

Study on Rapid Measurement of Wood Powder Concentration of Wood-Plastic Composites using FT-NIR and FT-IR Spectroscopy Techniques¹

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ABSTRACT

Wood-plastic composite (WPC) is a promising and sustainable material, and refers to a combination of wood and plastic along with some binding (adhesive) materials. In comparison to pure wood material, WPCs are in general have advantages of being cost effective, high durability, moisture resistance, and microbial resistance. The properties of WPCs come directly from the concentration of different components in composite; such as wood flour concentration directly affect mechanical and physical properties of WPCs. In this study, wood powder concentration in WPC was determined by Fourier transform near-infrared (FT-NIR) and Fourier transform infrared (FT-IR) spectroscopy. The reflectance spectra from WPC in both powdered and tableted form with five different concentrations of wood powder were collected and preprocessed to remove noise caused by several factors. To correlate the collected spectra with wood powder concentration, multivariate calibration method of partial least squares (PLS) was applied. During validation with an independent set of samples, good correlations with reference values were demonstrated for both FT-NIR and FT-IR data sets. In addition, high coefficient of determination (R^2_p) and lower standard error of prediction (SEP) was yielded for tableted WPC than powdered WPC. The combination of FT-NIR and FT-IR spectral region was also studied. The results presented here showed that the use of both zones improved the determination accuracy for powdered WPC; however, no improvement in prediction result was achieved for tableted WPCs. The results obtained suggest that these spectroscopic techniques are a useful tool for fast and nondestructive determination of wood concentration in WPCs and have potential to replace conventional methods.

Keywords: wood plastic composite, wood powder, fourier transform near-infrared and infrared spectroscopy, partial least-squares regression

1. INTRODUCTION

Wood plastic composites (WPCs) are normally produced by mixing plant fiber with polymer, or by adding wood fiber as filler in a

polymer matrix, and pressing or molding under high pressure and temperature (Ashori, 2008). The wood fibers are obtained from waste materials generated by lumber manufacturers, which are then processed to produce a consistent re-

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inforcing product. The use of these particles promotes the reclamation of wood waste that would otherwise be sent to landfills and ensures that no new trees are cut down to make the product. Wood-plastic composites also reduce energy use, since they melt at lower temperatures than typical plastics. In comparison to pure wood material products, WPCs are in general have advantages of being cost effective, high durability, moisture resistance, and microbial resistance.

In past ten years, WPCs have emerged as an important family of engineering materials. They have become prevalent in many building applications, such as decking, docks, landscaping timbers, fencing etc., partially due to the need to replace pressure-treated solid lumber (Pilarski and Matuana, 2005). The quality of products made from WPCs directly affected by its compositional constituents. The typical concentration of wood fibers in WPC has been between 50 - 70 wt%. However, higher concentration of wood fiber than plastic in WPCs is always recommended to get a quality product, ultimately rise the cost of final product. Because of economic gain, WPCs is susceptible to be sell with false high concentration of wood.

Over the past several years, a number of vibrational spectroscopic techniques have been developed that allow complex chemical information to be determined about the samples being scanned. Different spectroscopic techniques operate over different and limited frequency ranges depending on the process being

studied and the magnitude of the associated energy change. NIR signals are associated with molecular vibrations, specifically the overtones and combinations of fundamental vibrations. Chemical bonds between light atoms, such as C-H, O-H, and N-H, generally have high vibrational frequencies, which result in overtone and combination bands that are detectable in the NIR region of 780 - 2,500 nm (Osborne, Fearn, & Hindle, 1993). FT-IR spectroscopy focuses on the MIR region ($4,000 - 400 \text{ cm}^{-1}$) of the electromagnetic spectrum and monitors the fundamental vibrational and rotational stretching modes of molecules, which produce a chemical profile of the sample.

Vibrational spectroscopic techniques have been successfully used in wood industry for quality analysis of wood materials (Rowell, 2012, Li *et al.*, 2015). In principle, the vibrational spectroscopic techniques exploit the differences in biological composition that exists between different constituent of the sample. Lee *et al.*, (2010) used FT-IR spectroscopy in combination with principal component analysis for characterization and discrimination of five types of wood plastic composites and further suggested the purposed technique as a useful tool for determining the distribution profile of wood and plastic materials within different types of WPCs.

The aim of the present feasibility study was to evaluate the potential of FT-NIR and FT-IR spectroscopies, in combination with the application of multivariate calibration method of partial least squares regression analysis to determine

the wood powder concentration in WPC. Moreover, joint use of NIR and MIR region was evaluated for developing prediction model with the aim of to improve the prediction accuracy.

WPC is a composite material that combination of thermoplasticity (Polyethylene, Polypropylene, etc.) and woody reinforcement (wood flour, bamboo flour, etc.). WPC is able to improve plastic and wood's weakness such as machinability, durability and water-proofing, antimicrobial resistance. Also, this material is able to use as guard-rail, deck, outer-wall and fence which usually uses outside. However, there is no techniques to check WPC's quality in fields. Recently, there are that a lot of low quality products have been distributed. In the law, wood products should be over 51% wood content, but some of distribution dealers deceive. Because lower than 50% wood content is counted as plastic products, these products are out of law application range, and it is not able to apply legal dispositions. Therefore, it is need to develop rapid and accurate wood flour content measurement.

On this hence, radiocarbon (C-14) analysis of wood flour contents of WPC is studying by National Institute of Forest Science, and thermogravimetric analysis (TGA) to measure of wood flour contents of WPC is reported (Jeske *et al.*, 2012). However, using TGA is possible to quantitative analysis of known-elements, but it is hard to define unknown-elements of WPC. It is caused because wood flour and plastic matrix are shows a broad scope in 200 - 700 °C

(Helene *et al.*, 2012). In addition, nuclear magnetic resonance (NMR) method also has been studied (Scott *et al.*, 2004).

This study is trying to use non-destructive measuring method as like the existing method, and it is focused on improving accuracy and rapidly measurement by using FT-NIR method. Results of FT-NIR was analyzed Partial Least Squares Regression (PLS-R) to determine correlations between spectrum and wood flour contents. The wave length of FT-NIR was 1,000 - 2,500 nm, and two different status of samples are used. One is pellet status, and the other is ground in less than 1.4 mm. Also, PLS-R was done to determine correlation between status and spectrum.

2. MATERIALS and METHODS

2.1. Wood Plastic Composite Sample

In this study, WPC was provided by Ilsam Corporation (Korea) in two different forms: powder and tablet / pellet and with five different volume fraction of wood powder; 40, 45, 50, 55, and 58%. Other than wood powder, the WPC consist of coupling agent (2.4%), additives (10.8%), and the remaining concentration was thermoplastic material (polyethylene, polypropylene, etc.) WPC pellets were prepared by 92 mm twin conical extruder (Cincinnati, USA). These following concentrations of added wood powder covers the range usually present in commercial samples. 15 powdered and 20 tableted WPC samples for each concentration were

Table 1. Components of test specimens each WPCs

(Unit : w.t. %)					
No.	Wood Flour	PP	PE	Coupling Agent	Additives
W40	40	12	26.6		
W45	45	10.8	24		
W50	50	9.6	21.3	2.4	12.5
W55	55	8.4	18.6		
W58	58	7.2	16		

Table 2. Physical properties of PP and PE

	Type of thermoplasticity	Melt flow index (g/min)	Density (g/cm ³)
Polypropylene	Y120	2.0	0.96
polyethylene	B230A	0.33	0.96

measured using FT-NIR spectrometer; however, 20 samples for each concentration of both powdered and tableted WPC were scanned under FT-IR spectrometer.

2.2. Experiment methods

2.2.1. FT-NIR Spectra Collection

NIR spectra of (wood-plastic) mixture samples were collected using an FT-NIR spectrometer (Antaris II FT-NIR analyzer, Thermo Scientific Co., Waltham, MA, USA) equipped with an InGaAs detector. The NIR spectra were obtained over a range of 1,000 - 2,500 nm with a spectral resolution of 4 cm⁻¹. Therefore, the resulted spectra provides 1557 variables for each scanned sample. In order to collect the spectra of powdered samples in an effective way, the sample holder was furnished with an accessory that contain a central hole to maintain the uniform shape and thickness of sample over the irradiate surface through out the spec-

tra collection. However, for the tablet samples, a different sample holder was used which allowed us to fit the sample according to sample diameter for consistent sampling. The samples were analyzed in the reflectance mode, and a total of 32 scans for each sample were collected and averaged for analysis. A background scan was obtained before every sample with a golden slit. In order to avoid interference from the preceding sample, the sample holder was cleaned properly.

FT-NIR measurements were performed in the near-infrared region (1,000 - 2,000 nm) using an FT-NIR spectrometer (Antaris II FT-NIR Analyzer, Thermo scientific Co., USA). A background scan was obtained before every sample scan with an empty sample plate. Each sample was collected at a wavelength range between 4,000 and 10,000 cm⁻¹ (1,000 - 2,500 nm) at 4 cm⁻¹ intervals. The total of 32 successive scans from each sample was obtained, and the mean spectra were used for the analysis.

2.2.2. FT-IR measurements

FT-IR measurements were taken with a Nicolet 6700 (Thermo Scientific Co.) FT-IR spectrometer with a resolution of 4 cm^{-1} and an average of 32 scans. The collected spectral data covers a spectral range from $4,000 - 650\text{ cm}^{-1}$ and a total of 1738 Variables (wavebands). The FT-IR spectrometer was equipped with an attenuated total reflectance (ATR) accessory sampling technique which uses the phenomenon of total internal reflection. The reflectance spectra were collected by placing the sample on diamond crystal sampling plate clamped with a pointed tip. A single beam spectra before each sample was measured against air as a background. In order to eliminate the influence of residual from preceding sample, both ATR crystal and pointed tip were cleaned before measuring the new sample. The spectra collection was done by using OMNIC software.

2.2.3. Chemometrics for data processing

Spectroscopic data usually consists of several hundreds to thousands of variables and difficult to interpret directly because of the effect of a large number of factors, such as light scattering, base line shift, instrumental drift, and so forth. Therefore, the use of the chemometrics is always required to extract the relevant information and suppressing the effects of noise caused by the aforementioned reasons (Lohumi *et al.*, 2015). Thus, in this study, spectral data are first corrected manually by removing the unnecessary spectral region followed by the different preprocessing treatments, and finally, a

multivariate calibration model of partial least square regression (PLS-R) was developed to predict the added plastic concentration in WPC samples. MATLAB software version 7.0.4 (The Mathworks, Nitick, MA, USA) was used to perform all of the chemometric analyses.

2.2.3.1. Spectra preprocessing

Spectral data are not competent to direct analysis as the existing variability that caused by physical phenomenon such as; different in particle size, physical properties, etc., can leads to miss classification or false prediction. Therefore, preprocessing of spectral data has a great importance in chemometrics modeling. In this work, to correct unwanted signals, both FT-NIR and FT-IR raw spectra were pre-processed with seven different preprocessing methods and can be divided into two categories: scatter correction methods includes normalization (mean, maximum, and range), multiplicative scatter correction (MSC) and standard normal variate (SNV), and smoothing method includes Savitzky-Golay (SG) 1st and 2nd derivatives. The normalization methods generally adopted to suppress the variability in the spectra caused by scattering effect influenced by morphological changes among the sample (Fearn *et al.*, 2009, Esquerre *et al.*, 2012). The smoothing method of Savitzky-Golay use a smoothing of the spectra before calculating the derivatives to remove the baseline variation (Brown *et al.*, 2000).

Table 3. Statistics for the data sets (excluding number of samples, all units in %)

		Number of samples	Minimum	Maximum	Mean ± standard deviation
FT-NIR (powder)	Calibration	50 (10)	40	58	49.9 ± 6.57
	Prediction	25 (5)	40	58	49.9 ± 6.66
FT-NIR (tablets)	Calibration	65 (13)	40	58	49.9 ± 6.58
	Prediction	35 (7)	40	58	49.9 ± 6.63
FT-IR (powder & tablets)	Calibration	65 (13)	40	58	49.9 ± 6.58
	Prediction	35 (7)	40	58	49.9 ± 6.63

2.2.3.2. Modeling calibration and validation

A multivariate calibration method of partial least square regression (PLS-R) was then established separately with all seven kinds of pre-processing methods including raw spectra. PLS-R is a method to model the relationship between two matrices, data matrix (X), and response variable (Y). PLS-R is particularly suited to deal with a large number of spectral variables as it acts by compressing the large amount of spectral data into few latent variables able to describe the maximum covariance between response variable (Y) and spectral data (Kresta *et al.*, 1994).

The PLS-R is expressed as Eq. (1 & 2):

$$X = TP^T + E \dots\dots\dots (Eq. 1)$$

$$Y = UQ^T + F \dots\dots\dots (Eq. 2)$$

Where X is spectral data matrix and Y (response variable) is reference value matrix. The T and U are score matrices projected on linear combinations and the P and C are loadings matrices. The matrices of E and F represent error matrices for X and Y data, respectively. The detailed information of PLS-R

can be found elsewhere (Wold *et al.*, 2001).

The spectral data sets were divided into calibration and validation sets. Summary of the descriptive statistics for the calibration and validation data sets is given in Table 3. A leave-one-out cross validation method was used to select the optimum number of latent variables (factors) and to avoid overfitting problem. The optimum number of latent variables were chosen based on the lowest root mean square error of cross validation (RMSECV) and can be expressed as:

$$RMSECV = \sqrt{\frac{1}{z} \sum_{i=1}^z (y_i - \hat{y}_i)^2} \dots\dots\dots (Eq. 3)$$

Where z is the number of variables, the actual reference value; and the predicted value obtained from the PLS-R model developed with cross validation set.

3. RESULTS and DISCUSSION

3.1. Spectral interpretation

Fig. 1(a) shows the FT-NIR spectra of pure wood and plastic powder. A large variation in

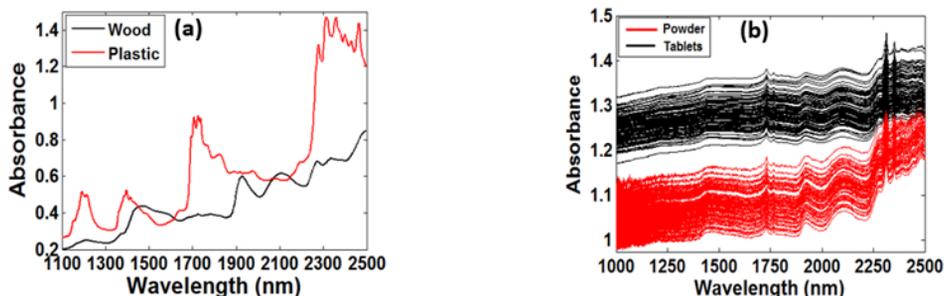


Fig. 1. FT-NIR spectra of pure wood and plastic powder (a), and FT-NIR spectra of powder and tablet form of WPC at different concentration of added plastic.

spectral pattern and intensity is obvious from the figure. The major peaks that represent chemical constituent in plastic can be seen around 1,200 nm, 1,400 nm, 1,740 nm, and the far end part of the spectra. On the other hand, the pure wood powder spectra comparatively lower intensity and few bellies around 1,490 nm, 1,930 nm, 2,110 nm and few small peaks around 2,300 nm. Wood powder has a lower intensity throughout the entire course of spectrum, but at two regions, 1,460 - 1,590 nm and 2,070 - 2,140 nm shows higher intensity. Absorption in the region of 1,460 - 1,590 nm are associate with O-H stretching and represent the moisture content (Osborne, 2006). The region from 2,070 - 2,140 nm centered at 2,110 nm attribute to combination of O-H and C-H stretching vibrations (Wojciak *et al.*, 2014).

The FT-NIR original spectra of the WPC for both powder and tablet form is given in Fig. 1(b). Tablet spectra shows higher intensity compare to powder spectra. The higher intensity for the tablet spectra could be because of the higher density of WPC tablet samples. However, both powder and tablet spectra shows a similar

spectral pattern, the only different can be observed at early part of the spectrum (1000 - 1340 nm). Powdered WPC spectra at this region seems to be affected by noise and might be caused by the variation in particle size among the samples.

Since, the FT-IR measurement technique is somehow different from FT-NIR measurement technique because FT-IR use ATR cell and scanning point is much smaller than that of FT-NIR. Therefore, the spectra were collected very carefully, and checked for any spectral abnormality caused by physical variation in samples as well as background effect. Fig. 2a shows typical FT-IR reflectance spectra of pure wood and plastic powder samples and the raw spectra of both powder and tablet WPC samples depicted in Fig 2(b). While spectra collection for both powdered or tablet samples, we observed two spectral regions [see Fig. 2(a) & 2(b)] that are very sensitive to physical variation in samples ($4,000 - 3,500 \text{ cm}^{-1}$), and background sensitive region ($2,400 - 2,280 \text{ cm}^{-1}$) presenta CO_2 artifact peak centered at $2,340 \text{ cm}^{-1}$. Both wood and plastic spectra shows

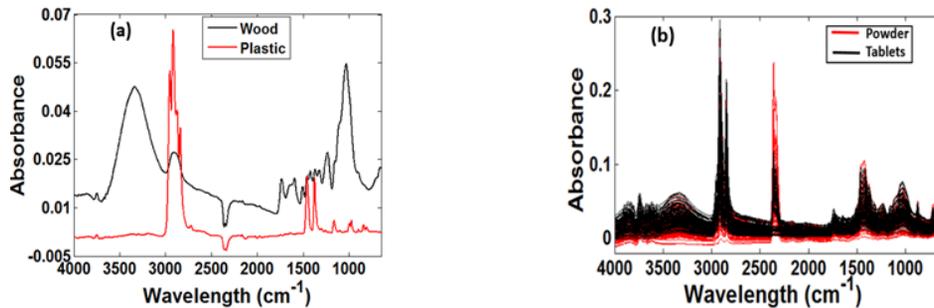


Fig. 2. FT-IR spectra of pure wood and plastic powder (a), and FT-IR spectra of powder and tablet form of WPC at different concentration of added plastic.

some distinct peaks at a particular spectral region. The spectrum of wood shows a high absorption region 3,500 - 3,100 cm⁻¹ because of the O-H stretching bond, and an other small bump at 3,000 - 2,800 cm⁻¹ related to C-H stretching in methyl and methylene groups (Kallavus *et al.*, 2015). A sharp peak around 1040 cm⁻¹, attribute to the linkage between the sugars units (Chen *et al.*, 2016). Plastic has a specific absorption band at 2,920 cm⁻¹, which correspond to a symmetric CH₂ stretching. The vibration at 1,470 cm⁻¹ possibly because of the amorphous content of polyethylene (Stark *et al.*, 2004).

3.2. Partial least square regression analysis

Spectra of samples from both FT-NIR and FT-IR spectroscopy were preprocessed with several previously discussed preprocessing methods and a PLS-R model was then developed to predict plastic concentration in WPC. For the PLS-R analysis, the spectral data of samples were arranged in a matrix that columns represent the number of variables (wavebands)

and rows represent the number of samples. The data sets were divided randomly in to calibration and prediction sets listed in Table 3. Abnormal spectral region can severely decrease the prediction accuracy of the developed model. Therefore the spectral region from 1,000 - 1,340 nm of FT-NIR spectra and 4,000 - 3,500 cm⁻¹, and 2,400 - 2,280 cm⁻¹ of FT-IR spectra were considered as irrelevant region to develop a robust model. It should be noted that PLS-R models were developed separately with tablet and powdered samples of WPCs.

Fig. 3 plots the data from actual concentration against the PLS-R predicted concentration for plastic content in WPC. PLS-R result obtained for FT-NIR data yield a good relationship ($R_p^2 = 0.87$) between actual and predicted concentration values for tablet samples; however, a relatively low coefficient of determination ($R_p^2 = 0.75$) and high standard error of prediction (SEP = 3.4) for powder samples was obtained using PLS-R model. The comparatively lower prediction accuracy for FT-NIR data of powder samples may be because of the fact that the variation in particle size highly in-

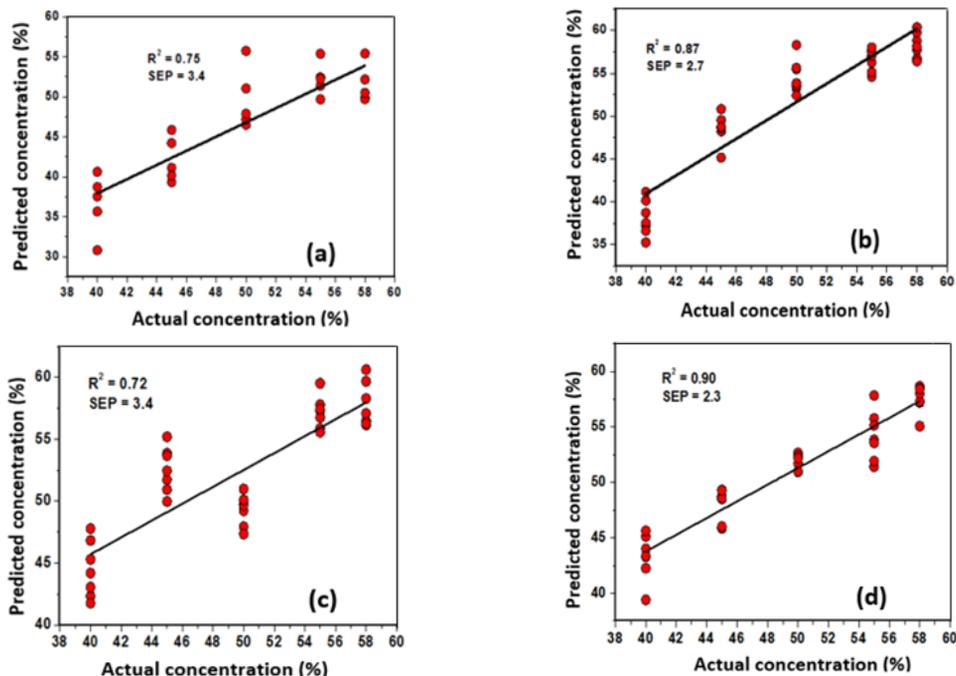


Fig. 3. Data from actual values versus PLS-R predicted values for FT-NIR data in the prediction sets for powder (a) and tablet (b), and for FT-IR data for powder (c) and tablet (d).

fluence the spectral data and lead to the misclassification or poor prediction. It must be noticed that all calibration and prediction sets for powder and tableted WPC from FT-NIR and FT-IR spectroscopy treated with aforementioned seven preprocessing methods and the best prediction results yielded are presented in Fig. 3 and Table 4.

In the case of FT-IR spectral data, following the aforementioned rules, PLS-R model developed with tablet WPC samples afforded higher prediction accuracy ($R_p^2 = 0.90$) than the model developed with powdered WPC ($R_p^2 = 0.72$). In both cases (FT-NIR and FT-IR) better results were achieved with tableted WPC. Moreover, no big difference in the number of selected fac-

tors can be seen in Table 4. Comparatively, PLS-R model developed with FT-IR data of powdered WPC show slower prediction accuracy than the model developed with FT-NIR data of the same. This may be a reason of smaller amount of sample used for ATR-FT-IR analysis.

The beta coefficient plots (Fig. 4) of the PLS-R model, which represent the spectral difference among different group of samples, demonstrate several distinct peaks that arose owing to the wood and plastic content of the WPC. The highest absolute values of the beta coefficient considered the most important wavebands responsible for the prediction and interpretation of the model (Kandpal *et al.*, 2016).

Table 4. Prediction results from the PLS-R model for different data sets

Methods	Preprocessing	Calibration		Prediction		Factors
		R ²	SEC (%)	R _p ²	SEC (%)	
FT-NIR	Max norm ^{a)}	0.99	0.51	0.75	3.41	7
	SNV ^{b)}	0.96	1.16	0.87	2.65	7
FT-IR	S-Golay 1 st c)	0.91	1.97	0.72	3.41	8
	S-Golay 1 st c)	0.92	1.78	0.90	2.31	7
Combine	Raw spectra	0.92	1.88	0.80	2.91	6
	Raw spectra	0.87	2.38	0.87	2.56	6

^{a)} Maximum normalization

^{b)} Standard normal variate

^{c)} Savitzky-Golay 1st

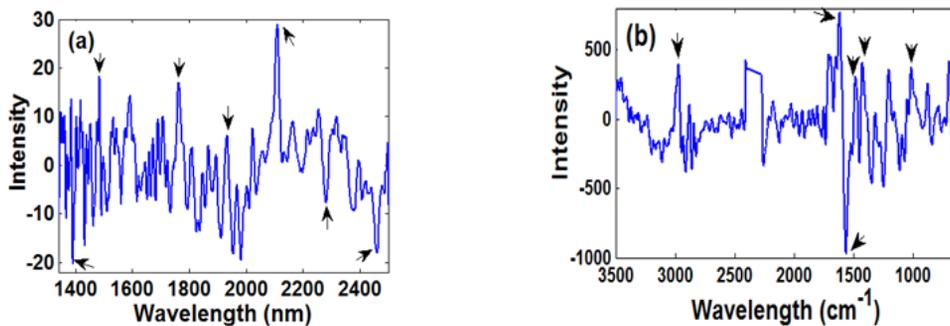


Fig. 4. Beta coefficient curve of PLS-R model developed with Maximum normalized FT-NIR spectra (a), and Savitzky-Golay 1st derivative preprocessed FT-IR spectra of powdered WPC samples.

In this study, the major absolute peaks in Fig. 2a and 2b (marked with arrow) are the same as those in pure wood and plastic spectrum of FT-NIR and FT-IR, respectively. The beta coefficient curve of PLS-R model developed with FT-NIR spectra of WPCs indicated peaks at 1,490, 1,930, 2,110, and 2,280 nm assigned to the wood component, and 1,395, 1,750 and 2,460 nm bands are related to the plastic concentration in WPC. In addition, the beta coefficient obtained for FT-IR data of WPCs shows some peaks at 2,970, 1,620, 1,560, 1,485, 1,425, and 1,030 cm⁻¹. These peaks at

particular wave bands can be considered significant because FT-IR spectra of pure wood and plastic [see Fig. 2a] represent the high absorption intensity at around similar bands. The beta coefficient curve given in Fig. 2 are from PLS-R model developed with FT-NIR and FT-IR data of tableted WPCs. However, the beta coefficient from the model developed with powdered data shows similar pattern (peaks) with few more minor peaks could be a reason of higher noise seen with the powder samples spectra.

3.3. Combined FT-NIR and FT-IR spectra

The combination of FT-NIR and FT-IR spectroscopy has been employed to investigate that whether performance of multivariate model can be improved by using a combination of NIR and MIR region of spectra. Before combining the spectra, unwanted regions from both FT-NIR and FT-IR spectra were discarded. Therefore, two PLS-R model, one with powder and another with tableted WPCs samples were developed with combined spectra without using any-preprocessing method. Table 4 shows the calibration and prediction results obtained for combined spectra. The model developed with combined spectra of powder samples afforded higher prediction accuracy ($R_p^2 = 0.80$, $SEP = 2.91$) than the model developed individually with FT-NIR data ($R_p^2 = 0.75$, $SEP = 3.41$) and FT-IR data ($R_p^2 = 0.72$, $SEP = 3.41$). However, no improvement in prediction result was yielded for combined data from tableted WPCs. It can be seen that results achieved when using the combined spectra tend to be slightly better or similar than those obtained using a single technique.

4. CONCLUSION

The added plastic concentration in WPCs has been evaluated because of the great practical importance of this concern. Spectral data were collected using FT-NIR and FT-IR spectroscopies and evaluated through multivariate analytical method of partial least squares regression

(PLS-R) approach to predict plastic concentration in WPCs. The presented results demonstrated that both FT-NIR and FT-IR spectroscopies combined with PLS-R is a useful tool for a rapid and reliable estimation of the plastic contents in WPCs. Comparison of the prediction models from the different spectroscopic methods indicated that FT-IR spectroscopy yielded slightly better accuracy than FT-NIR spectroscopy for tableted WPCs. However, limitation of developed technique occurred with powdered WPCs because of the variation in particle size. Further, the combination of these two spectroscopic techniques can be used to improve the prediction accuracy when powdered WPC is concerned.

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