ORIGINAL ARTICLE

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Mechanical and interfacial properties of plastic composite panels made from esterified bamboo particles

Abstract In this study, different extents of acetylated and butyrylated bamboo particle/plastic composites (BPPC) were produced by the flat-platen pressing process. The effect of esterification on mechanical and interfacial properties of BPPC was evaluated by a universal testing machine, X-ray diffractometer (XRD), ^{13}C CP/MAS nuclear magnetic resonance (NMR) spectrometer, and scanning electron microscope (SEM). The results showed that the internal bond (IB) and wood screw-holding strength of BPPC were significantly increased after acetylation, even though the weight gain of acetylated bamboo particles was only 2%. In addition, SEM micrographs revealed that acetylated bamboo particles were effectively trapped by the polymeric matrix on the IB-fractured surface of BPPC. These results indicate that the interfacial interaction between the bamboo particle and the polymeric matrix can be enhanced through acetylation.

Key words Bamboo particle/plastic composites · Mechanical properties · Interfacial properties · Esterification · X-ray diffraction

Introduction

In tropical areas, especially Asian countries, bamboo is a basic natural resource and plays a great role in the people's daily life and culture because of its rapid growth rate, excellent flexibility, and excellent machinability.[1,2] Today, bamboo is widely used as a raw material for construction, furniture, pulping, and handicraft works, resulting in many by-products (bamboo shavings and sawdust) from the bamboo-processing industry. Therefore, one of the unprecedented challenges in the bamboo products industry is how to improve the properties and performance of bamboo products by using the woody residues and thus recycling them. In addition to the many uses of bamboo, manufacturing of thermoplastic composites is becoming popular as a promising value-added option.[3–6] It is forecast that the demand for natural fiber/plastic composites will grow by about 50% and 60% per year for automotive applications and for building construction products, respectively.[7]

Using natural fibers together with plastic has many advantages, such as low density, low equipment abrasiveness, high stiffness and strength, relatively low cost, and good biodegradability.[8] However, in spite of these advantages, the use of natural fibers in industrial practice is limited by difficulties associated with surface interactions. The main drawback of natural fiber/plastic composites is the incompatibility between the hydrophilic lignocellulosics and the hydrophobic thermoplastic polymers. Many problems have been identified, including poor fiber dispersion caused by intermolecular hydrogen bonding,[9] and wide polarity differences of the surfaces that prevent efficient polymer/fiber bonding.[10,11] Poor interfacial adhesion leads to composites with poor mechanical properties.[12,13] Thus, over the past two decades, several physical and chemical approaches have been used to overcome these problems. One of these approaches is chemical modification of lignocellulosic materials to both increase their hydrophobicity[14] and improve their dimensional and thermal stability.[15,16]

Another problem is that most wood flour (or wood fiber)/plastic composites (WPC) are currently manufactured by injection molding or extrusion. However, in general, making composites with large particles is difficult with both processes. It is well known that the influence of particle size on the mechanical strength, especially on bending strength, of WPCs is great. A previous study showed that a WPC with large wood particles could be successfully prepared by a flat-platen pressing process, and it showed better strength properties than that with small particles.[17] Thus, in the present study, bamboo particle/high-density polyethylene (BP/HDPE) composites were produced by flat-platen pressing. In addition, the prepared composites with unmodified and esterified bamboo particles were characterized in terms of their morphology and mechanical

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behavior. Furthermore, the basic properties of esterified bamboo particles were investigated by various spectrometric techniques.

Experimental methods

Materials

Dried shavings of 3-year-old kei-chiku bamboo (makino bamboo; *Phyllostachys makinoi* Hayata) were provided by the local bamboo-processing factory and were chipped to particles (16–24 mesh) without any pretreatment. HDPE (LH901) was purchased from USI Co. (Kaohsiung, Taiwan) with a melt index of 0.95 g/10 min and density of 0.953 g/cm^3. Acetic anhydride, butyric anhydride, and dimethylformamide (DMF) were purchased from Sigma Chemical (St. Louis, MO, USA). The other chemicals and solvents used in this experiment were of analytical grade.

Esterification

Bamboo particles (1.2 kg) were immersed in a DMF solution containing 20 mM acetic anhydride (acetylation) or butyric anhydride (butyrylation) with potassium carbonate (1.1 mM) as a catalyst. The reaction was conducted at 100°C with stirring for 1–8 h to obtain bamboo materials esterified in different degrees. At the end of modification, the esterified bamboo particles were washed with acetone and then Soxhlet-extracted using acetone for 6 h to remove the residues of acetic acid or butyric acid. Finally, the esterified bamboo particles were dried at 105°C for 12 h, and the weight percent gain (WPG) was calculated as follows: $WPG = 100(M_1 - M_0)/M_0$, where M_0 and M_1 are the oven-dried weights of the bamboo particle before and after esterification, respectively.

Composite processing

The flat-platen pressing process used for manufacturing bamboo particle/plastic composites (BPPC) followed the process of Chen et al.[17] The weight ratios of oven-dried bamboo particle and HDPE powder were 0/100, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, and 80/20 (w/w), respectively. The target density of BPPC was 0.85 ± 0.05 g/cm^3 with a format of $30 \times 20 \times 1.2$ cm, and three boards were prepared for each weight ratio. All BPPC were produced in a two-step pressing process: first, hot pressing at 200°C for 8 min, and then finishing by cold pressing until the temperature of BPPC was down to 25°C (~12 min).

Determination of composite properties

To determine the properties of the composites, several tests, including flexural properties [modulus of rupture (MOR) and modulus of elasticity (MOE)], internal bond strength, and wood screw-holding strength were carried out according to Chinese National Standard (CNS) 2215. A specimen size of $23 \times 5 \times 1.2$ cm was used to evaluate MOR and MOE by the three-point static bending test with a loading speed of 10 mm/min and span of 18 cm. Specimens $5 \times 5 \times 1.2$ cm were used for internal bond strength and wood screw-holding strength at a tensile speed of 2 mm/min. The samples were conditioned at 20°C and 65% relative humidity for 2 weeks before testing. At least five specimens of each blend were tested.

X-ray diffraction measurement

X-ray diffractograms were obtained with an MAC science MXP18 instrument (Japan), and bamboo samples were prepared by powdering. The diffraction patterns were measured from $2\theta = 2°$ to $35°$ using $CuK\alpha_1$ radiation, at 40 kV and 30 mA. The crystallinity index (CrI) of the bamboo particle was calculated according to the following equation: $CrI (\%) = 100(I_{200} - I_{am})/I_{200}$,[18] where I_{200} is the maximum intensity of the 200 lattice reflection of the cellulose crystallographic form at $2\theta = 22°$ and I_{am} is the intensity of diffraction of the amorphous material at $2\theta = 18°$.

Solid-state CP/MAS ^{13}C-NMR analysis

Solid-state samples were examined by cross-polarization magic angle spin (CP/MAS) ^{13}C-nuclear magnetic resonance (NMR). The spectra were recorded on a Bruker DSX-400WB FT-NMR spectrometer (Germany) at a frequency of 100 MHz. Chemical shifts were calculated relative to tetramethylsilan (TMS) for NMR control.

FTIR-ATR spectral measurements

Fourier transform-infrared (FTIR)-attenuated total reflectance (ATR) spectra of unmodified and esterified bamboo samples were recorded on a Spectrum 100 FTIR spectrometer (Perkin-Elmer, USA) equipped with a DTGS (deuterated triglycine sulfate) detector and a MIRacle attenuated total reflectance (ATR) accessory (Pike Technologies, USA). The spectra were collected by co-adding 64 scans at a resolution of 4 cm^{-1} in the range from 650 to 4000 cm^{-1}.

Scanning electron microscopy

Scanning electron microscopy (SEM) was used to examine the morphology of bamboo particles and plastics in composites. After the internal bond test, the fracture surfaces of composites were dried and then sputtered with gold. A JEOL JSM-6330F scanning electron microscope (Japan) with field emission gun and accelerating voltage of 2.8 kV was used to collect SEM images for the composite specimen. The samples were viewed perpendicular to the fractured surface.

Analysis of variance

All results are expressed as mean ± SD (standard deviation). The significance of difference was calculated by Scheffe's test,[19] and values < 0.05 were considered to be significant.

Results and discussion

Effects of the bamboo particle content on mechanical properties of BPPC

The various mechanical properties present in the different composites are shown in Table 1. The MOR of all samples ranged from 9.9 to 25.6 MPa. The highest bending strength was found in the BPPC with 20% or 30% bamboo particle (BP), and both composites were not significantly different from the neat plastic (25.6 MPa). However, when the amount of BP was changed from 30% to 60%, the MOR slightly decreased from 23.1 to 17.6 MPa. This decline in MOR at more than 30 wt% BP may be attributable to aggregation of the woody materials, as reported by previous authors.[20,21] Moreover, a drastic decrease in bending strength was observed when the BP content was more than 60% (see Table 1). A possible explanation for this could be related to the lack of adequate interfacial adhesion between the bamboo particle and the plastic at a high BP loading.[22] Thus, the strength decreased significantly with increased content of bamboo material at 60%–80% in the BPPC. Similarly, the internal bond and the wood screw-holding strengths exhibited the same trend for the tested formulations in Table 1. With the MOE, the modulus increased as the amount of BP was increased up to 30% and then leveled off or remained constant up to 60% (~1.5 GPa). Once the BP loading exceeded 60%, however, a significant decrease in the modulus was observed. A similar effect has also been shown in wood particle–HDPE composite.[17] Accordingly, for mechanical and bamboo residue utilization considerations, the BPPC with 60 wt% of bamboo particles should be a suitable formulation.

Effects of acetylation on mechanical properties of BPPC

The static bending properties of BPPC with 60 wt% of acetylated or untreated bamboo particles are shown in Fig. 1. No significant differences in MOR were found among all the tested samples. The MOR of all acetylated BPPC (WPG 2–17) is around 19 MPa, which is similar to that of the unmodified one. However, the MOE of BPPC with acetylated BP is slightly lower than that of an unmodified sample, especially at the highest WPG. The modulus decreases significantly, from 1.8 to 1.6 GPa, when increasing the WPG of acetylated BP from 4 to 17. Similar results were reported by Pasquini et al.[23] This phenomenon should be influenced by the nature of lignocelluloses after acetylation. Figure 2 shows the evolution of X-ray diffraction patterns of acetylated bamboo particles. The results showed two diffraction peaks for $2\theta = 22°$ and 15° before treatment, by which the crystallinity index (CrI) calculated is 58.5%. The former peak is attributed to the 200 plane of the crystal lattice and
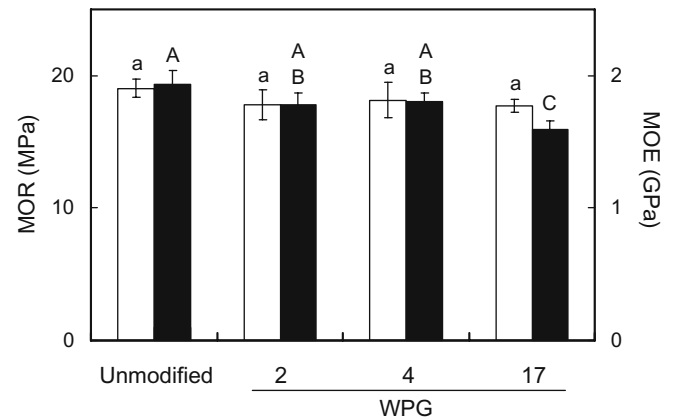
Fig. 1. Effect of acetylation on modulus of rupture (*MOR*) (*white bars*) and modulus of elasticity (*MOE*) (*black bars*) of bamboo particle/plastic composites (BPPC) [bamboo particle (BP)/high-density polyethylene (HDPE) = 60/40]. Error bar is SD (*n* = 5). Bars with different capital and lowercase letters indicate significant differences in MOE and MOR values, respectively (*P* < 0.05). *WPG*, weight percent gain

Table 1. Effect of mixture ratio of bamboo particle (BP) and high-density polyethylene (HDPE) on mechanical properties of bamboo particle/plastic composites (BPPC)

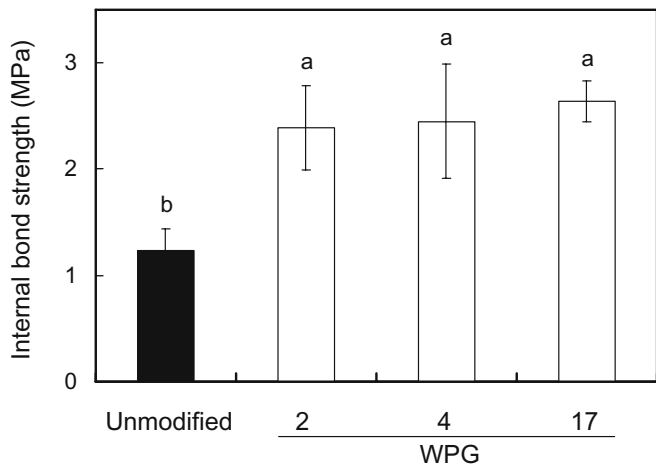| BP/HDPE (wt%)	Density (g/cm^3)	MOR (MPa)	MOE (GPa)	Internal bond strength (MPa)	Wood screw-holding strength (N)
0/100	0.74 ± 0.06[b]	25.6 ± 0.7[a]	0.9 ± 0.2[cd]	>5*	913.4 ± 60.4[a]
20/80	0.76 ± 0.01[b]	23.7 ± 0.8[ab]	1.2 ± 0.0[bc]	>5*	813.2 ± 44.1[ab]
30/70	0.82 ± 0.01[ab]	23.1 ± 0.4[ab]	1.5 ± 0.0[ab]	>5*	823.8 ± 43.8[ab]
40/60	0.85 ± 0.00[a]	22.1 ± 0.8[bc]	1.5 ± 0.1[ab]	2.7 ± 0.1[a]	850.8 ± 34.3[a]
50/50	0.86 ± 0.01[a]	19.9 ± 1.1[cd]	1.5 ± 0.1[a]	2.7 ± 0.1[a]	838.2 ± 20.6[a]
60/40	0.89 ± 0.02[a]	17.6 ± 0.3[d]	1.4 ± 0.1[ab]	2.0 ± 0.3[b]	833.1 ± 59.2[a]
70/30	0.85 ± 0.03[a]	13.5 ± 1.5[e]	1.0 ± 0.2[cd]	0.7 ± 0.1[c]	642.4 ± 128.9[b]
80/20	0.81 ± 0.05[ab]	9.9 ± 1.9[f]	0.8 ± 0.2[d]	0.3 ± 0.1[c]	442.0 ± 120.2[c]

*Over maximum limitation (5 MPa)
Values are mean ± SD (*n* = 5)
Means in each column with different superscript letter are significantly different (*P* < 0.05)
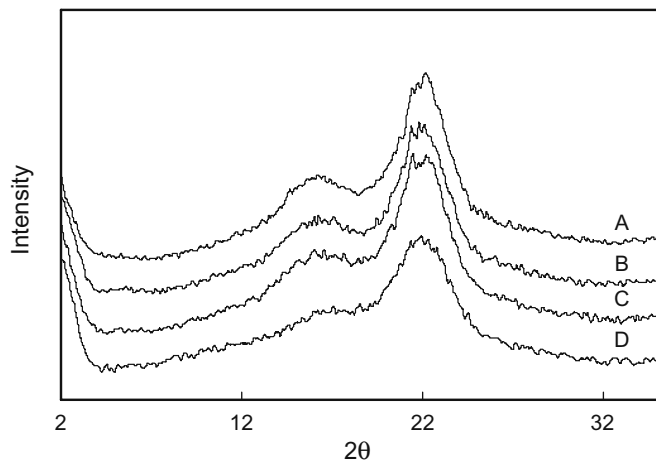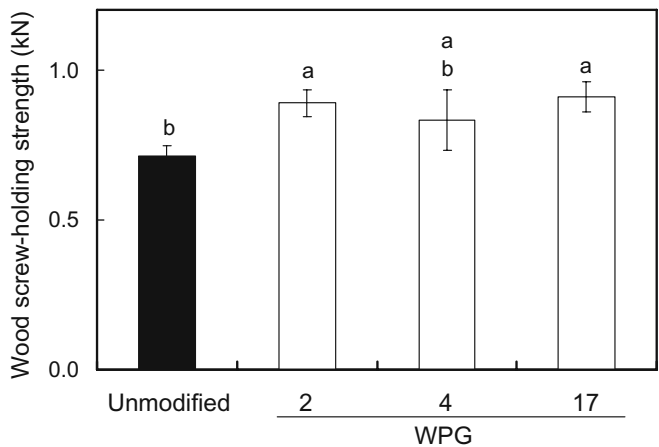
Fig. 2. X-ray diffraction diagrams of different extent of acetylated bamboo particles: unmodified (*A*); WPG 2 (*B*); WPG 4 (*C*); WPG 17 (*D*)

Fig. 4. Effect of acetylation on wood screw-holding strength of BPPC (BP/HDPE = 60/40). Error bar is SD (*n* = 5). Bars with different letters indicate significant differences among groups (*P* < 0.05)
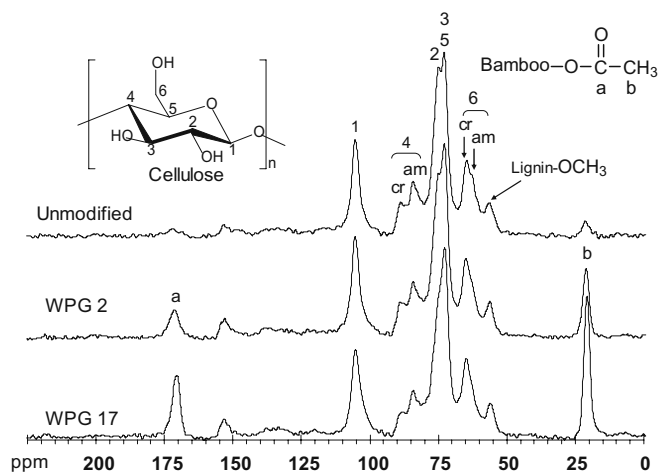

Fig. 3. Effect of acetylation on internal bond strength of BPPC (BP/HDPE = 60/40). Error bar is SD (*n* = 5). Bars with different letters indicate significant differences among groups (*P* < 0.05)

Fig. 5. [13]C cross-polarization magic angle spin (CP/MAS) nuclear magnetic resonance (NMR) spectra of bamboo particles acetylated to different extents

the latter peak corresponds to the reflection of planes 110 and 1ĪO of the native cellulose lattice. After acetylation, however, both peaks vanish gradually (Fig. 2B–D). These results are similar to the diagram obtained by Thiebaud et al.[24] In addition, the extent of decrystallization is a function of ester content (or WPG). In other words, the extent of acetylation greatly influenced crystallinity index, so with a higher extent of acetylation there was a lower crystallinity index. When the WPG of acetylated bamboo particles achieved 2, 4, and 17, their CrI were calculated as 56.6%, 54.2%, and 48.3%, respectively. This reduction trend is completely congruent with the MOE changes seen in Fig. 1. Therefore, it is evident that the MOE of BPPC is greatly influenced by the crystallinity of lignocelluloses.

Furthermore, the internal bond is the best single measure of the quality of lignocellulosic composites because it indicates the strength of the bonds between particles. Figure 3 shows that acetylation can improve the internal bond strength of BPPC. However, it can be seen that, regardless

of the WPG of acetylated bamboo particles, the strength of BPPC increased from the original 1.2 MPa to ~2.5 MPa after acetylation. Similar results were also observed in the wood screw-holding strength of BPPC (Fig. 4). These results indicate that even though the weight gain of acetylated bamboo particles was only 2%, acetylation also provides sufficient interfacial adhesion with the plastic matrix. Compared with unmodified composites, the internal bond and wood screw-holding strengths of acetylated composites were improved by 94% and 25%, respectively.

Reactive characteristics of acetylation of bamboo particles

In this study, solid-state CP/MAS ^{13}C-NMR was used to elucidate the characteristics of BP before and after acetylation. Figure 5 shows a characteristic carbohydrate pattern, namely C1 (105.3 ppm), C4 (88.7 and 84.3 ppm), C2 (74.8 ppm), C3, C5 (72.8 ppm), and C6 (64.6 and 62.8 ppm) for unmodified BP.[25] Hemicellulose carbons also resonated

Fig. 6. Scanning electron microscopy (SEM) micrographs of different extents of acetylated BPPC (BP/HDPE = 60/40): unmodified (**A**); WPG 2 (**B**); WPG 4 (**C**); WPG 17 (**D**). *Arrows*, BP

Fig. 7. Effects of different esterifications on mechanical properties of BPPC (BP/HDPE = 60/40). Error bar is SD ($n = 5$). Bars with different letters indicate significant differences among groups ($P < 0.05$)
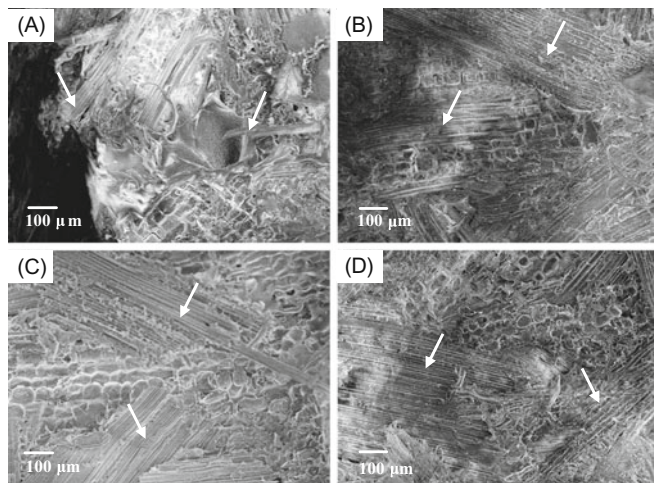
in the same field as cellulose ones, so they could not be precisely identified. The chemical shift at 56.5 ppm corresponded to lignin methoxy groups and the broad signal between 125 and 160 ppm to aromatic rings of lignin. However, after acetylation, the intensities of peaks at 74.8 (C2) and 62.8 (amorphous C6) ppm decreased and shifted downfield, and the decrease in signal intensity was more pronounced in C2, while no significant change was observed at 72.8 ppm (C3). In addition, peaks at 170.1 ppm (CH$_3$COO–) and 20.7 ppm (CH$_3$COO–), assigned to the acetyl group, were also found in Fig. 5 (WPG 2 and WPG 17). These results indicate that the order of reactivity of cellulose hydroxyl groups was C2–OH > C6–OH > C3–OH when the bamboo particles were modified by acetic anhydride in DMF. This result is similar to those reported by Dicke[26] and Jebrane and Sèbe.[27]

Morphology of acetylated bamboo particles and HDPE composites

SEM micrographs of the fractured surface for the composite samples (Fig. 6), correspond to the surface of fractured BPPC with 60 wt% of unmodified BP (Fig. 6A), acetylated BP of 2 WPG (Fig. 6B), acetylated BP of 4 WPG (Fig. 6C), and acetylated BP of 17 WPG (Fig. 6D). Although only selected parts of the BPPC are shown in the figures, they are assumed to be as representative of the sample as possible. Accordingly, the unmodified bamboo particles (Fig. 6A) are pulled out practically completely from the polymeric matrix; i.e., the fracture of the BPPC did not lead to the bamboo particles breaking. This result clearly indicates that the interfacial adhesion between the bamboo particle and the plastic was very weak. However, as shown in Fig. 6B–D, the SEM micrographs reveal that acetylated bamboo particles are well trapped by the polymeric matrix, regardless of the extent of acetylation. Thus, a better stress transfer from the weaker plastic phase to the bamboo particle through the interface could be expected; this is believed to
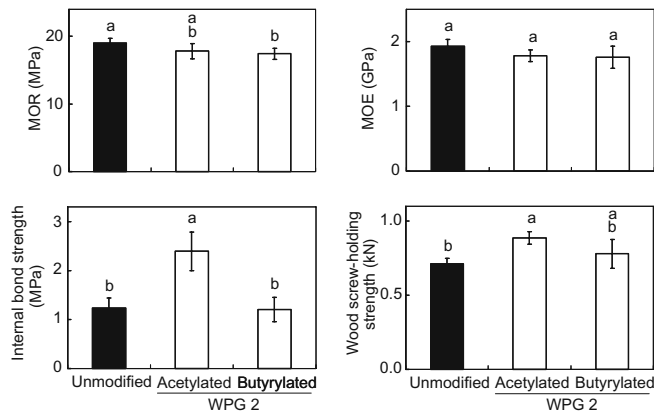
be caused by improved interfacial interaction, resulting in high internal bond strength (see Fig. 3).

Effects of different esterifications on mechanical properties of BPPC

To understand the influence of acyl chain length of esterified bamboo particles on mechanical properties, MOR, MOE, internal bond strength, and wood screw-holding strength of BPPC with WPG = 2 of acetylated and butyrylated bamboo particles were further investigated in this study. Except for the internal bond strength, there were no significant differences between acetylation and butyrylation (Fig. 7). Compared with the acetylated composite, there was a twofold decrease in the internal bond strength of the butyrylated composite. In other words, the effect of butyrylation on increasing the strength of composites was not significant. This phenomenon might be attributed to the relatively hydrophobic butyryl group being not sufficient to substitute for the hydroxyl group of the bamboo particle by butyrylation at 2 WPG, which does not efficiently improve the interfacial adhesion between the polymeric matrix and lignocellulosic material. In addition, FTIR spectra (Fig. 8) also clearly confirmed that the intensity of the C = O stretching band of the butyrylated bamboo particle at 1735 cm^{-1} was obviously lower than that of an acetylated one. The effect of different esterifications with higher WPG samples on properties of BPPC should be the subject of future systemic and detailed investigation.

Conclusions

Bamboo particle/plastic composites with high bamboo particle loading (60 wt% of bamboo particles) were successfully prepared by the manufacturing method of forming and flat presses similar to particleboard production. Additionally, it was shown that the acetylation of bamboo particles is a useful method to improve the interfacial interaction of BPPC. When the degree of acetylation had achieved only
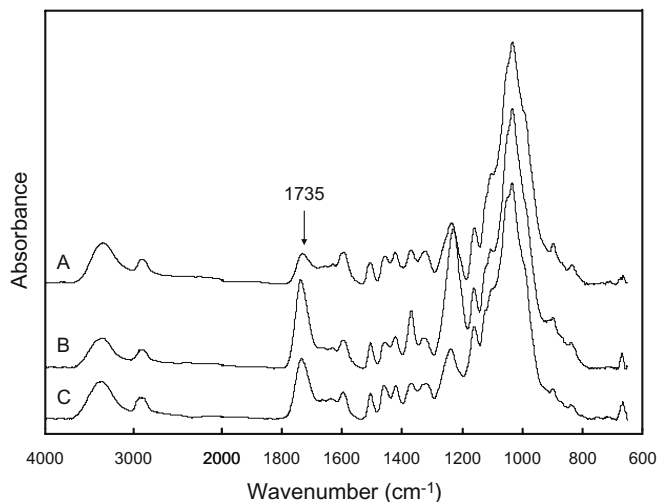
Fig. 8. Fourier transform-infrared (FTIR) spectra of different esterified bamboo particles (WPG 2): unmodified (*A*); acetylated (*B*); butyrylated (*C*)

2 WPG, the composites exhibited an excellent interfacial adhesion between the acetylated bamboo particle and polymeric matrix. Compared with the unmodified composites, the acetylated composites improved the internal bond strength by 94% and the screw-holding strength by 25%. To our knowledge, this is the first report to address the properties of acetylated BPPC made by the flat-platen pressing process. Furthermore, as it is known that the hydroxyl groups of bamboo are most often the reactive sites, the evidence of occurrence of the chemical modification was checked by solid-state ^{13}C-NMR. The results revealed that the order of reactivity of bamboo cellulose was C2–OH > C6–OH > C3–OH when it was modified by acetic anhydride in DMF. Moreover, according to XRD analysis, the crystallinity of cellulose in bamboo particles decreased with increasing WPG. In addition, it is well known that acetylation not only improves the dimensional stability of lignocellulosic materials but also increases their decay resistance. Thus, future studies should focus on the durability and mildew resistance of the acetylated BPPC during natural weathering, and the physicochemical properties of weathered specimens also should be evaluated.

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