

Research Article

Characterization of Biochar Properties Affected by Different Pyrolysis Temperatures Using Visible-Near-Infrared Spectroscopy

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Rapid characterization of biochar for energy and ecological purpose utilization is fundamental. In this work, visible and near-infrared (vis-NIR) spectroscopy was used to measure ash, volatile matter, fixed carbon contents, and calorific value of three types of biochar produced from pine wood, cedar wood, and cotton stalk, respectively. The vis-NIR spectroscopy was also used to discriminate biochar feedstock types and pyrolysis temperature. Prediction result shows that partial least squares (PLS) regression calibrating the spectra to the values of biochar properties achieved very good or excellent performance with coefficient of determination (R^2) of 0.86~0.91 and residual prediction deviation (RPD) of 2.58~3.32 for ash, volatile matter, and fixed carbon, and good prediction with R^2 of 0.81 and RPD of 2.30 for calorific value. Linear discrimination analysis (LDA) of the principal components (PCs) produced from PCA of wavelength matrix shows that three types of biochar can be successfully discriminated with 95.2% accuracy. The classification of biochar with different pyrolysis temperatures can be conducted with 69% accuracy for all three types and 100% accuracy for single type of cotton stalk. This experiment suggests that the vis-NIR spectroscopy is promising as an alternative of traditionally quantitative and qualitative analysis of biochar properties.

1. Introduction

Biochar is the product of thermal degradation of organic materials in the absence of air (pyrolysis). Except for being an excellent material for energy purpose utilization, biochar has also been described as a possible means to improve soil fertility as well as other ecosystem services and sequester carbon (C) to mitigate climate change [1, 2]. Conversion of agricultural wastes into biochars not only can save natural resources but also protect environment. Biochar quality is mainly influenced by its feedstock type as well as pyrolysis conditions. However, it is very cost- and time-consuming to determinate biochar properties, for example, to measure calorific value, by using conventionally physic-chemical means in laboratory. Thus, it is necessary to develop an alternative for rapid characterization of biochar properties.

Recently, near-infrared reflectance spectroscopy (NIRS) has received increasing attention because it is characteristic

of rapid measurement, ease to use, and absence of agents. This technology has been used for the analysis of biomass feedstock properties. Sanderson et al. [3] used the NIRS to determine the chemical compositions of several woody and herbaceous feedstocks, such as ethanol extractives, ash, and lignin. Labb   et al. [4] found the orthogonal signal correction- (OSC-) treated kernel PLS method achieved highest coefficient of correlation and lowest root-mean square of error (RMSE) for the prediction of ash and char content of three types of woody biomass (red oak, yellow poplar, hickory) and three herbaceous biomasses (switch grass, corn stover, sugarcane bagasse). Allison et al. [5] measured several key compositional parameters (alkali index, nitrogen, carbon, and ash contents) in two species of energy grass (switch grass and reed canary grass) by Fourier transform infrared spectroscopy. Nkansah et al. [6] used the NIRS as a process analysis technology tool for the rapid characterization of physical (bulk density) and chemical

properties (insoluble and acid-soluble lignin, extractives, ash) of yellow-poplar. Fagan et al. [7] predicted the moisture, calorific value, ash, and carbon content of two dedicated bioenergy crops (*Miscanthus* and Short Rotational Coppice Willow). All reports above focused on the measurement of biomass feedstock properties. To our knowledge, however, there is no report on using the NIRS to predict biochar properties.

As known, weak overtones and combinations of the IR fundamental vibrations due to the stretching and bending of N–H, O–H, and C–H groups dominate the NIR (780~2500 nm) and electronic transitions in the visible (350~780 nm) portions of the electromagnetic (EM) spectrum [8]. It might be practicable to use the visible and near-infrared (vis-NIR) spectroscopy for the characterization of biochar properties, although using IR range (4000~400 cm⁻¹) may produce higher performance than vis-NIR range, as shown in other reports [9, 10].

This study aims to explore the potential of using vis-NIR spectrum from 350 to 1100 nm to determinate componential contents, that is, ash, volatile matter, and fixed-carbon contents and calorific value of three selected types of biochar produced from cedar wood, pine wood, and cotton stalk under different pyrolysis temperatures. We also investigated the feasibility of the vis-NIR spectroscopy to discriminate biochar feedstock types and pyrolysis temperatures.

2. Material and Methods

2.1. Sample Preparation. Pine and cedar wood chips were collected from a local timber production factory. Cotton stalk was gathered from a local farmland. The biomass wastes were left outdoors for air-drying one month. The dried materials were broken by machine and further ground to pass through a 40 mesh screen. Biochar was produced via the pyrolysis of the raw biomass at various temperatures under oxygen-limited conditions, which allowed the materials to be charred rather than combusted. Briefly, the biomass was placed in a ceramic pot covered with a fitting lid, and underwent pyrolysis in a muffle furnace with peak temperatures of 300, 350, 400, 450, 500, 550, and 600°C for 2 h. For each temperature, four biochar samples were prepared for cedar and pine wood and 8 samples for cotton stalk. A total of 112 samples were used with 28, 28, and 56 samples for cedar wood, pine wood, and cotton stalk biomass, respectively.

From each sample, 4~5 g was used for laboratory measurement of ash, volatile matter contents (wt.%), and calorific value (MJ/kg). The ash and volatile matter contents were measured according to the criteria of NY/T1881.4-2010 (China) and the calorific value according to the standard of GB/T 213-2003 (China). Fixed carbon content was calculated by 1 – ash (wt.%) – volatile matter (wt.%). All measurements were conducted on the dry base.

2.2. Spectrum Measurement. A subsample of about 5 g was loaded into a static ring cup and measured with a Maya 2000 spectrophotometer (Ocean Optics, USA) equipped with an

optic fiber for light transmission. The light source was a halogen bulb of 3000 K. The spectrophotometer provided spectra with wavelengths from 200 to 1150 nm. Due to the noise at both ends, only the range from 350 to 1100 nm was remained for further investigation. All spectra were recorded in diffuse reflectance mode. Before sample spectral scanning, ten reference scans were taken on a ceramic standard supplied with the spectrophotometer. Twenty photometric scans were conducted and averaged for each sample.

2.3. Spectral Data Processing. The spectra were treated with the Unscrambler X10.1 (CAMO, Oslo, Norway). Spectral homogeneity of samples was tested before model calibration. A principal components analysis (PCA) of wavelength matrix was carried out for the residual X-variance calculation on all samples. The Hotelling T^2 ellipse based on the first and second principal components (PC1 and PC2) was used to find sample outliers. No samples were found outside the Hotelling T^2 ellipse. The spectra were then randomly divided into two separate sets for calibration (50%) and prediction (50%).

Several spectral preprocessing algorithms, such as Savitzky-Golay smoothing, multiplicative scatter correction (MSC), standard normal variate (SNV), the 1st and 2nd detrendings, and the 1st and 2nd derivatives, were investigated but were not useful for improving prediction performance. The only beneficial spectrum transformation was baseline offset correction (BOC), conducted by $f(\lambda) = r(\lambda) - \min r(X)$, where $r(\lambda)$ is the reflectance at a wavelength of λ and X denotes all wavelength variables. The value of the lowest point in a spectrum is subtracted from all the variables. Using BOC can eliminate light-path difference caused by rough sample surface.

The BOC-transformed spectra in the calibration set were subjected to a partial least squares (PLS) regression to build a calibration model for each biochar property. All PLS models were conducted with leave-one-out cross-validation and the number of factors (NFs) used in the PLS models was determined by minimizing the predicted residual error sum of squares (PRESS). The performance of PLS models was evaluated for the prediction set.

PLS model performance was evaluated by the root mean squared error of calibration (RMSEC), cross-validation (RMSECV), and prediction (RMSEP) as follows:

$$\text{RMSE} = \sqrt{\frac{\sum (X_m - X_p)^2}{N}}, \quad (1)$$

where X_m is the reference value, X_p is the PLS-predicted value, and N is the number of samples used for calibration or prediction. PLS models were also evaluated by the residual predictive deviation (RPD), which is the ratio of standard deviation of reference values of the prediction set to the RMSEP. The criteria adopted for RPD classification [8] was that an RPD value below 1.5 indicates very poor model predictions and that such a value could not be useful; an RPD value between 1.5 and 2.0 indicates a possibility of distinguishing between large and small values, while a value between 2.0 and 2.5 makes approximate quantitative

TABLE 1: Average value and standard deviation of ash, volatile matter (VM), fixed carbon (Fc) contents, and calorific value (CV) of three types of raw biomass and charred materials under different pyrolysis temperatures.

Feedstock type	Material property	Raw		Pyrolysis temperature						
		biomass (control)		300°C	350°C	400°C	450°C	500°C	550°C	600°C
Cedar wood	Ash (wt.%)	0.3 ± 0.1		1.5 ± 0.3	1.6 ± 0.1	1.6 ± 0.1	1.8 ± 0.1	1.8 ± 0.1	2.0 ± 0.3	2.1 ± 0.1
	VM (wt.%)	82.4 ± 0.1		37.5 ± 0.7	34.8 ± 0.3	32.6 ± 0.7	29.3 ± 0.5	25.8 ± 0.6	21.5 ± 0.5	18.4 ± 0.4
	Fc (wt.%)	17.3 ± 0.1		61.1 ± 0.9	63.7 ± 0.3	65.8 ± 0.7	69.0 ± 0.4	72.4 ± 0.6	76.5 ± 0.4	79.5 ± 0.4
	CV (MJ/kg)	20.5 ± 0.1		25.1 ± 0.1	25.8 ± 0.1	25.5 ± 0.1	27.1 ± 0.1	27.9 ± 0.1	28.9 ± 0.1	29.5 ± 0.1
Pine wood	Ash (wt.%)	0.2 ± 0.1		2.5 ± 0.1	1.7 ± 0.1	2.0 ± 0.1	2.6 ± 0.1	3.1 ± 0.1	4.1 ± 0.1	4.7 ± 0.3
	VM (wt.%)	84.4 ± 0.2		37.3 ± 0.4	32.5 ± 0.4	30.5 ± 0.3	27.0 ± 0.5	23.1 ± 0.2	19.9 ± 0.2	17.2 ± 0.7
	Fc (wt.%)	15.4 ± 0.2		60.2 ± 0.5	65.8 ± 0.5	67.5 ± 0.3	70.4 ± 0.6	73.8 ± 0.2	76.0 ± 0.2	78.1 ± 0.9
	CV (MJ/kg)	19.2 ± 0.1		25.6 ± 0.1	25.9 ± 0.1	26.5 ± 0.1	27.6 ± 0.1	28.6 ± 0.1	28.7 ± 0.1	28.8 ± 0.1
Cotton stalk	Ash (wt.%)	5.4 ± 0.1		6.0 ± 0.1	7.6 ± 0.1	8.3 ± 0.2	9.5 ± 0.2	9.2 ± 0.2	11.1 ± 0.1	10.1 ± 0.1
	VM (wt.%)	74.4 ± 0.1		32.2 ± 0.3	29.9 ± 0.3	24.6 ± 0.3	23.7 ± 0.3	20.8 ± 0.6	18.2 ± 0.7	15.6 ± 1.2
	Fc (wt.%)	20.2 ± 0.1		61.8 ± 0.4	62.5 ± 0.4	67.1 ± 0.1	66.9 ± 0.1	70.0 ± 0.4	70.7 ± 0.6	74.2 ± 1.4
	CV (MJ/kg)	18.8 ± 0.1		25.7 ± 0.1	25.2 ± 0.1	26.9 ± 0.1	25.3 ± 0.1	26.4 ± 0.1	26.9 ± 0.1	27.6 ± 0.1

TABLE 2: Intercorrelation coefficients among ash, volatile matter (VM), fixed carbon (Fc) contents, and calorific value (CV) of biochar samples.

	Ash (wt.%)	VM (wt.%)	Fc (wt.%)	CV (MJ/kg)
Ash (wt.%)	1	-0.52	0.03	-0.14
VM (wt.%)	-0.52	1	-0.87	-0.73
Fc (wt.%)	0.03	-0.87	1	0.94
CV (MJ/kg)	-0.14	-0.73	0.94	1

predictions possible. For RPD values between 2.5 and 3.0 and above 3.0, the prediction is classified as good and excellent, respectively. We also evaluated the model performance by means of the coefficient of determination (R^2) for prediction. In fact, R^2 indicates the percentage of the variance in the Y variable that is accounted for by the X variable [11]. An R^2 between 0.50 and 0.65 indicates that more than 50% of the variance in Y is accounted for by variable X , so that discrimination between high and low concentrations may be made. An R^2 between 0.66 and 0.81 indicates approximate quantitative prediction, whereas an R^2 between 0.82 and 0.90 reveals good prediction. Calibration models having an R^2 above 0.91 are considered to be excellent. Generally, a good model prediction would have large values of R^2 and RPD, and small values of RMSEC, RMSECV, and RMSEP.

To find potential patterns in the vis-NIR spectra for discriminating feedstock types and pyrolysis temperatures of biochars, linear discriminant analysis (LDA) algorithm was used to investigate the first 10 principal components (PCs) obtained from the PCA. Potential clusters for different biochar types were separated with the Mahalanobis distance [12]. The PC-LDA models were developed for the calibration set and validated with the prediction set. The PC-LDA method was also used for discriminating biochar pyrolysis temperatures based on the combined spectra of all three biochar types. By comparison, an extra PC-LDA model was built for the single-type spectra of cotton stalk, as the number of cedar wood and pine wood biochar samples was

not sufficient to develop an individual model for each of both types. Parsimonious PC-LDA models were determined in terms of minimal number of PCs for best prediction performance.

3. Results and Discussion

3.1. Characteristics of the Samples. The componential contents and calorific values of the raw biomass and charred materials were shown in Table 1. Generally, the values of each property changed with pyrolysis temperature. Taking cedar wood as an example, volatile matter (VM) contents decreased sharply from 82.4% in raw biomass to 37.5% at 300°C till to 18.4% at 600°C. Fixed carbon (Fc) content increased significantly from 17.3% in raw biomass to 61.1% at 300°C till to 79.5% at 600°C. Ash content also increased with pyrolysis temperatures. Ash contents at 300°C (1.5%) and 600°C (2.1%) were 5 and 7 times greater than that in raw biomass (0.3%). Calorific values also increased from 20.5 MJ/kg in raw biomass to 25.1 MJ/kg at 300°C and 29.5 MJ/kg at 600°C. These changes of biochar properties affected by pyrolysis temperatures were consistent with other reports on woody biomass pyrolysis [13–15]. This observation indicates that biomass carbonization was accelerated with pyrolysis temperatures [14, 16]. Generally, the primary thermal degradation of biomass occurs at a lower pyrolysis temperature. The pyrolytic volatiles were further cracked into low molecular weight organics and gases rather than biochar as the pyrolysis temperature increased [17]. Losses in hydrogen and oxygen content at high pyrolysis temperature were attributed to the cleavage and cracking of weak bonds within the biochar structure [18].

Table 2 presents the intercorrelation among ash, volatile matter, fixed carbon, and calorific value of all biochar samples. Obviously, there is a strong correlation between volatile matter and fixed carbon with correlation coefficient (r) of -0.87, which indicates that gasification of volatile matter at high temperature will lead to the increase of fixed carbon content. The correlation coefficient of 0.94

TABLE 3: Statistics of calibration and prediction samples.

Biochar property ^a	Calibration set		Prediction set	
	Range	Mean \pm s.d. ^b	Range	Mean \pm s.d.
Ash (wt.%)	1.4~11.2	4.5 \pm 3.3	1.4~11.1	4.5 \pm 3.3
VM (wt.%)	14.6~37.5	26.3 \pm 6.7	14.9~38.0	26.3 \pm 6.6
Fc (wt.%)	60.2~79.5	69.2 \pm 5.7	59.6~79.9	69.2 \pm 5.7
CV (MJ/kg)	25.1~29.5	26.9 \pm 1.4	25.0~29.6	26.9 \pm 1.4

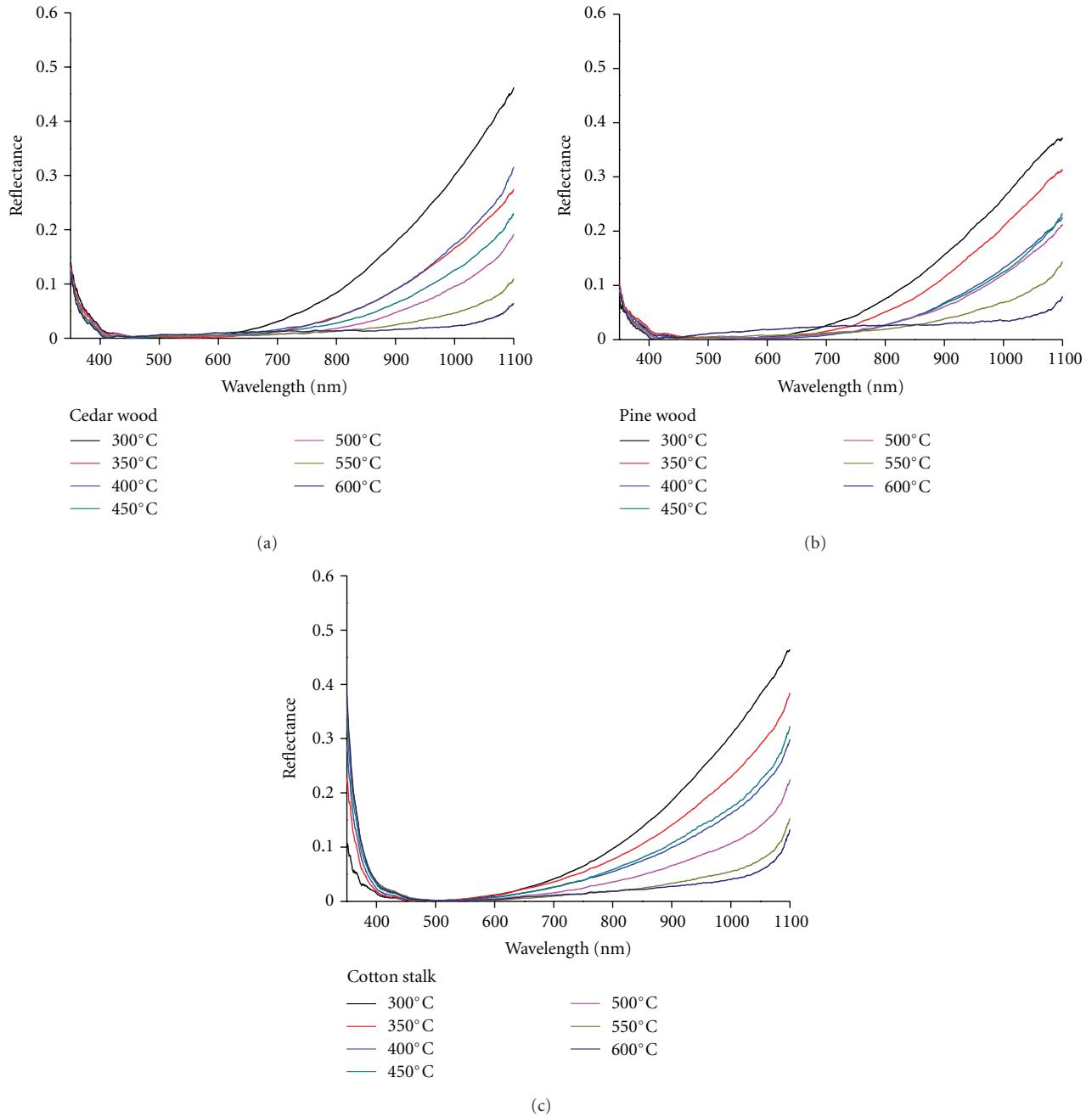
^aVM: volatile matter content; Fc: fixed carbon content; CV: calorific value.^bs.d.: standard deviation.

FIGURE 1: Mean BOC-transformed spectrum for the biochars produced from cedar wood (a), pine wood (b), and cotton stalk (c) affected by different pyrolysis temperatures.

TABLE 4: Calibration and prediction results of partial least squares (PLS) regression models developed for ash, volatile matter (VM), fixed carbon (Fc) contents and calorific value (CV) with BOC-transformed spectra of biochar samples.

Biochar property	NFs ^a	Calibration				Prediction		
		R ²	RMSEC ^b	R ²	RMSECV ^c	R ²	RMSEP ^d	RPD ^e
Ash (wt.%)	8	0.99	0.3	0.94	0.8	0.91	1.0	3.32
VM (wt.%)	3	0.89	2.2	0.87	2.4	0.86	2.4	2.77
Fc (wt.%)	3	0.90	1.8	0.88	2.0	0.86	2.1	2.70
CV (MJ/kg)	3	0.83	0.56	0.80	0.63	0.81	0.60	2.33

^aNumber of factors used in PLS model.

^bRoot mean squared error of calibration.

^cRoot mean squared error of cross-validation.

^dRoot mean squared error of prediction.

^eResidual predictive deviation, which is the ratio of standard deviation of reference values of the prediction set to the RMSEP.

between fixed carbon and calorific value may indicate the contribution of fixed carbon to biochar calorific value. Biochar properties for the calibration and prediction sets are statistically summarized in Table 3.

Figure 1 shows the changes of BOC-transformed spectra with increasing pyrolysis temperatures for each type of biochar. The spectra showed low reflectance in visible range, which corresponded to the black color of biochar. However, the reflectance became strong in the near infrared range. Pyrolysis reaction can weaken the macromolecular structure of biomass and produce biochar that is more fragile and likely to be broken [19]. Increasing pyrolysis temperature may result in a growing proportion of biochar particles with smaller size distribution [14]. The changes of biochar compositions and particle sizes may contribute to the spectrum changes.

3.2. PLS Determination of Biochar Properties. The PLSR models calibrating the wavelengths to biochar properties achieved good or excellent performance for calibration and prediction (Table 4). For example, the PLS model for ash content produced R² of 0.94 and RMSECV of 0.8% for cross-validation, and R² of 0.91, RMSEP of 1.0%, and RPD of 3.32 for prediction. The PLS models for volatile matter and fixed carbon content were both developed with 3 factors and achieved similar performance with R² of 0.87~0.88 and RMSECV of 2.0~2.4% for cross-validation, and R² of 0.86, RMSEP of 2.1~2.4 and RPD of 2.70~2.77 for prediction. By comparison, the PLS model for biochar calorific value produced middle-level accuracy with R² of 0.80 and RMSECV of 0.63 MJ/kg for cross-validation, and R² of 0.81, RMSEP of 0.60 MJ/kg and RPD of 2.33 for prediction.

3.3. Discrimination of Biochar Types and Pyrolysis Temperatures. It is difficult to identify biochar types from their visual darkness. PCA was used to convert the wavelength matrix into a small number of PCs. Linear discrimination analysis (LDA) was then conducted for the obtained PCs to find potential patterns for discriminating different biochar types. Table 5 showed the discrimination accuracy of the PC-LDA models with various numbers of PCs in calibration and prediction. For the calibration set, these models

TABLE 5: Influence of the number of principal components (PCs) on the discrimination accuracy of biochar types using principal components—linear discriminant analysis (PC-LDA) models in calibration and prediction.

Number of PCs used in PC-LDA models	Discrimination accuracy of biochar types	
	Calibration	Prediction
6	100%	90.5%
5	100%	88.1%
4	100%	95.2%
3	95.3%	90.5%
2	88%	88.1%

performed perfect with 100% discrimination accuracy with 4 or more PCs, 95.3% with 3 PCs, and 88% with 2 PCs. For the prediction set, however, the models reached the best performance of 95.2% accuracy when 4 PCs were involved, which indicated a LDA model for more than 4 PCs overfitting and that for less than 4 underfitting. Thus, the most parsimonious model for discriminating the targeted biochar types was the LDA for 4 PCs.

Table 6 shows the result of developing PC-LDA models to discriminate biochar pyrolysis temperatures. The performance of these models changed with the increasing number of PCs. For the combined types of biochar, the LDA for 4 PCs achieved best discrimination accuracy with 98.4% for calibration and 69.0% for prediction. For the single type of cotton stalk, although LDA for 3 PCs obtained 100% discrimination accuracy for the calibration set, the model just produced 85.7% accuracy for the prediction set. At least 5 PCs were needed for model prediction with 100% accuracy.

Although we did not exploit the issue of how different charring temperatures make influences on the molecular structures of the three targeted types of biochar, several published reports may lend us some useful knowledge on it. For examples, Peng et al. [20] revealed by scanning electron microscopy (SEM) that with increasing temperature, biochar particles became smaller and retained less original cell structure. Also, the Fourier transform infrared spectra of biochars derived from rice straw [20], pitch pine [14], and wood [15] showed that charring temperature alters the functional group of biochar, indicating a weaker adsorption intensity

TABLE 6: Influence of the number of principal components (PCs) on the discrimination accuracy of pyrolysis temperatures of single type of cotton stalk and combined types of biochar using principal components—linear discriminant analysis (PC-LDA) models in calibration and prediction.

Number of PCs used in PC-LDA model	Single type of cotton stalk		Combined types	
	Calibration	Prediction	Calibration	Prediction
6	100%	100%	100%	45.2%
5	100%	100%	100%	52.4%
4	100%	85.7%	98.4%	69.0%
3	100%	85.7%	92.1%	69.0%
2	95.2%	78.6%	90.5%	66.7%

of O, H, and aliphatic C but stronger adsorption intensity of aromatic C with charring temperature. Uzun et al. [21] reported that degradation of hemicelluloses and cellulose takes place at 200~300°C and 300~400°C, respectively, while lignin degradation takes place at 200~700°C. Kim et al. [14] found that formation of highly ordered aromatic structure of biochar begins at 400°C. As the chemical, physical, and morphological changes of biochar affected by charring temperature would result in spectral changes of molecular vibration, spectroscopic analysis should be a useful tool for biochar quality monitoring and feedstock identification, although more efforts must be extended for its practical application.

4. Conclusions

In the study, vis-NIR reflectance spectra with wavelengths from 350 to 1100 nm were calibrated to the ash, volatile matter, and fixed carbon contents and calorific values of three types of biochar produced from cedar wood, pine wood, and cotton stalk under various pyrolysis temperatures. The spectra were also used to discriminate biochar feedstock types and different pyrolysis temperatures. Conclusions can be drawn as follows.

- (1) Componential contents and calorific value of biochar can be predicted successfully by PLS models developed for the vis-NIR spectra. In our case, PLS models developed for BOC-transformed spectra achieved very good or excellent prediction accuracy with R^2 of 0.86~0.91 and RPD of 2.58~3.32 for ash, volatile matter, and fixed carbon contents, and good prediction with R^2 of 0.81 and RPD of 2.30 for calorific value.
- (2) It is feasible to discriminate biochar feedstock types based on a vis-NIR spectrum. In our case, the LDA model for the principal components (PCs) obtained from PCA of vis-NIR spectra produced excellent discrimination accuracy of 95.2% for the three targeted biochar types. The number of PCs should be optimized to avoid overfitting and underfitting of the PC-LDA model.
- (3) It is possible to discriminate biochars produced under different pyrolysis temperatures by PCA of vis-NIR spectra. In our case, PC-LDA model produced 100% discrimination accuracy of cotton stalk

biochars produced under 300~600°C at interval of 50°C. Although the discrimination of pyrolysis temperatures for the three biochar types based on the combined spectra only produced 69% accuracy in prediction, it may suffer from the small number of biochar samples. Thus, we suggest that larger number of biochar samples should be examined if a robust model for discriminating biochar types and/or pyrolysis temperatures is to be developed.

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