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Eprints ID : 16731

To link to this article : DOI:10.1007/s10973-015-5176-x
URL : <http://dx.doi.org/10.1007/s10973-015-5176-x>

To cite this version : Haddou, Geoffrey and Dandurand, Jany and Dantras, Eric and Maiduc, Huynh and Thai, Hoang and Giang, Nguyen Vu and Trung, Tran Huu and Pontains, Philippe and Lacabanne, Colette *Mechanical and thermal behaviour of bamboo flour-reinforced XLPE composites*. (2016) *Journal of Thermal Analysis and Calorimetry*, vol. 124 (n° 2). pp. 701-708. ISSN 1388-6150

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Mechanical and thermal behaviour of bamboo flour-reinforced XLPE composites

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Abstract In the present work, the influence of natural fillers (bamboo flour/BF) on crosslinked polyethylene (XLPE) has been discussed. The main difficulty for the preparation of the composites is to establish adhesion between hydrophilic natural fillers and hydrophobic matrix. Consequently, coupling agent (CA) as silane must be added to promote interface. In this work, different amounts of tetraethyl orthosilicate (TEOS) were used as coupling agent and SEM images allow us to control the quality of the interface. Differential scanning calorimetry (DSC) shows that the XLPE crystallinity is unmodified upon the introduction of BF. The absence of transcrystallinity is important for preserving the ductility of composites. The dynamic mechanical analyses (DMA) highlight an optimum of shear modulus for a TEOS content of 5 mass% (CA5). Above this amount, the mechanical properties are reduced. These results were confirmed by the SEM observations which highlighted the decrease in the interface between the XLPE and BF. Furthermore, neither the introduction of BF nor the treatment with TEOS exerts an influence on the relaxation temperature: there is no plasticization. The combination of DSC/DMA allows us to show that higher viscoelasticity favouring impact strength is observed in XLPE/BF CA5 composites.

Keywords Dynamic mechanical analysis · DSC · Bamboo fibre · Compatibility · XLPE · Coupling agent

Introduction

Polymer composites reinforced with natural fillers like wood fibre or flour have acquired general popularity over the two last decades [1–4]. These environmentally friendly composites [5–7] have many remarkable properties in comparison with conventional composites using glass fibres and mineral fillers because of their low density and low cost. Often, compatibilizers between natural filler and polymeric matrix optimize the mechanical properties [8]. Consequently, wood polymer composites are used in many applications such as lumber, decking, railing, window profiles, door frames, furniture and pallets [9, 10]. However, the use of wood to make such composites might have consequences on the exploitation and protection of forest even in the case of fast-growing species [11]. Bamboo is abundantly available in many countries. The total bamboo forest area in the world has reached 22 million hectares. Moreover, bamboo is one of the fastest renewable plants with a maturity cycle of 3–4 years. Bamboo possesses excellent mechanical properties in comparison with its mass due to its hierarchical structures [12]. With such advantages, bamboo fillers promise to become one of the best natural reinforcement in polymeric composites [13–20]. Their use can help to reduce the demand for wood flour and environmental impacts associated with wood fibre harvesting.

Composites based on bamboo filler and polymer matrix exhibit some disadvantages, like poor adhesion between matrix and filler that easily absorbed moisture. Indeed, the main components of bamboo, cellulose and hemicellulose, possess a strong hydrophilic character. Consequently,

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hydrophobic properties of the matrix are required: polyolefins such as high-density polyethylene (HDPE) are satisfactory. Then, compatibility between the filler and the matrix is considered as the key to enhance properties of this composite [21, 22]. One route is to use copolymers as coupling agent [23, 24]. Recent reports on the filler-reinforced composites show that hybrid fillers (combining two or more different types of fillers) have been mainly used. Some studies have explored the effect of mineral filler and lignocellulosic fibres on mechanical properties, thermal stability and weathering resistance [25, 26]. HDPE/bamboo composites with different nanoclays and maleated polyethylene (MAPE) contents were fabricated by melt compounding. The X-ray diffraction (XRD) data showed that the clay was exfoliated only when 1 % clay was added to pure HDPE. For the HDPE/BF composites, tensile strength, bending modulus and strength were improved with the use of MAPE; however, the use of the clay in the system led to reduced mechanical properties. Another method is the modification of the bamboo fibre surface by using suitable surfactants as coupling agents [13]. Li et al. [27] reported the acetylation of Chinese bamboo flour that was chemically modified with acetic anhydride by using trichloroacetic acid as an activation agent. Several studies reported on hydrophobic modifications of bamboo flour [28]. A specific interest was devoted to silane coupling [29–32] that significantly improve adhesion in composites. Alkali treatment affects the swelling of cellulose fibres in converting cellulose structure in a thermodynamically more stable one. Subsequent chemical modification with silane for alkali-treated wood fibres would increase adhesion with polymer matrix in wood polymer composites. The use of silanes transforms hydrophilic surface of wood fibres into hydrophobic surface by silanol formation and bonding between siloxane and fibres [33]. Lee et al. [34] showed the effects of chemical modification (silane coupling) and filler loading on the fundamental properties of the bamboo fibres filled polypropylene (PP). The addition of aminopropyltrimethoxysilane (AS) and tetramethoxy orthosilicate (TMOS) after the alkali pretreatment of bamboo fibres improved adhesion between the bamboo fibres and PP matrix. Consequently, the resultant composites had improved tensile, flexural and impact strength.

Tabari et al. [35] have added SiO₂ nanoparticles during mixing of wood flour and PP using the injection moulding process. The results showed that the bending strength of the material significantly increases in comparison with original material without SiO₂ nanoparticles. However, mineral nanofillers used in the wood polymer composite also showed a few drawbacks like the agglomeration of nanofillers in the matrix and no chemical interaction with other components.

In order to overcome these problems in this study, the Vietnamese bamboo flour (BF) was modified by tetraethyl orthosilicate (TEOS) in ethanol solution to form in situ SiO₂ nanoparticles on the BF surface. The chemical bonds were established through condensation reaction of hydroxyl groups between the BF and SiO₂ nanoparticles. Therefore, SiO₂ could show a fine scattering on the BF and played an important role in improving the mechanical properties of HDPE matrix. In addition, dicumyl peroxide (DCP) played the role of the crosslinking agent of the matrix. Scanning electron microscopy allowed us to observe the bamboo flour/XLPE interface. After checking that the physical structure of the matrix was not modified, the performances of the composite were investigated under shear strain.

Experimental

Materials

Polymer matrix

High-density polyethylene (HDPE), with density of 0.96 g cm⁻³, was purchased from LG Chemical Company (Korea). During processing, HDPE was crosslinked by dicumyl peroxide (DCP) (98 %) provided by Merck Company (Germany). Crosslinked HDPE was referred as XLPE.

Bamboo flour

The BF (moisture lower than 12 %) of *Dendrocalamus barbatus* (North Vietnam) was provided by VNDD Ltd. (Vietnam). The BF was ground and passed through 100-mesh sieve to select the final grains that were dried in an oven for 5 h at 100 °C to totally remove humidity. Tetraethyl orthosilicate (TEOS) with 0.934 g cm⁻³ was purchased from Daejung Company (Korea). The BF was added into the ethanol solution containing TEOS with various weight ratios: $m_{\text{TEOS}}/m_{\text{BF}} = 5$ and 10 mass%, in which ammonia solution was used to adjust the pH of the mixture between 8 and 9. Ethanol solution (C₂H₅OH) (99.7 %) and ammonia solution (NH₄OH) (25 %) are commercial products of Duc Giang Chemical Company (Vietnam). After 30 min, distilled water was dropped into above mixture and maintained at a temperature of 50 °C with magnetic stirring for 2 h. After that, the BF was filtered and washed by alcohol under low pressure to remove the residual TEOS. Finally, the BF was dried in a vacuum oven at 80 °C for 8 h.

Processing of composites

Treated or untreated BF (with 5 and 10 mass% of TEOS) was mixed with HDPE (containing 0.1 % DCP in

comparison with HDPE mass) at ratio $m_{\text{HDPE}}/m_{\text{BF}} = 60/40$ by melt-mixing method in a Haake intermixer at 185 °C for 5 min; the rotor speed was 80 rpm. Then, the above composites were pressed into thin sheets form using Toyo Seiki Press machine (Japan). They were subjected for 2 min at a pressure of 5 MPa at a temperature of 190 °C. Then, the final samples were cooled down and kept at room temperature at least 1 day before further characterization and analysis. In this study, the crosslinked matrix (using DCP in HDPE) is referred as XLPE. The series of XLPE/bamboo flour composites were investigated: a BF content of 40 mass% corresponds to an optimum of the final mechanical properties according to the literature [35]. The sample formulation is reported in Table 1: CA0 designates composites with untreated bamboo flour, and CA5 and CA10 correspond to composites containing bamboo flour, respectively, treated by 5 and 10 % of coupling agent (CA).

Methods

SEM

Scanning electron microscopy (SEM) was used for the observation of cryo-fractured surfaces obtained by breaking XLPE/BF composites frozen in liquid nitrogen. The surfaces were observed by using a JSM 6700F (JEOL, Japan) and equipped with field emission gun, at a voltage of 5 kV.

Differential scanning calorimetry

Differential scanning calorimetry (DSC) was performed on a Diamond DSC (PerkinElmer, USA). The samples between 10 and 15 mg were placed in aluminium pans. The crystallinity was evaluated from 20 to 150 °C at 20 °C min⁻¹.

Dynamic mechanical analysis

Dynamic mechanical analyses (DMA) were performed using an ARES set-up (Rheometric Scientific, USA). The measurements were taken from -140 to 100 °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 1 shows the morphology of composites via SEM images of cryo cuts. In Fig. 1a, a void between XLPE and BF can be observed. This discontinuity means that the compatibility between the matrix and the blends is quite poor. In Fig. 1b, for the composite CA5, the gap at the interface disappears. This image indicates an improvement in the compatibility between XLPE and BF due to the addition of 5 mass% TEOS during composite processing. In the case of the composite CA10, it is clear that 10 mass% TEOS decreases the adhesion between XLPE and BF.

Wang et al. [36] create coupling between wood flour and PP using maleic anhydride-grafted PP (MAPP) or silane as coupling agents. In the case of the use of silane, they highlighted that there is a critical ratio which is equal to 1.5 mass% for 50 mass% in wood fibres. This ratio is inferior to ours. For future study, it will be necessary to determine more precisely the critical ratio of TEOS in XLPE/BF composites. Above this ratio, the properties of the composites are unchanged or even damaged. Wu et al. [24] proposed several hypotheses: by-product formation, increase in the concentration of unreacted coupling agent and interference between interactions. They also determine the limits in coupling agent proportions from 2 to 8 mass%. Our limit ratio is consistent with these values.

Physical structure

Melting peaks of XLPE and 3 samples of composite CA0 are shown in Fig. 2. The degree of crystallinity for XLPE was calculated using the following equation:

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

where χ_c is the crystallinity ratio, ΔH_m is the measured melting enthalpy, and ΔH_∞ is the theoretical melting enthalpy of 100 % crystalline polyethylene taken here as 290 J g⁻¹ [37].

The thermal characteristics for each sample are indexed in Table 2.

The melting temperature of XLPE is 136 °C with a high and sharp peak. For CA0 composites, the melting temperature increases and reaches 140 °C. The melting of composites is higher than for XLPE. This shift can be explained by the following considerations: the presence of BF increases the thermal inertia because BF mainly composed

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

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

Fig. 1 SEM images of **a** CA0, **b** CA5 and **c** CA10 composites

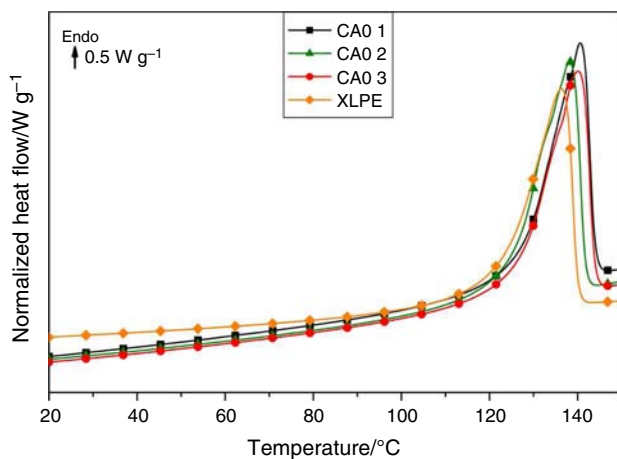
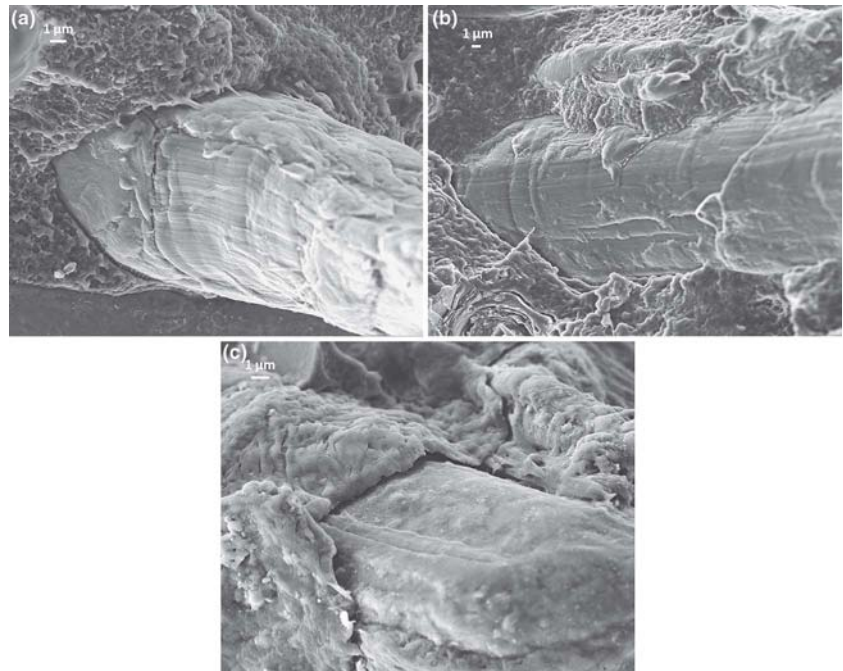


Fig. 2 DSC curve of XLPE and CA0 composites

Table 2 The thermal characteristics of bulk XLPE and CA0 composites

Samples	$T_m/^\circ\text{C}$	$\Delta H_m/\text{J g}^{-1}$	$\chi_c/\%$
XLPE (reference)	136	192	66
CA0 1	141	177	61
CA0 2	138	183	63
CA0 3	140	182	62
Average ^a	140 (1)	181 (3)	62 (1)

^a Numbers between brackets are the standard deviation

of cellulose is a better thermal insulator than XLPE. Moreover, the used heating rate is quite high so that the composites are not completely at equilibrium. The mean

value of crystallinity for the composites is 62 % which is close to the value of bulk XLPE: 66 %. It shows that BF has no significant influence on the crystallinity of XLPE.

Earlier studies involved treatment that contains polymeric sequences compatible with the matrix. In such conditions, the introduction of natural fillers may have an influence on the matrix's crystallinity [38]. Bouzouita et al. [39] showed the crystallinity of PP increased by 11 % when hemp fibres were introduced. This evolution was associated with the presence of transcrystallization at the interface between PP and fibres. Joseph et al. [40] had similar results using short sisal fibre-reinforced PP. The variation of crystallinity is dependent upon the treatment of the filler. Analogous data have been reported by Kumar et al. [41] for short bamboo fibres/LLDPE composites. It should also be noted that Suñol and Saurina [42] observed that the crystallinity of wood fibre/HDPE composites is independent from its environment. Along these lines, it is not surprising that in our composites, there is no modification of the crystallinity.

Mechanical relaxation modes

Matrix

In order to identify the role of the filler on the dynamic mechanical properties of XLPE/BF composites, the relaxation of XLPE matrix was first examined. Storage modulus G' and loss modulus G'' of HDPE and XLPE are illustrated, respectively, in Figs. 3 and 4.

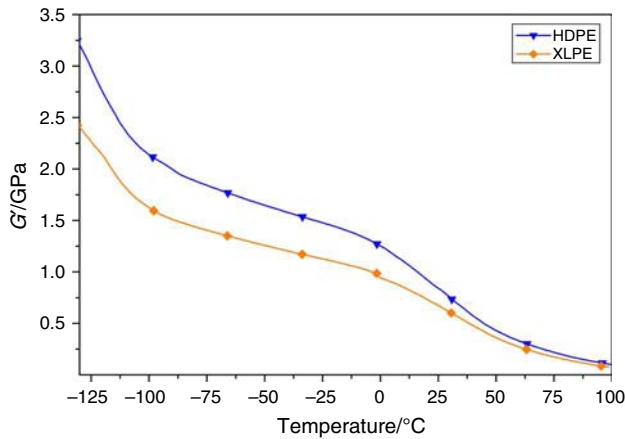


Fig. 3 Shear storage modulus G' of HDPE and XLPE

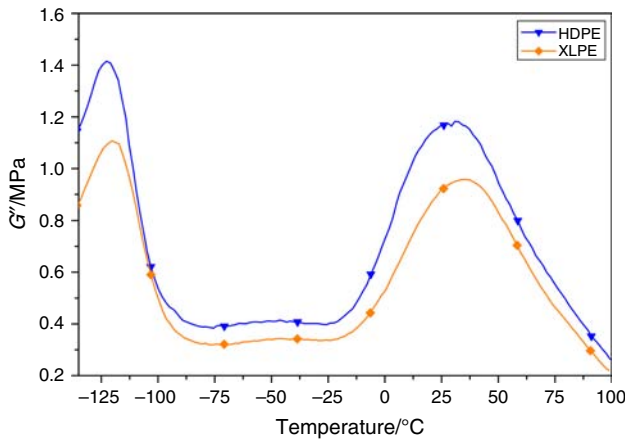


Fig. 4 Shear loss modulus G'' of HDPE and XLPE

Figure 3 highlights higher value of the conservative modulus in HDPE compared with XLPE: this observation is consistent with the fact that crosslinking with DCP restricts molecular mobility [43, 44]. Khonakdar et al. [43] also showed a decrease of 23 % of the bending properties due to crosslinking. In this study, the glassy plateau of XLPE is 25 % lower than the HDPE one i.e. in good agreement with previous findings. There is a decrease in the G' values with increasing temperature due to the relaxation of the mobile sequences between entanglements. The step located around 30 °C corresponds to the relaxation of sequences in the amorphous–crystalline interphase: this mode is generally designated as α_c . Note that the rubbery plateau has the same value for both XLPE and HDPE. Khonakdar et al. [43] reported a different behaviour: the rubbery modulus of XLPE decreases like the glassy modulus due to a decrease in crystallinity. Figure 4 shows the γ relaxation mode at -120 °C which is associated with the anelastic relaxation of polyethylene sequences of amorphous phases free of defects of both HDPE and XLPE. This intrinsic relaxation is

associated with the glass transition of polyethylene. Around -40 °C, a small peak, generally designated as β , reflects the mobility of sequences of the amorphous domains containing chemical defects such as crosslinks. At 40 °C, the α_c relaxation is associated with the reorientation of defects at the boundary of the crystalline phase of the matrix [45, 46]. It is interesting to note that the temperatures of all relaxations are independent from the chain architecture. This result is consistent with previous findings on a systematic study of polyethylene with different short-chain branching [47].

Influence of bamboo flour on the relaxation modes of composites

In this section, the influence of the introduction of BF with various percentages of coupling agent was studied by DMA.

Figure 5 presents the storage modulus G' of XLPE and XLPE/BF composites. Even for CA0, the composite has a higher storage modulus than for XLPE. Liu et al. [48] have also observed an improvement in the mechanical behaviour by introducing untreated BF in a HDPE matrix. The increase in the modulus of XLPE upon the introduction of untreated BF may be due to Vander Waals interactions between BF and the matrix. When BF is treated with 5 mass% of TEOS, the glassy modulus is improved, while the rubbery modulus remains analogous with the one of CA0. Above 40 °C, the influence of TEOS is negligible: it indicates that the introduction of BF untreated or treated does not restrict the mobility of XLPE chains in the rubbery state. In the case of CA10, the glassy plateau is lower than CA5. It might indicate that remaining TEOS forms nodules in the matrix. DMA allows us to determine the critical content of coupling agent. These results are consistent with structural observations from SEM. The molecular origin of the decrease in the conservative modulus above 5 % CA might be by-products formation and an increase in unreacted coupling agent. Consequently, the increase in CA acts against the improvement in mechanical properties.

The dependence of G'' on temperature for XLPE and the XLPE/BF composites is shown in Fig. 6. Then, the γ , β and α_c relaxations are resolved. The temperatures of the peaks are not significantly modified by the introduction of BF. In other words, there is no plasticization due to the BF introduction. It is important to mention here that 5 mass% of TEOS appears as optimum since, for 10 mass% of TEOS, the magnitude of the γ mode decreases. Consequently, the impact strength decreases.

The γ relaxation located at -120 °C corresponds to the viscoelastic energy loss associated with the glass transition of the amorphous phase free from chemical defects. A broadening of the γ peak appears around -80 °C. This broadening increases by treating BF with 5 mass% of

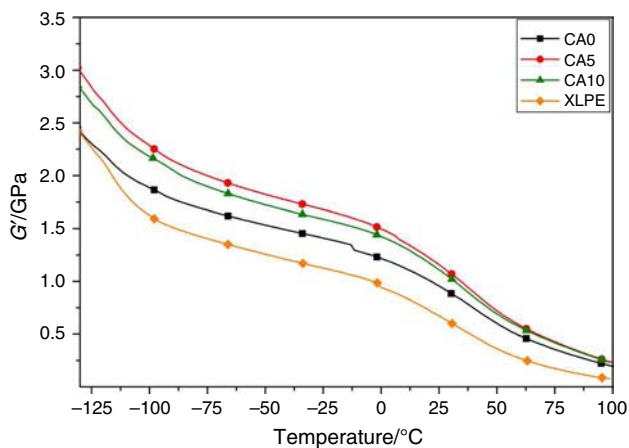


Fig. 5 Storage modulus G' for XLPE and XLPE/BF composites

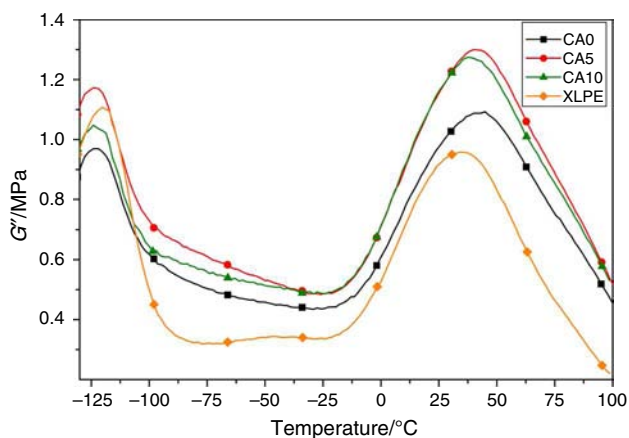


Fig. 6 Loss modulus G'' for XLPE and XLPE/BF composites

TEOS: it indicates that this phenomenon is dependent upon the presence of bamboo flour within the amorphous XLPE phase. The amorphous phase containing BF is segregated. A bump is observed at -40°C in XLPE. According to previous data on polyethylene with different short-chain branching, this event has been assigned to the mobility in amorphous regions of XLPE with chemical defects. The α_c mode, situated around 40°C , has been associated with the mobility in the interfacial regions at the periphery of crystallites. Its magnitude is higher in all XLPE/BF composites showing that there is also a contribution of the mobility at the polymer/BF interface.

Conclusions

The evolution of thermal and mechanical properties of XLPE/bamboo flour (BF) composites, observed by a combination of DSC/DMA analyses, is discussed using as reference bulk XLPE. SEM observations show the

existence of a continuity of matter at the interface for percentages of TEOS (CA) close to 5 mass%. For 10 mass% of TEOS, a void is observed between XLPE and BF. The study of the physical structure performed by DSC shows the crystallinity was unmodified upon the introduction of fillers. The absence of transcristallinity is important for preserving the ductility of composites.

DMA analyses show that the introduction of BF improves the glassy modulus owing to Vander Waals interactions. This effect is amplified in XLPE/BF due to polar interactions created by the coupling agent. The amount of 5 mass% of CA corresponds to an optimum of viscoelasticity. Above this critical content, the storage modulus decreases due to a bad interphase as observed by SEM. The anelastic relaxation modes of XLPE/BF composites are located at the same temperatures than for the XLPE matrix showing the absence of plasticization independently from the coupling agent content.

The γ relaxation located at -120°C corresponds to the viscoelastic energy loss associated with the glass transition of the amorphous phase free from chemical defects. It exhibits, around -80°C , a shoulder characteristic of the presence of bamboo flour within the amorphous XLPE phase: it indicates a segregation of the amorphous phase containing BF. The α_c mode, situated around 40°C , has been associated with the mobility in the interfacial regions. In XLPE, this molecular mobility is located at the periphery of crystallites. Since its magnitude of the α_c mode is higher in all XLPE/BF composites, the mobility at the periphery of BF is also involved.

Finally, combined DSC/DMA analyses allow us to identify the molecular origin of the anelastic properties of XLPE/BF composites as a function of CA content.

Acknowledgements The work was done in the framework of the International Associated Laboratory "FUNCTIONAL COMPOSITE MATERIALS" (FOCOMAT) supported by CNRS and VAST. The financial support of Assystem and ANRT is greatly acknowledged.

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