Quantitative Analysis of Biomass in Three Types of Wood-Plastic Composites by FTIR Spectroscopy

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Biomass content greatly affects the properties of wood-plastic composites (WPCs). Determination of the biomass in WPCs is important for the development of WPCs. In this study, transmission Fourier transform infrared (FTIR) spectroscopy was used for biomass quantification in the following WPCs: Moso bamboo/polypropylene (PP) composites, Chinese fir/PP composites, and poplar/PP composites. The bands in the region of 1060 to 1030 cm^{-1} were considered characteristic of biomass. The peak at 1377 cm^{-1} was typical of PP. The peak intensities ratios (PIRs) of biomass to PP were determined, and the biomass contents were plotted against the PIRs. The achieved coefficients of determination (R^2) of the calibration fits exceeded 0.96. The results of validation showed that the range of the relative prediction deviations for biomass within WPC species was lower than ± 5.0%. Additionally, all three WPC species were combined into one data set, and a mixed model was constructed that had a slight decrease in the quality of the correlation (R^2 = 0.93). The range of the relative prediction deviations for biomass between WPC species did not exceed ± 9.0%.

Keywords: Fourier transform infrared (FTIR) spectroscopy; Wood-plastic composites (WPCs); Quantitative analysis; Biomass; Polypropylene (PP)

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INTRODUCTION

Wood-plastic composites (WPCs) are mainly composed of lignocellulosic biomass in a thermoplastic matrix. Lignocellulosic biomass is a natural biopolymer composite comprising cellulose, lignin, and hemicellulose. In China, Moso bamboo, Chinese fir, and poplar are the most common biomass species used in manufacturing WPCs. Isotactic polypropylene (PP) is a synthetic polymer with many excellent properties that have made it one of the most widely used thermoplastics. As a combination of biopolymers and synthetic polymers, WPCs bring together the advantages of biomass and plastic (Pilarski 2005). In addition, as a kind of green polymer material, WPCs not only increase the utilization rate of biomass, but also resolve the environmental problems caused by solid wastes of polymer products (Stark and Matuana 2007). Therefore, the market for WPCs has increased enormously over the past few decades. They are widely used in outdoor decking, railings, siding, and door frames (Chen *et al*. 2006; Lei and Wu 2012).

It is well known that the biomass to plastic ratio in WPCs influences the properties of the composites. For example, as biomass content increases, the dimensional stability of WPCs will decrease (Cheng and Wang 2009; Cheng and Shaler 2010; Tamrakar and Lopez-Anido 2011). In general, tensile, impact, and flexural strength of WPCs decrease, but tensile, flexural modulus, and Brinell hardness increase with increasing biomass

content within a certain range (Jin 2010; Atuanya *et al.* 2013; He *et al.* 2013). According to the American Society for Testing and Materials (ASTM), the distinction between WPCs and "plastic lumber" is that the weight percentage of plastic in WPCs must be less than 50%. In other words, biomass content in WPCs plays an important role in determining whether products are considered standard. Therefore, accurate quantification methods for biomass in WPCs are needed for further development of WPCs. However, only a few studies have addressed the compositional analysis of WPCs. Some researchers have used thermogravimetric analysis (TGA) to determine biomass content in WPCs. Because the degradation process of plastic and biomass cannot be separated completely, there are large deviations from predicted values. Moreover, it is necessary to know the formulation composition in advance (Ahmad Fuad *et al.* 1994; Renneckar *et al.* 2004; Jeske *et al.* 2012). Analytical pyrolysis (Py) has also been used for biomass quantification in WPCs (Windt *et al.* 2011). This process is not only expensive, but also time-consuming.

Fourier transform infrared (FTIR) spectroscopy is a rapid, simple, and inexpensive technique that is widely used for quantitative analysis in many fields (Rohman and Che Man 2009; Wang *et al.* 2010; Kaufhold *et al.* 2012). For example, FTIR has been used for the determination of clay minerals, quartz, and carbonates, as well as organic matter in shale (Chen *et al.* 2014). FTIR has also been used to quantify lignin contents in wood and wood decayed by brown-rot fungus (Rodrigues *et al.* 1998; Pandey and Pitman 2004). In a recent study, a distribution profile of plastic and wood within WPCs by FTIR was attempted (Lee *et al.* 2010). However, there have been no studies concerning the determination of biomass content in WPCs by FTIR. It should be pointed out that WPCs used in previous studies have been a single type of WPC. In addition, most of the WPCs were simple mixtures of wood flour and thermoplastic, with no additives included. From this point of view, a general quantitative method for different types of WPCs that include a small amount of additives has stronger applicability, but it is difficult to achieve because of the discrepancies between different biomass species.

In the present study, FTIR spectroscopy was used to study three types of WPCs. The objectives were to develop a simple and inexpensive technique for estimating the content of biomass in single types of WPCs and to demonstrate the feasibility of constructing a general model for different types of PP-based WPCs.

EXPERIMENTAL

Materials

Isotactic polypropylene (density: 0.9 g/cm^{-3}, melt flow index: 3.5 g/10 min) was purchased from Jia Li Xin Plastics Materials Co. Ltd, China. Chinese fir (*Cunninghamia lanceolata* (Lamb.) Hook.) and Moso bamboo (*Phyllostachys pubescens* Mazel) were obtained from Zhejiang province. Poplar (*Populus cathayana* Rehd.) was obtained from Jilin province. Biomass samples were ground in a grinder and screened in a vibratory sieving machine. Then, biomass particles of 250 μm (60 mesh) size were obtained. Calcium carbonate, aluminic ester, and Tissuemat E (low molecular mass polyethylene) were used as toughener, coupling agent, and lubricant, respectively. Pentaerythritol tetrakis (1010) and dilauryl thiodipropionate (DLTP) were used as primary antioxidant and auxiliary antioxidant, respectively. These additives were purchased from Kang Gao Te Plastic Technology Co. Ltd, China.

Methods

Preparation of WPCs

Biomass particles were dried at 105 °C for 24 h before processing. All WPCs were produced by three processing steps: (1) Biomass modification: biomass particles were stirred in a high speed mixer (Type GH-10DY; Bei Jing Huasco Plastics Machinery Co. Ltd, China), and the coupling agent was added to the mixer when the temperature reached 110 °C. A fixed weight ratio of coupling agent to biomass of 1:100 was employed. Ten minutes later, the modified biomass particles were discharged into a sealed container; (2) Mixing: PP, additives, and the modified biomass particles were mixed in the high speed mixer for 15 min. The ratios of antioxidant, lubricant, and toughener were fixed at 0.4%, 0.2%, and 5%, respectively; and (3) Extruding: the mixture was extruded by the twin-screw extruder (Labo Plastomill Toyo Seiki, Japan). The processing temperature for extrusion was set at 168 °C for the feeding section, 172 to 182 °C for the compression section, and 172 °C for the metering section, respectively. The rotary speed of the twin-screw was 1.5 to 3 rpm. The WPC samples were stored in a desiccator.

Twenty-seven reference WPC samples were used for calibration. In addition, 12 independent testing samples were used for validation. The formulations are summarized in Table 1.

Table 1. Summary of Sample Formulations

WPCs	Components	Reference samples		Testing samples	
		No. of samples	Content range (wt%)	No. of samples	Content range (wt%)
Moso bamboo/PP composites	Bamboo	9	29.7−57.6	4	36.8−49.6
	PP		36.1−64.4		44.2−57.2
	Additives		5.9−6.3		6.0−6.2
Poplar/PP composites	Poplar	9	29.7−57.7	4	43.6−53.7
	PP		36.0−64.3		40.1−50.3
	Additives		6.0−6.3		6.1−6.2
Chinese fir/PP composites	Chinese fir	9	29.5−57.4	4	33.7−49.5
	PP		36.4−64.5		44.4−60.3
	Additives		6.0—6.2		6.0—6.1

FTIR spectral measurements

The FTIR analysis of WPCs was performed on a Spectrum One FTIR spectrometer (Perkin Elmer, USA) equipped with a deuterated triglycine sulfate (DTGS) detector. The WPC samples were ground to a fine powder by A11 disintegrator (IKA Group, Germany). The grain size of the samples must be less than 74 μm (200 mesh). Then, 1- to 2-mg sample powders were mixed with potassium bromide to a 1 to 1.5% concentration. The mixture was then pressed into a transparent pellet for measure. The spectra were collected in the range from 4000 to 400 cm^{-1} at a spectral resolution of 4 cm^{-1}, and 16 scans were taken per sample. Each sample was tested five times.

The peak heights of the IR spectra were measured with Spectrum v 5.0.1 software. The heights of the peaks were measured from the baseline, which was constructed by drawing a straight line from 1870 to 780 cm^{-1}. A vertical line from the top of the peak to this baseline represents the peak height.

Data analysis

The linear regression analysis was performed using Microsoft® Excel 2010. The ratio of the biomass peaks relative to the PP peaks was calculated. These peak intensity ratios (PIRs) of biomass/PP were then plotted against the biomass contents and univariate regression models for biomass determination were established. The accuracy of the regression models were validated by testing samples. Relative prediction errors were calculated to assess the predictability of the model.

RESULTS AND DISCUSSION

FTIR Analysis of Biomass, Polypropylene, and WPCs

The FTIR spectra of bamboo, Chinese fir, poplar, PP, and calcium carbonate are shown in Fig. 1. Lignocellulosic biomass is mostly composed of lignin, hemicellulose, and cellulose, but there are small differences between the proportion and structure of the chemical compositions in different biomass species. Therefore, the FTIR spectra of Moso bamboo (bamboo species), Chinese fir (softwood species), and poplar (hardwood species) showed a similar basic structure with small differences.

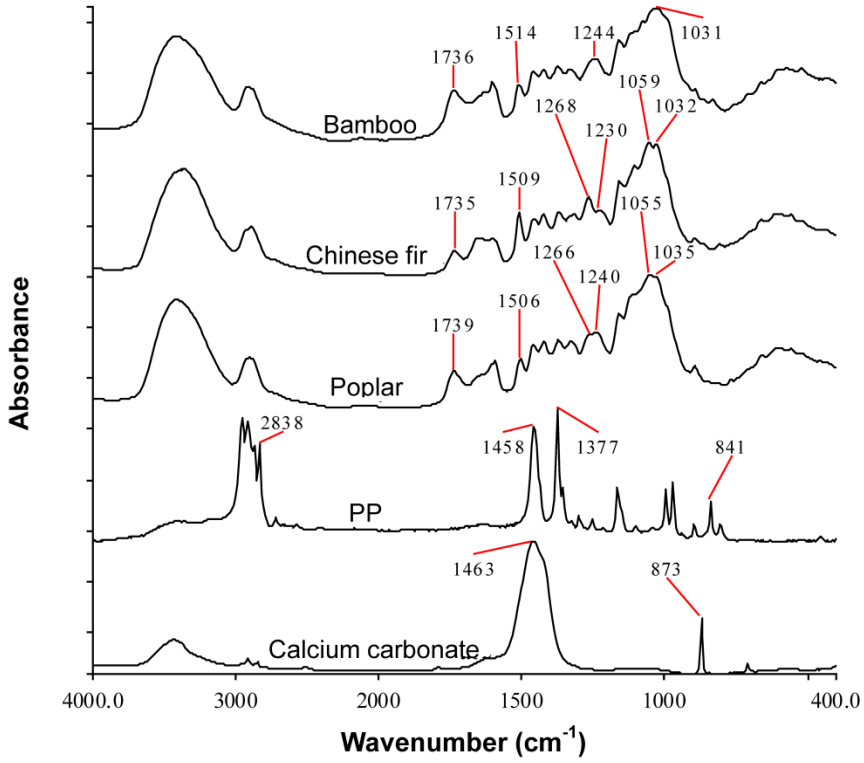


Fig. 1. FTIR spectra of bamboo, Chinese fir, poplar, PP, and calcium carbonate

Detailed descriptions of the peak assignments have been provided in another article (Pandey and Pitman 2003). The major absorption peaks for the current study are chosen as follows: the non-conjugated C═O in hemicellulose stretching vibration absorption peak is seen at around 1740 cm^{-1}, the absorption peak at around 1510 cm^{-1} is assigned to aromatic skeletal vibration, and the absorption peaks in the region from 1060 to 1030 cm^{-1} are

assigned to C—O stretching vibration in holocellulose. However, the relative intensities of bands show small differences. The relative intensities of peaks at around 1740 cm^{-1} are greater in Moso bamboo and poplar, whereas the peak at around 1510 cm^{-1} is stronger in Chinese fir. This is because of a higher holocellulose to lignin ratio in poplar and Moso bamboo as compared to the Chinese fir (Colom *et al.* 2003; Pandey 1999). The strong peak of Moso bamboo at 1244 cm^{-1} indicates the high proportion of syringyl units in bamboo lignin. This is different from that of Chinese fir, which has a high amount of guaiacyl units, as demonstrated by strong absorption at 1268 cm^{-1} (Wang and Ren 2008).

The spectrum of PP is significantly different from that of biomass. The strong absorption peaks of PP are observed at 1458 cm^{-1} due to CH_2 bending vibrations and at 1377 cm^{-1} attributed to CH_3 bending vibration (Kitching and Donald 1998). The absorption peaks at 2838 cm^{-1} and 841 cm^{-1} are contributed by a CH_2 asymmetric stretching vibration and CH_2 rocking vibrations, respectively (Morent *et al.* 2008). These absorption peaks are specific to PP.

Figure 2 shows the spectra of three types of WPCs. The spectra of three WPC species with the same biomass content are very similar and the small differences appear in the fingerprint region. These differences are consistent with the discrepancies between the spectra of the three biomass species.

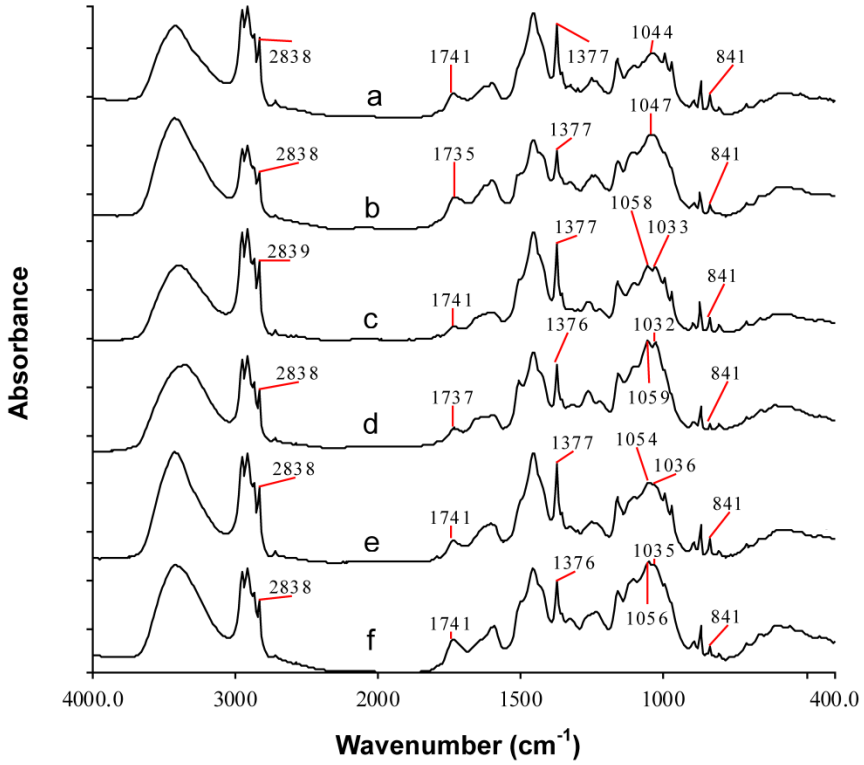


Fig. 2. FTIR spectra of three types of WPCs with different biomass contents: (a) bamboo/PP composites (29.7%), (b) bamboo/PP composites (57.7%), (c) Chinese fir/PP composites (29.5%), (d) Chinese fir/PP composites (57.4%), (e) poplar/PP composites (29.7%), and (f) poplar/PP composites (57.7%)

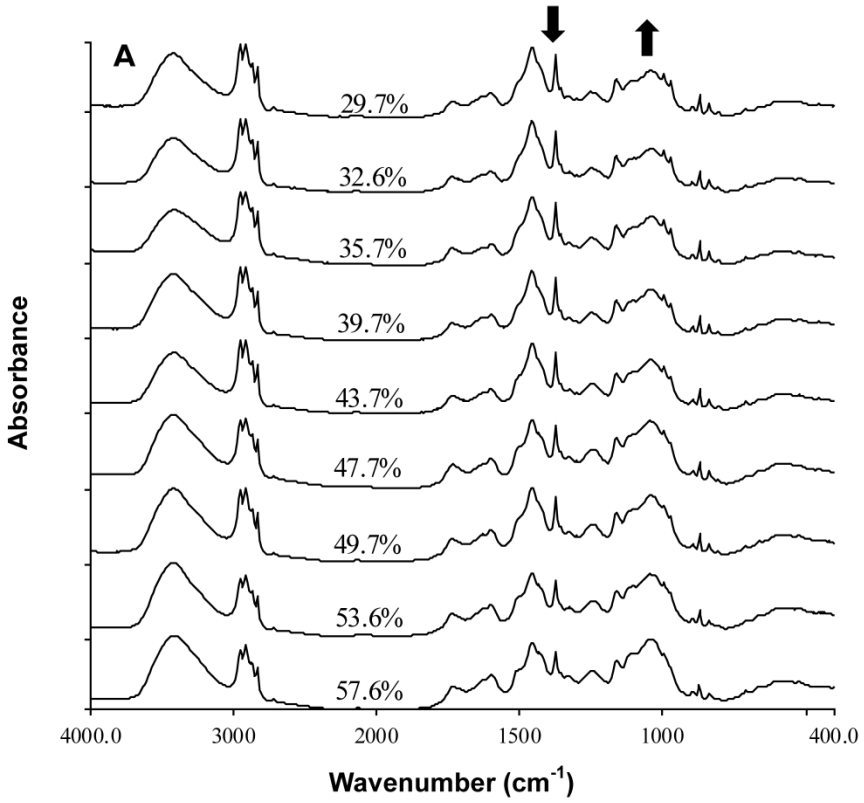
As expected, the characteristic bands of both biomass and PP are observed in the spectra of WPCs. However, the absorption peaks at around 1510 cm^{-1} and 1458 cm^{-1} were overlapped by the strong absorption peak of nanometer calcium carbonate at 1463 cm^{-1}.

Fortunately, most of the specific absorption peaks of biomass can be easily distinguished from those of PP. For example, the peak shapes of WPCs at around 1740 cm^{-1} and in the region of 1060 to 1030 cm^{-1} are almost identical to those of biomass, which suggests that these peaks are mainly contributed by biomass. The absorption peaks of WPCs at 2838 cm^{-1}, 1377 cm^{-1}, and 841 cm^{-1} are very similar to those of PP, which indicates that these peaks belong to PP. It is important to mention that the peak intensities of PP at 1377 cm^{-1} and biomass in the region of 1060 to 1030 cm^{-1} are moderate and they are not easily influenced by other absorption peaks. Thus, these peaks are more suitable for biomass prediction.

To verify the characteristic absorption peaks of biomass and PP, additives in WPCs were further analyzed by FTIR. The results showed that Tissuemat E, DLTP, and 1010 had weak absorption peaks in the region of 1062 to 1047 cm^{-1}, but the amount of additives in WPCs was very small. Consequently, the strongest absorption bands in the region of 1050 to 1040 cm^{-1} were considered to be characteristic of bamboo, while the strongest peaks in the region of both 1060 to 1050 cm^{-1} and 1040 to 1030 cm^{-1} were taken as features of Chinese fir and poplar, respectively. The absorption peaks of PP at 1377 cm^{-1} was chosen as the reference.

Developing the Univariate Regression Models for Biomass Measurement in WPCs

Figure 3 provides the IR absorbance spectra of WPCs with different biomass contents. For Moso bamboo/PP composites, the characteristic peak intensities of bamboo in the region of 1060 to1030 cm^{-1} increased gradually, while the intensity of the PP peak at 1377 cm^{-1} decreased significantly when the ratio of bamboo to PP increased. Similarly, the same trend could be observed in both poplar/PP composites and Chinese fir/PP composites.



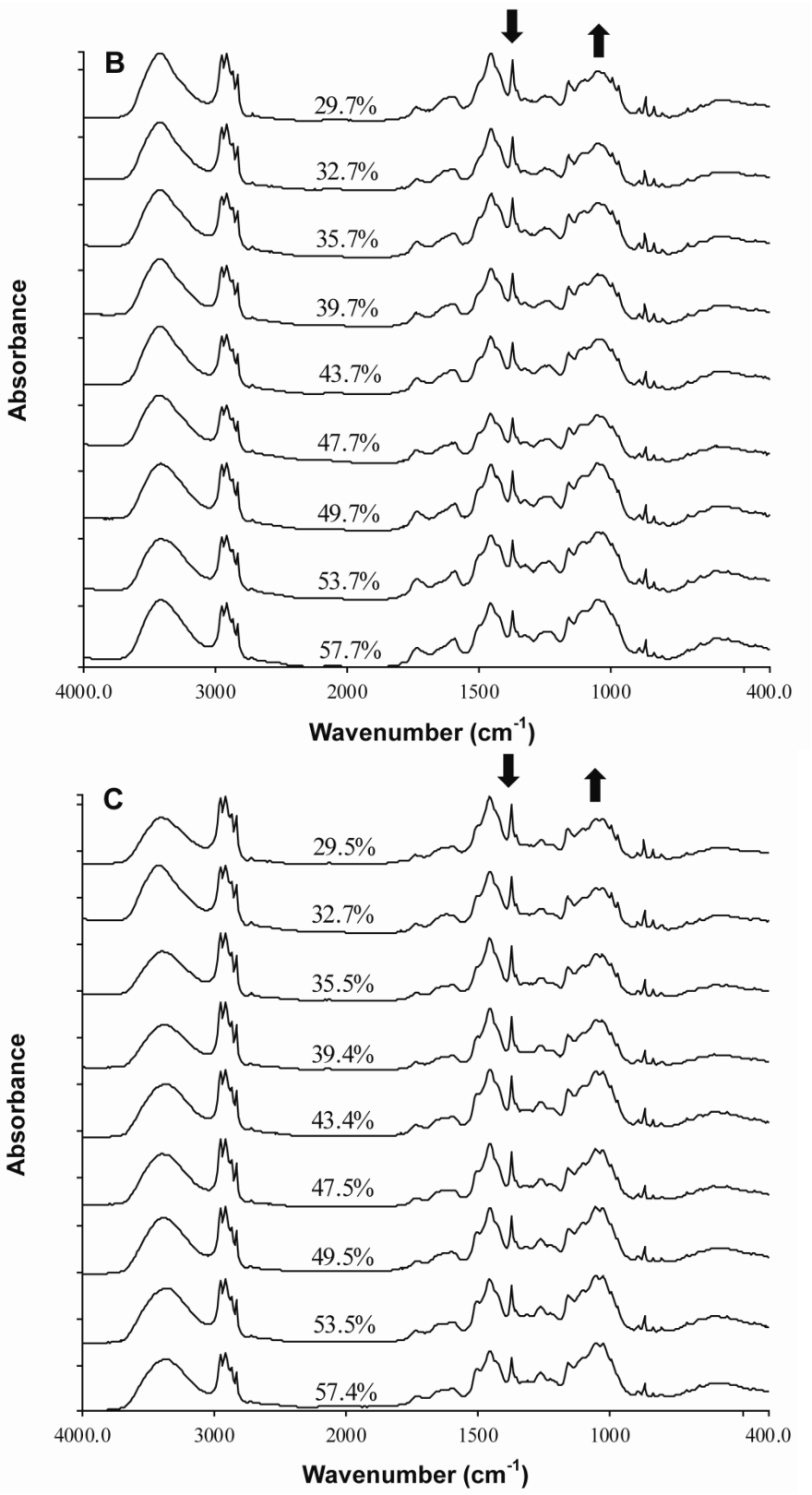


Fig. 3. FTIR spectra of three different types of WPCs with different biomass contents: (A) bamboo/PP composites, (B) poplar/PP composites, (C) Chinese fir/PP composites

From Fig. 3, it is clear that the content of biomass in WPCs was directly associated with the relative intensities of characteristic bands. The PIRs of biomass/PP were regressed against the biomass contents in WPCs and univariate regression equations for biomass prediction were obtained. The correlations between the biomass content and the PIRs are shown in Fig. 4.

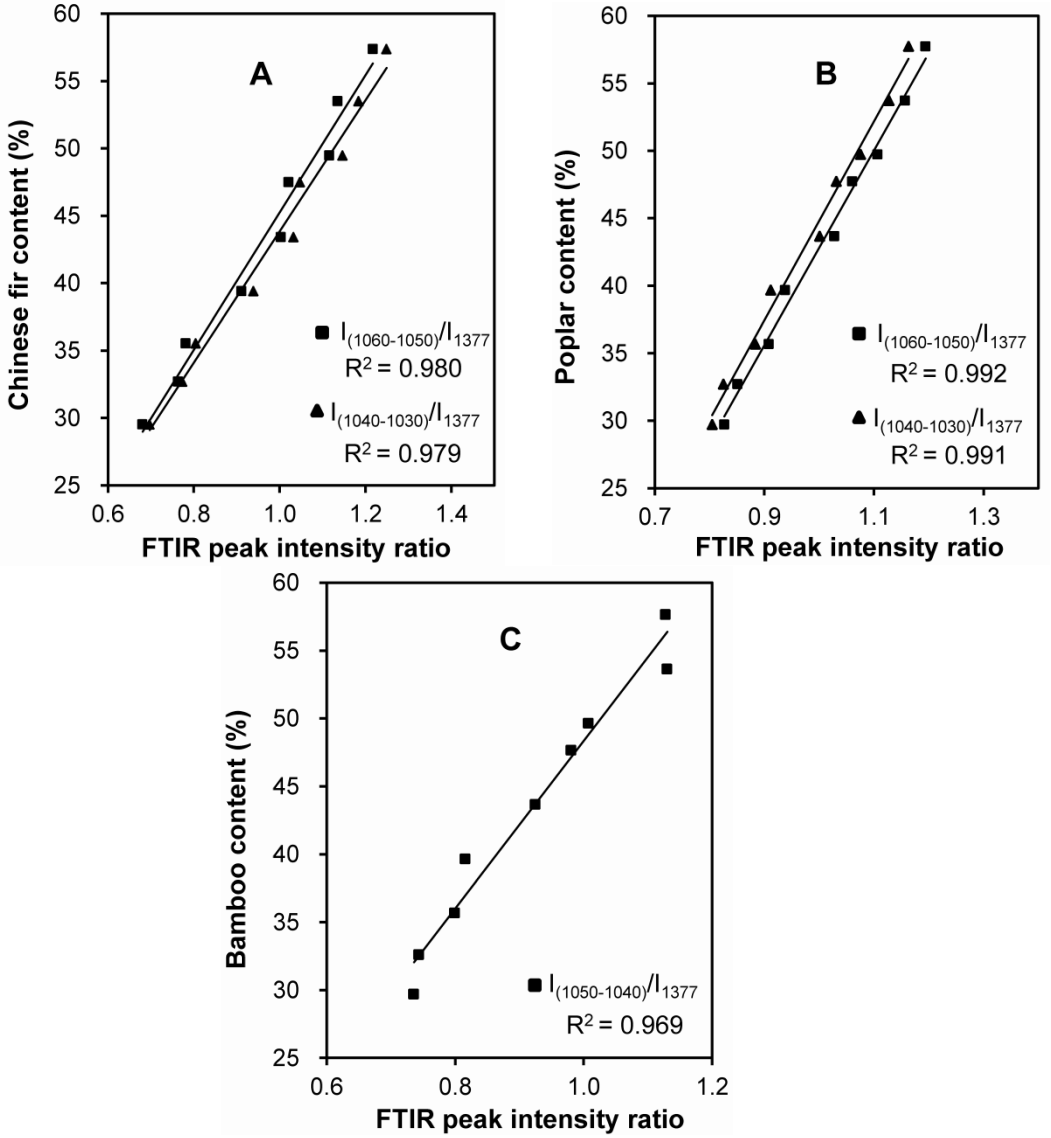


Fig. 4. Correlation between the biomass content and the FTIR PIRs for single types of WPCs: (A) Chinese fir, (B) poplar, and (C) bamboo. The R^2 corresponding to each curve is also shown.

Good correlations were obtained, with coefficients of determination (R^2) over 0.96. For Poplar/PP composites and Chinese fir/PP composites, the best correlations between the biomass content and the PIRs were obtained by taking the strongest peak in the region of 1060 to 1050 cm^{-1} as references for biomass. In addition, there were strong correlations between the biomass content and the PIRs when the strongest peak in the region of 1040 to 1030 cm^{-1} was used as the reference for biomass. For Moso bamboo/PP composites, a significant correlation between the biomass content and the PIRs was found when the strongest peak in the region of 1050 to 1040 cm^{-1} was used as the reference for bamboo.

The broad peaks of biomass in the region of 1060 to 1030 cm^{-1} contain the information about the lignin, although they are mainly contributed by the holocellulose. In addition, the intensities of the peaks in this region are greater. Therefore, the peaks in this region can accurately reflect the biomass content in the composites. Interestingly, the positions of the most representative peaks for three biomass species exhibit small difference. This may be linked to the differences between the proportion and structure of the chemical compositions in three biomass species, as mentioned previously. The results showed that the predictive equations (Table 2) could predict biomass content in single types of WPCs.

Table 2. Predictive Equations for Biomass Determination

Models	PIRs	Components	Predictive equations	R^2
Intra-group models	$I_{(1050\text{-}1040)}/I_{1377}$	Bamboo	Y = 61.699 x − 13.345	0.969
	$I_{(1060\text{-}1050)}/I_{1377}$	Chinese fir	Y = 48.682 x − 4.8364	0.980
	$I_{(1040\text{-}1030)}/I_{1377}$		Y = 50.935 x − 5.6855	0.979
	$I_{(1060\text{-}1050)}/I_{1377}$	Poplar	Y = 73.158 x − 30.455	0.992
	$I_{(1040\text{-}1030)}/I_{1377}$		Y = 73.644 x − 28.839	0.991
Inter-group models	I_a/I_{1377}	Biomass	Y = 57.735 x − 11.717	0.930
	I_b/I_{1377}		Y = 54.451 x − 9.5715	0.899
[a] the strong peak in the region of 1050−1040 cm^{-1} for bamboo, 1040−1030 cm^{-1} for two wood species				
[b] the strong peak in the region of 1050−1040 cm^{-1} for bamboo, 1060−1050 cm^{-1} for two wood species				

The strongest peak in the regions of both 1060 to 1050 cm^{-1} and 1040 to 1030 cm^{-1} were treated as the references for two wood species, while the strongest peak in the region of 1050 to 1040 cm^{-1} was the reference for bamboo and the peak at 1377 cm^{-1} was taken as the reference for PP. Mixed models based on three WPC species were constructed. The plots of the biomass/PP PIRs *versus* the biomass content are shown in Fig. 5.

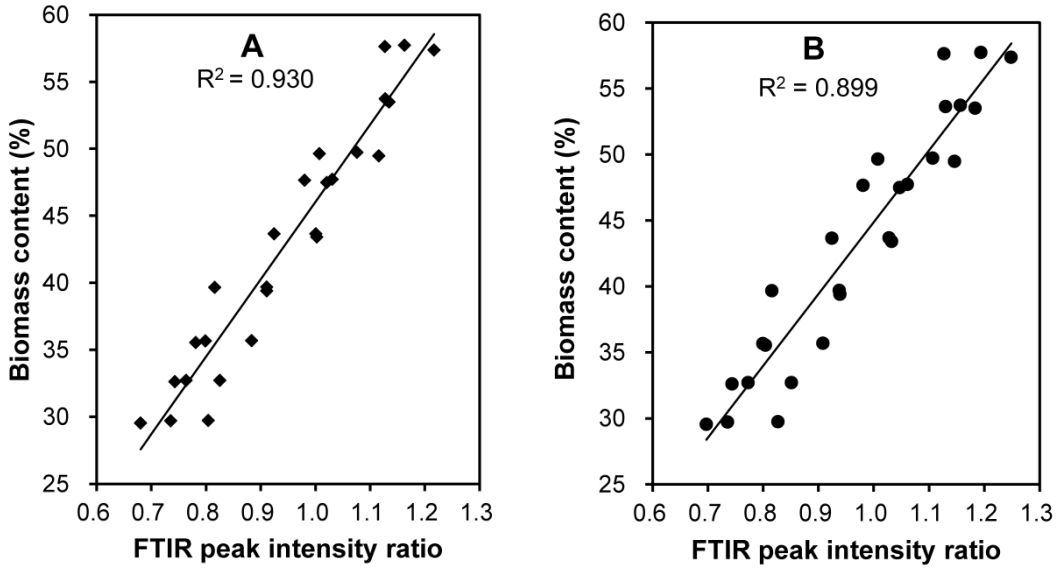


Fig. 5. Correlation between biomass content and FTIR PIRs for three types of WPCs: (A) the strongest peak in the region of 1040 to 1030 cm^{-1} for two wood species and (B) the strongest peak in the region of 1060 to 1050 cm^{-1} for two wood species

Obviously, in both cases there were high correlations between the biomass content and the PIRs. A higher coefficient of determination (R^2 = 0.93) between the biomass content and the PIRs was obtained with the two wood species reference peaks in the region of 1040 to 1030 cm^{-1}. However, the correlation decreased slightly compared to that of the univariate regression models based on single types of WPCs. This is not surprising given the differences between the three types of biomass. It should be noted that although there are differences between the three species, the results of linear regression show that these differences do not prevent data from all three WPC species from being used to produce mixed models for predicting biomass content. The predictive equations are presented in Table 2.

Generally, there were higher correlations between the biomass content and the PIRs for both intra-group models and inter-group models when the strongest peak in the region of 1040 to 1030 cm^{-1} was treated as the reference for two wood species. Therefore, the regression equations based on the relative intensity of the peak in the region of 1040 to 1030 cm^{-1} for two wood species and 1050 to 1040 cm^{-1} for bamboo can be used for biomass prediction.

Validation of the Linear Regression Models

Four testing samples with different ratios of bamboo and PP were used to validate the accuracy of the predictive equation for Moso bamboo/PP composites. The same approach was used for Chinese fir/PP composites and poplar/PP composites. The results are given in Table 3.

Table 3. Summary of the Accuracy of the Models for Single Types of WPCs

Bamboo (%)			Chinese fir (%)			Poplar (%)		
AV[a]	PV[b]	RD[c]	AV[a]	PV[b]	RD[c]	AV[a]	PV[b]	RD[c]
36.8	35.1	4.6	33.7	35.1	−4.2	43.7	44.4	−1.6
40.8	40.4	1.0	45.5	45.2	0.7	47.7	46.8	1.9
43.7	44.5	−1.8	47.5	47.9	−0.8	49.7	50.9	−2.4
49.6	48.6	2.0	49.5	50.9	−2.8	54.7	55.2	−0.9

[a] Actual values

[b] Predicted values

[c] Relative prediction deviations = (Theoretical values − Predicted values)/ Theoretical values × 100

As shown in Table 3, the predicted contents of biomass in WPCs were close to the actual values, with low deviations. Although relatively high deviations were found for a few samples, the range of the relative prediction deviations was lower than ± 5.0%. The possible reasons for the deviations may be (1) raw materials loss during the preparation of WPCs resulted in the differences between the theoretical component contents and the actual contents, (2) the WPC powders were not absolutely homogenous and a small quantity of sample was used in the FTIR analysis, (3) analytical noise and measurement errors in FTIR measurements, and (4) the effect of the absorption bands of the additives on the characteristic bands of biomass and PP. The testing samples were not used in developing linear regression models; thus the results could be viewed as a prediction of the biomass in unknown WPC samples. The prediction accuracy of the method described

above is better than that of TGA, as reported by Renneckar *et al.* (2004) and Jeske *et al.* (2012), and is comparable to the accuracy of results obtained by using TGA, as reported by Fuad *et al.* (1994), although WPCs used in the present study included additives. More importantly, FTIR method is more convenient and efficient compared to those thermo-analytical methods. The findings demonstrate that the univariate regression models for intra-group can perform successfully for biomass measurement in WPCs.

All 12 testing samples were used to validate the accuracy of the mixed model. The results are shown in Table 4. Expectedly, the accuracy of the inter-group model slightly decreased compared to the accuracy of the intra-group models. In the case of Moso bamboo/PP composites, the relative prediction deviations for bamboo from intra-group model ranged from −1.8% to 4.6%, while the relative prediction deviations from inter-group model were between 3.0% and 8.7%. This general trend was also seen in both Chinese fir/PP composites and poplar/PP composites. As mentioned before, this is mostly due to the differences between the proportion and structure of the chemical compositions of three biomass species. Despite all this, the maximum relative deviations were still lower than ± 9.0%. The results of validation showed that the mixed model, constructed with three types of WPCs, could effectively predict biomass content in different types of PP-based WPCs.

Table 4. Summary of the Accuracy of the Mixed Model

Bamboo (%)			Chinese fir (%)			Poplar (%)		
AV[a]	PV[b]	RD[c]	AV[a]	PV[b]	RD[c]	AV[a]	PV[b]	RD[c]
36.8	33.6	8.7	33.7	34.5	−2.4	43.7	45.7	−4.6
40.8	38.6	5.4	45.5	45.9	−0.9	47.7	47.6	0.2
43.7	42.4	3.0	47.5	49.0	−3.2	49.7	50.8	−0.7
49.6	46.2	6.9	49.5	52.4	−5.9	53.7	54.1	6.4
[a] Actual values								
[b] Predicted values								
[c] Relative prediction deviations = (Theoretical values − Predicted values)/ Theoretical values × 100								

CONCLUSIONS

1. The FTIR spectra of three types of PP-based WPCs were measured by direct transmittance method. The characteristic bands of biomass and PP were also determined. The biomass contents were plotted against the PIRs of biomass to PP. As a result, good correlations were obtained ($R^2 > 0.96$).
2. In all cases, the ranges of the relative prediction deviations for biomass determination were lower than ± 5.0%. The results showed that transmission mode FTIR could be used for biomass measurement within WPC species.
3. Even though there were subtle differences between the three WPC species, all of the samples could be combined into one mixed model with an R^2 value of 0.93. The results of validation showed that the range of the relative prediction deviations was not over ±

9.0%. The results of our preliminary analysis show that it is feasible to construct a general model for biomass determination between different types of PP-based WPCs.

4. More research is needed regarding the application of FTIR to other types of WPCs. Furthermore, it is possible to use a general model constructed by FTIR combined with multivariate statistical methods to accurately predict the content of biomass in a wide variety of WPC species.

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