β relaxations of PA 11 are well resolved. For the β relaxation, the temperature of the peak maximum is not shifted upon the introduction of the BF filler regardless of untreated or treated. The magnitude of this β relaxation increases with the presence of BF and also upon the treatment: it can be explained by a slight increase of the water uptake [44]. The widening of the relaxation indicates a higher heterogeneity of the glassy state upon the introduction of BF filler.

For PA 11, the α relaxation temperature at the maximum of the peak is $T\alpha = 35$ °C: this value is consistent with the glass transition as measured by DSC. For PA 11/BF composites, an additional component at $T\alpha = 43$ °C is recorded. The amorphous region is biphasic: a neat PA zone and a zone with BF giving the higher $T\alpha$. The shift may be explained by the lowering of the thermal

Fig. 3 DSC curves of neat PA 11 and composites CA0, CA5 and CA10: the glass transitions are on the *left* and the melting points on the *right*

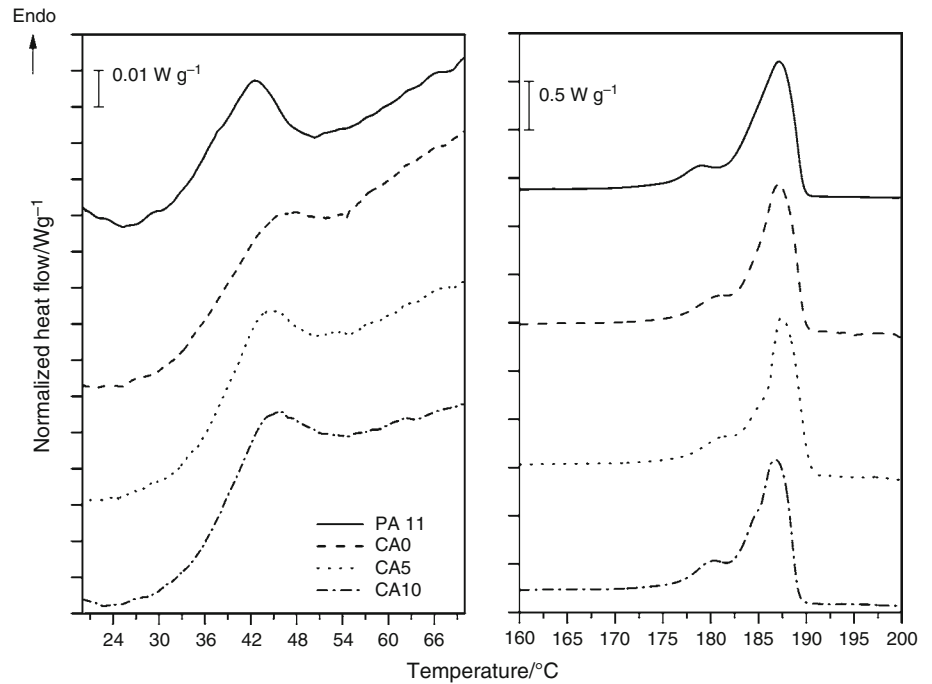


Table 2 Thermal parameters of PA 11 and composites

Samples	$T_g/^\circ\text{C}^a$	$T_m/^\circ\text{C}^b$	$\Delta H_m/\text{J g}^{-1b,c}$	$\chi_c/\%$	PA 11/BF measured ratio/mass%
PA 11 (reference)	35 (2) ^d	188 (1)	51 (1)	21 (1)	–
CA0	37 (2)	187 (1)	53 (2)	22 (1)	62 (2)
CA5	37 (1)	187 (1)	54 (1)	22 (1)	63 (1)
CA10	38 (1)	187 (1)	54 (1)	22 (1)	62 (1)

^a The glass transition temperature was determined during the first scanning

^b The melting point and the melting enthalpy were determined during the second scanning

^c The melting enthalpies for the composites were normalized by the PA 11 content

^d SD is indicated in parentheses

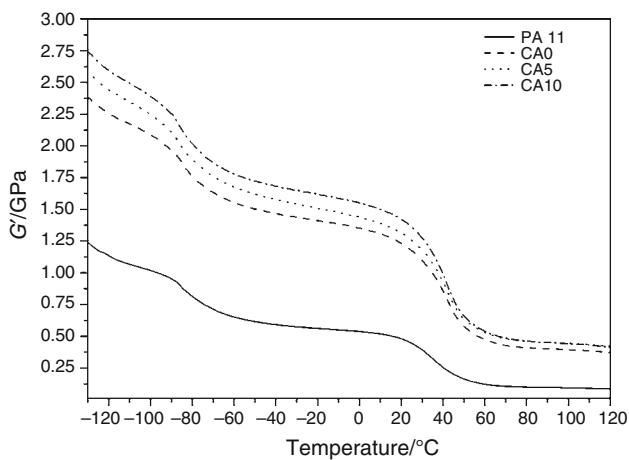


Fig. 4 Conservative shear moduli G' of PA 11 and composites CA0, CA5 and CA10

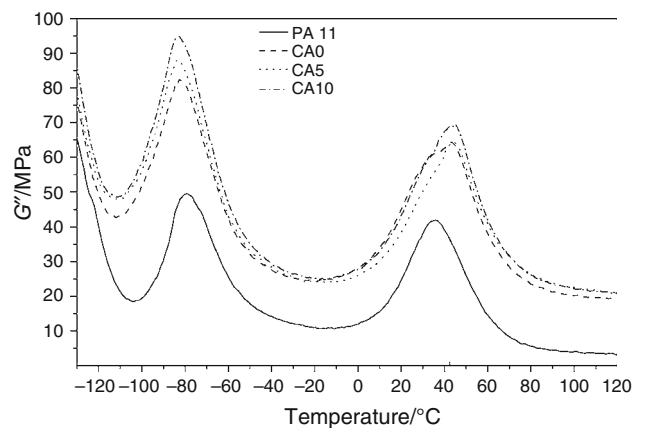


Fig. 5 Loss shear moduli G'' of neat PA 11 and composites CA0, CA5 and CA10

conduction of the PA/BF domains due to the presence of BF filler. The delay that does not play any role in a few mg sample in DSC becomes significant for DMA samples of some g. A shoulder can be observed at about 35 °C for the composites: it is less visible for CA5 due to a lack of homogeneity of the samples.

Conclusions

Innovative bamboo flour (BF)-reinforced polyamide 11 (PA 11) composites were processed and analysed by DSC and DMA, in comparison with neat PA 11. The observations by SEM show interfacial adhesion between PA 11 and BF. The SEM images did not highlight an influence of the presence of TEOS, which is used as coupling agent, or its concentration.

DSC analyses highlight that there is no significant modification of the percentage of crystallinity upon introduction of BF. Concerning the amorphous phase, only a slight shift of the glass transition of PA 11 from 35 °C (PA 11) to 38 °C (PA 11/BF composites) is recorded. This shift can be explained by physical bonds at the interface polyamide/BF.

DMA analyses allow us to explore the role of BF filler onto the anelastic properties of PA 11/BF composites. A slight improvement of the shear modulus G' is observed when the concentration in TEOS increases. The β relaxation at -80 °C is associated with the mobility of the complexes free amide groups/water molecules. There is no shift of the peak due to the presence of BF filler. The magnitude and width of the relaxation increase with BF and also with TEOS treatment. These observations highlight the increase of hydrogen-bonded water in various sites of the vitreous state.

The α relaxation is associated with the anelastic mobility liberated at the glass transition. For PA 11/BF composites, it is constituted by a complex peak with two components: the lower-temperature one is shifted from 35 °C for PA 11 to 37 °C for composites, just like in DSC. The higher-temperature one shows a maximum at 43 °C. The splitting of the α relaxation reflects the existence of two regions in the amorphous phase: neat PA 11 amorphous domains and PA 11/BF amorphous domains with a lower thermal conductivity.

For further study, we will extend this investigation to polyamide/bamboo fibre composites.

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