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Tung-Lin Wu, Yi-Chi Chien, Tsai-Yung Chen and Jyh-Horng Wu*

The influence of hot-press temperature and cooling rate on thermal and physicomechanical properties of bamboo particle-polylactic acid composites

Abstract: Extrusion and injection moldings are standard processes for fabricating natural fiber-reinforced plastic composites, but both processes are generally not suitable for production of large-size pieces and products with high loadings of lignocelluloses. In this study, a medium-density bamboo plastic composite (BPC) was completely and successfully manufactured from bamboo fibers and polylactic acid (PLA) in the ratio of 1:1 by the flat-platen pressing process. The effects of pressing and cooling parameters on the thermomechanical properties of the BPC_{PLA} have been investigated. The BPC_{PLA} prepared at temperatures >180°C and cooling rates >10°C min^{-1} exhibited superior mechanical properties and matrix crystallinity. Under these conditions, a stronger interaction between the filler and the polymer matrix occurs and the mobility of the molecular chains at the interface decreases, which leads to a higher stiffness of the composite.

Keywords: bamboo particle, bamboo plastic composite (BPC), green composite, polylactic acid, processing parameter, thermomechanical property, wood plastic composite (WPC)

***Corresponding author: Jyh-Horng Wu**, Department of Forestry, National Chung Hsing University, Taichung 402, Taiwan, Phone: +886-4-22840345, Fax: +886-4-22851308, e-mail: eric@nchu.edu.tw
Tung-Lin Wu: Department of Forestry, National Chung Hsing University, Taichung 402, Taiwan
Yi-Chi Chien: Department of Forestry, National Chung Hsing University, Taichung 402, Taiwan
Tsai-Yung Chen: Department of Forestry, National Chung Hsing University, Taichung 402, Taiwan

Introduction

Bamboo, a perennial woody grass belonging to the Gramineae family, is widely distributed across Asia (Scurlock et al. 2000), where it is extensively used as a raw material for handicraft, furniture and other constructions. Large amounts of bamboo shavings and sawdust are available as by-products of the bamboo-processing industry. Therefore, one of the challenges is to also utilize the by-products. The utilization of bamboo fibers for reinforcement in biocomposites could be one of the solutions. Bamboo fibers have excellent physical properties (Yang et al. 2009; Yu et al. 2011; Qu et al. 2012).

The development of green composites from biodegradable polymers and natural fibers is of great interest. Natural fibers such as wood, bamboo, flax, ramie, jute, kenaf and hemp fibers can be a renewable and cheaper substitute for synthetic fibers and have numerous advantages: low cost, low density, high toughness, good specific strength properties and biodegradability (Xu et al. 2001; Balasuriya et al. 2002; Zhang et al. 2002; Oksman et al. 2003). In the context of the wood industry, one speaks about wood plastic composites (WPCs). WPCs and related composites have remarkable mechanical properties and long durability (Lee et al. 2010; Kumar et al. 2011; Devi et al. 2012).

If the plastic component is biodegradable, the WPCs are environmentally benign: they degrade in composting processes and do not emit toxic or noxious components (Shogren et al. 2003; Graupner et al. 2009; Krouit et al. 2010; Gregorova et al. 2011). Among all of the biodegradable polymers, polylactic acid (PLA) is of increasing commercial interest. Their aliphatic polyester chains are biodegradable and compostable. PLA-based composites have relatively high strength and can be easily processed with standard equipment (Garlotta 2001). PLA is a transparent, crystalline and brittle polymer with a relatively high melting point. Because of its brittleness, PLA generally needs to be reinforced for practical applications (Huda et al. 2006; Ikeda et al. 2008; Cheng et al. 2009).

During the past decades, natural fibers have replaced many conventional inorganic fillers or reinforcements in various composite applications. Plastic composites based on bamboo fibers (bamboo plastic composites, BPCs) are highly estimated as they have a high strength and growth

rate in comparison with other natural fibers (Mi et al. 1997; Fujii and Okubo 2003; Lee and Ohkita 2004). BPC_{PLA} investigations hitherto focused primarily on the effects of fiber type, fiber content, fiber modification and functional additives on the mechanical and thermal properties of the composites (Lee et al. 2004; Lee and Wang 2006; Tokoro et al. 2008). However, there is little information available on the effects of the pressing process on the thermal and physicomechanical properties of BPC_{PLA}s.

The present paper would like to fill this gap, and it focuses on the effects of the press temperature and cooling rate on the BPC_{PLA} properties. To the best of our knowledge, this is the first report to address this issue.

Materials and methods

Preparation of the bamboo particles and polymers

Dried shavings from 3-year-old kei-chiku bamboo (makino bamboo; *Phyllostachys makinoi* Hayata) were provided by the local bamboo-processing factory. Bamboo particles were prepared by hammer-milling and sieving; particles between 30 and 60 mesh were investigated. PLA (NCP0001; NatureWorks®) was purchased from the Wei Mon industry Co., Ltd. (Taipei, Taiwan) and had a melt flow index of 4–8 g 10 min^{-1}, density of 1250 kg m^{-3} and melting temperature (T_m) of 140–150°C. The PLA pellets were ground in an industrial mill to reduce their particle size to less than 20 mesh before composite processing.

Composite processing

The flat-platen pressing process was applied according to our previous paper (Hung and Wu 2010; Lee et al. 2010). The weight ratio of the oven-dried bamboo particles (moisture content <3%) to PLA powder was 1:1. The target density of the BPC_{PLA}s was 780±30 kg m^{-3}. The dimensions of the BPC_{PLA}s fractions were 300 mm×200 mm with thicknesses of 4 and 12 mm. The BPC_{PLA}s were produced in a two-step pressing process as follows: (1) hot pressing (2.5 MPa) at 165, 180, 195 or 210°C for 12 min and (2) finishing by cold pressing (2.5 MPa) at rates of 0.4, 10, 15 or 40°C min^{-1} until the core temperature of the BPC_{PLA}s decreased to 40°C.

Determining the composite properties

The density, water absorption (WA), thickness swelling (TS), flexural properties and internal bond (IB) strength were determined according to the Chinese National Standard CNS 2215. The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined by the three-point static bending test with a loading speed of 10 mm min^{-1} and with a span of 180 mm (specimen size: 230 mm×50 mm×12 mm). The IB strength was tested on specimens with dimensions of 50 mm×50 mm×12 mm at a tensile speed of 2 mm min^{-1}. The samples were conditioned at 20°C and 65% relative humidity for 2 weeks before testing.

The thermal properties of the BPC_{PLA}s were characterized by differential scanning calorimetry (DSC) by means of the instrument DSC-7 (Perkin-Elmer, Buckinghamshire, UK). The samples (ca. 2–3 mg) were encapsulated in aluminum pans and heated from 40 to 180°C at a heating rate of 10°C min^{-1} under N_2 flow (20 ml min^{-1}). The glass transition temperature (T_g), melting temperature (T_m) and heat of melting (ΔH_m) were determined for each of the composite samples. The crystallinity of the PLA matrix (X_c) was determined according to Islam et al. (2010) with the formula $X_c\ (\%) = \Delta H_m / \varphi \Delta H_m^{\ 0} \times 100$, where ΔH_m is the experimental heat of fusion determined from the DSC measurement, $\Delta H_m^{\ 0}$ is the assumed heat of fusion of the fully crystalline PLA (93 J g^{-1}) and φ is the weight fraction of PLA in the composites.

The dynamic mechanical properties of the BPC_{PLA}s were measured in a single-cantilever bending mode (DMA 8000, Perkin-Elmer) at a heating rate of 2°C min^{-1} and a frequency of 1 Hz. The storage modulus (E'), loss modulus (E'') and loss tangent (tan δ) were recorded over a temperature range of 0–100°C. The dimension of the sample was 30 mm×10 mm with a thickness of 4 mm. The bamboo particles and the PLA matrix in the composites were examined by scanning electron microscopy (SEM) with a Hitachi TM-1000 (Tokyo, Japan) scanning electron microscope at 15 kV.

Results are the mean (±SD) of n=5 samples. The significance of difference was calculated by Scheffe's test; $P<0.05$ were considered to be significant.

Results and discussion

Mechanical properties

The flexural properties of the BPC_{PLA}s with different press temperatures and cooling rates are presented in Figure 1. As shown in the figure, the MOR data were increased from 16.1 to 26.1 MPa as the press temperature was elevated from 165 to 210°C, but the changes were statistically not significant between 180 and 210°C (Figure 1a). The MOE values were 1.9, 2.4, 2.6 and 3.0 GPa at 165, 180, 195 and 210°C, respectively. Accordingly, the flexural properties, especially the flexural MOEs, of the BPC_{PLA}s can be improved at higher press temperatures. Similar results were reported by Bernard et al. (2011).

The cooling rates do not have essential effects on the flexural properties of the BPC_{PLA}s (Figure 1b) in the range of 10, 15 and 40°C min^{-1}. However, when the cooling rate was lowered from 10 to 0.4°C min^{-1}, the MOR and MOE data decreased from 25.6 MPa and 2.7 GPa to 8.2 MPa and 2.1 GPa, respectively. Apparently, low cooling rates deteriorate the interface properties between the bamboo particles and the PLA matrix.

The IB of the BPC_{PLA}s also varies as a function of press temperature and cooling rate (Figure 2). Namely, the IBs

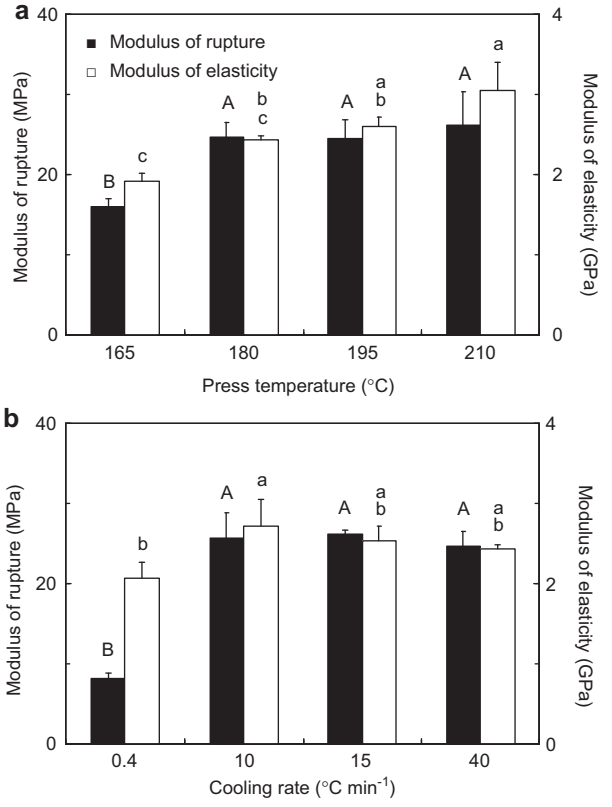

Figure 1 Effects of press temperature (a) and cooling rate (b) on the modulus of rupture (MOR) and modulus of elasticity (MOE) of the BPC_{PLA}s. Values are means±SD (n=5). Bars with different capital and lowercase letters indicate significant differences in the MOR and MOE values, respectively (P<0.05).

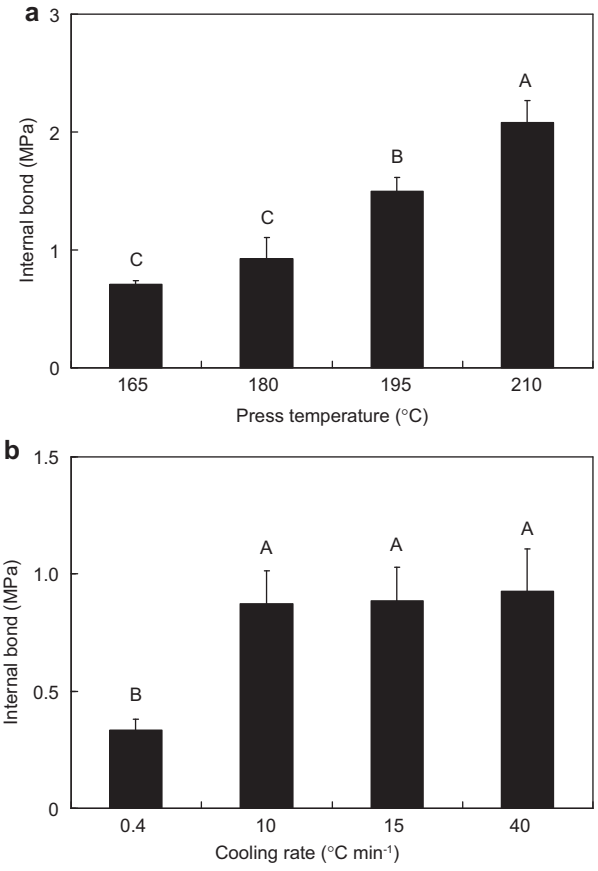

Figure 2 Effects of press temperature (a) and cooling rate (b) on the internal bond (IB) of BPC_{PLA}s. Values are means±SD (n=5). Bars with different capital letters indicate significant differences (P<0.05).

of the BPC_{PLA}s increased from 0.7 to 2.1 MPa as the press temperature was elevated from 165 to 210°C (Figure 2a). However, the IB exhibits no dependence of the cooling rates between 10 and 40°C min^{-1} (Figure 2b). However, when the cooling rate was lowered from 10 to 0.4°C min^{-1}, the IB was remarkably decreased from 0.9 to 0.3 MPa. Lee et al. (2010, 2012) demonstrated that the MOR, MOE and IB data of WPCs are in the range of 8.5–25.3 MPa, 1.0–2.4 GPa and 0.7–2.2 MPa, respectively. Accordingly, the BPC_{PLA}s have even better mechanical properties if produced under optimal conditions.

Thermal properties

Thermal properties, including the T_g, T_m and ΔH_m, can be directly measured by DSC (Table 1). There are no significant differences in the T_g of the PLA matrix in the temperature range from 165 to 195°C. However, at 210°C the T_g decreased from 62.0 to 58.1°C. Similarly, the T_m also decreased at 210°C. Moreover, the ΔH_m and X_c were significantly increased at higher temperatures. For a semicrystalline polymer composite like PLA, the matrix crystallinity influences the mechanical properties (Pei et al. 2010). This is the reason why the data presented in Figure 1a improved at higher temperature. However, only the ΔH_m and X_c data of the PLA matrix were dependent on press temperature and cooling rates (Table 1). In the latter case, mainly between 10 and 0.4°C min^{-1}, the ΔH_m and X_c increased from 3.0 J g^{-1} and 12.5% to 35.4 J g^{-1} and 44.0%, respectively. This finding confirms the known fact that crystallinity and cooling rate are closely interrelated. The mobility of polymer chains is impeded at a high cooling rate, thereby limiting the ability of the chains to form regular structures (spherullites) within the matrix (Gao and Kim 2000). In general, the lower the cooling rate, the higher are the crystallinity indices and mechanical properties, but this statement is not valid for the extremely low cooling rate of 0.4°C min^{-1}. The highest crystallinity (44.0%) was observed at this cooling rate and also the lowest flexural properties (Figure 1b) and IB data (Figure 2b).

Press temp. (°C)	Cooling rate (°C min^{-1})	T_g (°C)	T_m (°C)	ΔH_m (J g^{-1})	X_c (%)
165	40	62.1±0.3^{A}	147.0±2.8^{AB}	1.7±0.3^{C}	2.2±0.3^{C}
180	40	62.0±1.1^{A}	151.0±0.2^{A}	1.8±0.1^{C}	2.7±0.1^{C}
195	40	62.0±0.2^{A}	145.5±0.2^{BC}	5.1±0.3^{B}	7.9±0.5^{B}
210	40	58.1±1.1^{B}	142.5±0.7^{C}	8.3±0.6^{A}	21.0±1.5^{A}
180	0.4	ND	166.1±13.7^{a}	35.4±0.7^{a}	44.0±0.9^{a}
180	10	62.2±2.3^{a}	150.3±0.7^{a}	3.0±0.5^{bc}	12.5±2.2^{b}
180	15	60.2±2.5^{a}	146.6±1.9^{a}	3.6±0.5^{b}	4.7±0.7^{c}
180	40	62.0±1.1^{a}	151.0±0.2^{a}	1.8±0.1^{c}	2.7±0.1^{c}

Table 1 Effects of press temperature and cooling rate on thermal properties of BPC_{PLA}s. Values are means±SD (n=5). Different letters within a column indicate significant difference at P<0.05. ND, not determined.

The substantial increase in the degree of crystallinity caused matrix shrinkage, severe cracking and initiation of void formation (Figure 3a, b). Jabarin and Lofgren (1994) and Beg and Pickering (2008) observed similar trends for surface cracks on weathered WPCs and neat plastics, which were ascribed to elevated polymer crystallinity.

Water absorption and thickness swelling

The WA and TS data of the BPC_{PLA}s as a function of process parameters are presented in Table 2. At different press temperatures, the WA after immersion for 24 h ranged from 42.2% to 43.8%, but there were no significant differences among the data. Similarly, the TS data oscillating between 2.6% and 3.3% were generally consistent with the trend of WA. In contrast, the WA and TS data were affected by the low cooling rate at 0.4°C min^{-1}, at which WA was 69.5% and TS was 16.6%. Compared with a previous report (Lee et al. 2009), the TS and WA data of all BPC_{PLA}s were less than those of Kenaf-PC_{PLA} produced by the carding process at 200°C. Das et al. (2000) pointed out that the water in composites could be stored in the cell walls, the cell lumens and the voids between the lignocellulose and polymer matrix, with water located in the cell wall causing the greatest dimensional change. It can be supposed that in the present study the majority of the water was located primarily in the cell lumen and the voids, except for the BPC_{PLA}s processed at a cooling rate of 0.4°C min^{-1}. In the latter case, the water is probably in the cell wall, and this is responsible for the dimensional instability in immersion tests.

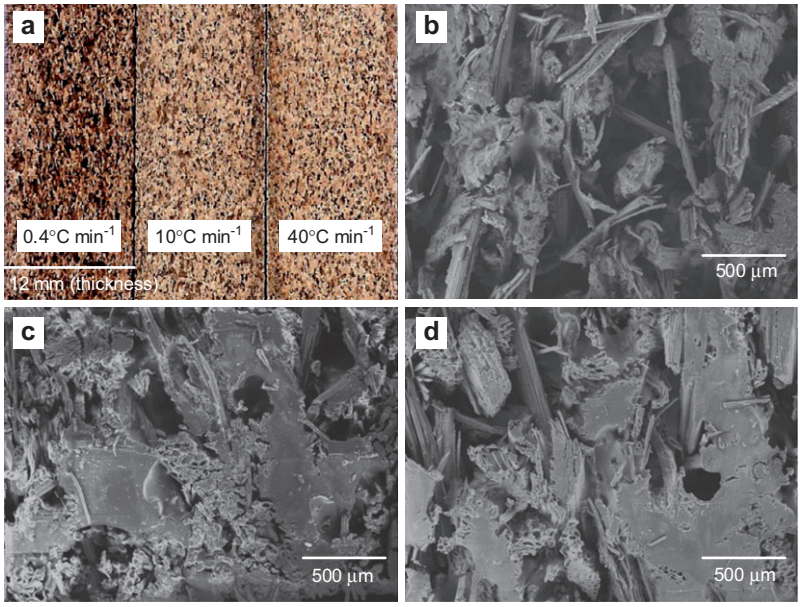

Figure 3 Surface images (a) and SEM micrographs of BPC_{PLA}s processed at cooling rates of 0.4 (b), 10 (c) and 40°C min^{-1} (d).

Press temp. (°C)	Cooling rate (°C min^{-1})	Density (kg m^{-3})	WA (%)		TS (%)	
			2 h	24 h	2 h	24 h
165	40	761±7^{A}	39.3±1.2^{AB}	42.6±0.6^{A}	3.1±0.2^{A}	3.3±0.1^{A}
180	40	776±5^{A}	41.3±3.2^{A}	42.4±2.9^{A}	2.5±0.4^{AB}	2.6±0.3^{B}
195	40	757±11^{A}	39.5±1.1^{AB}	43.8±1.1 ^{A}	2.3±0.2^{B}	2.9±0.1^{AB}
210	40	762±36^{A}	35.9±2.5^{B}	42.2±2.3^{A}	2.0±0.5^{B}	3.0±0.5^{AB}
180	0.4	774±12^{a}	65.4±4.8^{a}	69.5±3.1^{a}	16.2±0.7^{a}	16.6±0.6^{a}
180	10	782±17^{a}	45.2±3.8^{b}	46.9±3.6^{b}	3.1±0.2^{b}	3.4±0.2^{b}
180	15	803 ± 3^{a}	44.6±2.3^{b}	45.6±2.5^{b}	3.0±0.7^{b}	3.1±0.4^{b}
180	40	776±5^{a}	41.3±3.2^{b}	42.4±2.9^{b}	2.5±0.4^{b}	2.6±0.3^{b}

Table 2 Effects of press temperature and cooling rate on water absorption (WA) and thickness swelling (TS) of BPC_{PLA}s. Values are means±SD (n=5). Different letters within a column indicate significant difference at P<0.05.

Dynamic mechanical properties of BPC_{PLA}s

The storage modulus (E') is sensitive to the interfacial adhesion between the lignocellulose and the polymer matrix (Hong et al. 2008). Figure 4 is a compilation of data obtained by dynamic mechanic analysis (DMA) showing the plots of the dynamic flexural E' and loss tangent (tan δ) as a function of press temperature (Figure 4a) and cooling rates (Figure 4b). The E' values of all composites decreased with increasing temperature because of the increasing chain mobility of the PLA matrix. A precipitous drop in E' can be expected near the T_g of the PLA (ca. 60°C) (Lee et al. 2009). As illustrated in Figure 4a, larger storage moduli occur at higher press temperatures, in agreement with data of static mechanical properties. The sample hot-pressed at 210°C exhibited the highest E'. The increase in E' of semicrystalline thermoplastic composites can be attributed to several factors, e.g., the nature of the crystalline-amorphous interface, filler loading and morphology (Spinu and McKenna 1994; Gregorova et al. 2011). All these factors have an impact on the interfacial adhesion between the filler and the polymer matrix and affect the physicomechanical and thermal properties of the composites.

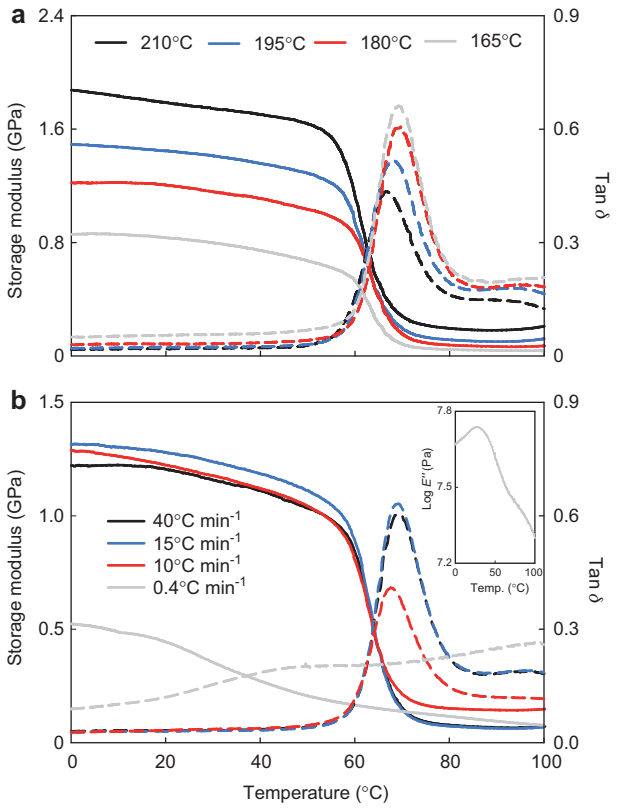

Figure 4 Data calculated from dynamic mechanical measurements (DMA). Effects of press temperature (a) and cooling rate (b) on the storage modulus (solid line) and tan δ (dotted line) of BPC_{PLA}s.

Tan δ, a measure of material-related damping properties, is an indication of molecular motions in the materials, which contributes at the interface to damping or energy dissipation (Hong et al. 2008; Chen et al. 2012). As shown in Figure 4a, the intensity of the tan δ peak decreased with increasing press temperatures, confirming the stronger interaction between the different components at higher press temperatures. Moreover, the E' data are essentially the same at various cooling rates (Figure 4b). An exception is, however, the cooling rate of 0.4°C min^{-1}, where E' suffered a 66% decrease at ambient temperature. This finding is in agreement with MOR (Figure 1b) and IB (Figure 2b) data. The intensity of tan δ generally decreased with lowering cooling rates, and there was no specific PLA T_g peak for the composite processed at a cooling rate of 0.4°C min^{-1}. Alternatively, the loss modulus response (log E'') of this composite revealed a significant β relaxation process (ca. 25°C) of lignocelluloses, which is attributed to the micro-Brownian motions of lignin and/or lignin-hemicellulose complexes plasticized with residual

moisture (Jebrane et al. 2011). This result is interpreted as the formation of spot welding-like discontinuous layers of the PLA matrix at a low cooling rate. This is the reason why the damping profile of this composite is so different.

Surface images and SEM micrographs in Figure 3 visualize the influence of cooling rates. As pointed out earlier, the BPC_{PLA} with 0.4°C min^{-1} has the most voids, which contribute to a higher wettability and to reducing the interfacial adhesion between the bamboo particle and the PLA matrix.

Conclusions

The thermomechanical characteristics of the BPC_{PLA}s remarkably varied depending on the process parameters. The crystallinity of the PLA in the BPCs increased from 2.2% to 21.0% as the press temperature was elevated from 165 to 210°C. In general, the flexural strength and modulus of the BPC_{PLA}s also increased at higher press temperatures, but the cooling rates had no significant influence on these data. However, at the extremely low cooling rate of 0.4°C min^{-1}, the crystallinity of the PLA was as high as 44.0%. At this cooling rate, MOR, MOE and IB data were the lowest (8.2 MPa, 2.1 GPa and 0.3 MPa, respectively). The SEM micrographs reveal many voids for the composites produced at the lowest a cooling rate. The high PLA crystallinity is supposed to be responsible for the formation of cracks, which deteriorate the physicomechanical properties.

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