Lignin Content of Agro-forestry Biomass Negatively Affects the Resultant Biochar pH

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The biomass type is one of the most important factors that affects biochar properties, but the relationship between the major constituents of different biomasses and the properties of the derived biochars, especially the pH, is still unclear. In this study, the cellulose, hemicellulose, lignin, and ash contents of 30 representative agro-forestry biomasses and the pH, surface acid functional groups, and carbonates of 150 resultant biochars prepared with different heat treatment temperatures (HTTs) were examined. The results showed that the lignin content of the biomass had a strong negative correlation with the biochar pH ($r = -0.428$, $P < 0.01$). When the HTT was 300 °C, a high lignin content in the biomass was found to have increased the acid functional groups in the biochar, which resulted in a relatively low pH. When the HTT was greater than or equal to 400 °C, the high lignin content in the biomass negatively correlated to the carbonates, which led to a low biochar pH. It was concluded that the lignin content of agro-forestry biomass affects the biochar pH negatively, and the acid functional groups and carbonates have different effects at different HTTs.

Keywords: Biochar; pH; Lignin; Functional groups; Carbonate

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INTRODUCTION

Biochar has broad application prospects in soil improvement because of its special physicochemical properties. The optimum biochar with the desired properties should be selected based on the huge differences in the biochar physicochemical properties and various soil types, as well as the specified requirements for soil quality improvement (Lehmann 2007; Gusiatin *et al*. 2016). Therefore, biochar design and modification has become a new method to improve soil. Recently, studies on biomass pyrolysis have revealed that the characteristics of the resulting biochar can vary remarkably depending on the biomass type and pyrolysis conditions (Lehmann and Joseph 2015; Suliman *et al*. 2016). Many studies have investigated the impact of pyrolysis conditions on the physicochemical properties of the resulting biochar (Demirbas 2004; Demirbas 2006; Lv *et al*. 2010; Nanda *et al*. 2016), but little is known about the role of the biomass type. The pH is one of the most important properties when biochar is used in soil improvement. Previous studies have analyzed the effect of the pyrolysis conditions and the ash, functional groups, and carbonate contents in the biomass on the biochar pH. Studies have shown that the biochar pH increases gradually with an increase in the heat treatment temperature (HTT) (Wei *et al*. 2017; Yakout 2017). There is no noticeable effect from the heating rate on the biochar pH during intermediate pyrolysis (Angin 2013). A biochar prepared from a biomass with a high ash content has a high pH (Enders *et al*. 2012). Carbonates and functional groups are the major alkaline components in biochars (Yuan *et al*. 2011). Few studies have identified the major impacting factors and mechanisms of the biochar pH in terms of the chemical composition of the biomass. Therefore, the objective of this study was to investigate the impact mechanism of the chemical composition in the biomass on the biochar pH. A variety of biochars produced with different HTTs were used. These results were expected to provide guidance for biochar design and preparation when biochars are used in agricultural soils.

EXPERIMENTAL

Materials

Thirty types of agro-forestry biomass wastes were collected in China. Apple tree branches, grapevines, poplar branches, and bamboo were collected from the Shenyang sapling market. Pine sawdust was collected from a building site in Shenyang. Hazel, walnut, and melon seed shells were provided by Honest Nut Shop in Tieling, Liaoning Province. Buckwheat husks were collected from the Kulunqi farmland in Tongliao, Inner Mongolia Province. Bagasse was collected from the Qingjiang sugar factory in Qinzhou, Guangxi Province. Wheat straw, wheat husks, millet straw, millet husks, and cotton straw were collected from a farmland in Zhengzhou, Henan Province. Maize straw, peanut shells, rice husk, soybean straw, soybean pods, rice straw, peanut straw, bean straw, sorghum straw, corncobs, hemp straw, abutilon straw, and furfural residues were collected from the farmland of Shenyang Agricultural University. Pine needles and pine cone samples were collected from the green area of Shenyang Agricultural University.

All of the samples were dried overnight at 65 °C until the initial moisture content (ranging from 40% to 90%) dropped to less than 10%. The samples were then ground to a particle size of less than 1 cm.

Methods

Biochar preparation

The samples were loaded in lid-covered porcelain crucibles, and then pyrolyzed in a muffle furnace (SX-12-10, Zhongxingweiye, Beijing, China) at HTTs of 300 °C, 400 °C, 500 °C, 600 °C, and 700 °C with a heating rate of approximately 15 °C/min and a residence time of 30 min. Any loss in homogeneity caused by accidental combustion was prevented by removing the crucibles and leaving them to cool at room temperature with the lids on. The biochar produced was weighed and stored in airtight Falcon vials prior to further analysis.

Analysis of the biomass and biochar

The main chemical composition of biomass samples includes lignin (acid-insoluble and acid-soluble lignin), holocellulose (sum of the cellulose and hemicellulose contents), and pentosan. All of these components were measured using the standard methods of the People's Republic of China. The acid-insoluble lignin was obtained after removing the polysaccharides from the extracted biomass by hydrolysis with 72% sulfuric acid using GB/T 2677.8 (1994). The acid-soluble lignin was detected in the filtrate, where the biomass was extracted with 3% sulfuric acid during testing of the acid-insoluble lignin. The acid-soluble lignin was tested quantitatively on the basis of the ultraviolet absorbance of the filtrate (GB/T 10337 2008). Sodium chlorite was used to remove lignin from the biomass

(GB/T 2677.10 1995). The holocellulose was then obtained from the total residues. The pentosan content was primarily measured using the furfural content generated by heating the biomass with 12% hydrochloric acid based on a volumetric method or colorimetric determination (GB/T 2677.9 1994). The ash content was determined as the residue that remained after the biomass was burned at 750 °C until a constant weight was recorded (ASTM D1762-84 2007).

The pH was determined through equilibration for 90 min on a shaker according to the method described by Rajkovich *et al*. (2012). A modified biochar to deionized H_2O (w/v) dilution of 1:20 was used. The CO^{2-}-C content was determined with 1 M HCl for the analysis of the carbonate content, which is outlined in ASTM D4373-14 (2014).

The acid functional groups content was also measured. First, the biochar samples were preprocessed according to the method in Fidel (2012). Then, 1 g of biochar (particle size < 0.417 mm) was shaken for 24 h with 0.05 M HCl, and then it was washed twice with 1 M $CaCl_2$ and four times with deionized water, at a solution to biochar ratio of 50:1. Lastly, the samples were dried for more than 60 h at 50 °C.

The total content of the surface acid groups of the biochar products was determined with Boehm titration (Boehm 1994). A sample of the pretreated biochar weighing 0.15 g was added to 15 mL of 0.1 M NaOH solution and shaken with an end-over-end shaker for 40 h. The slurry was then filtered. An aliquot of 5 mL of the NaOH filtrate was transferred to 10 mL of 0.1 M HCl solution, which neutralized the unreacted base. The solution was back titrated with 0.1 M NaOH in the presence of a phenolphthalein indictor. All of the experiments were done in triplicate.

FTIR analysis was performed using a Bruker Tensor 27 using the KBr method, in a proportion of 1:300 (biochar/KBr) in weight, and pressed in pellet shape, through scanning between 4000 and 400 cm^{-1}.

Statistical analysis

The data were summarized with the means and standard deviations. Excel 2011 (Microsoft, Redmond, WA, USA) and SPSS software (version 17.0, SPSS Inc., Chicago, IL, USA) were used to conduct the data analysis. The relationship among the biomass components, carbonates, acid functional groups, and pH were investigated by computing Pearson's correlation coefficients.

RESULTS AND DISCUSSION

Diverse Biomass Chemical Composition and Various Biochar pH Values

The biomass used in this study showed great variation in the chemical composition. The cellulose, hemicellulose, lignin, and ash contents ranged from 24.89% to 58.03% (% dry matter), 1.26% to 32.47%, 11.38% to 60.87%, and 0.75% to 14.82%, respectively (Table 1).

Because of the variation in the biomass type, the biochar pH changed dramatically (Fig. 1). When the HTT was less than 500 °C, the biomass type significantly influenced the biochar pH. When the HTT was greater than or equal to 500 °C, the biomass type also affected the biochar pH, but the effect was less obvious than in the last phase. The biochar pH increased with an increase in the HTT. In particular, the rice husk biochar resulted in the maximum pH of 10.85 after pyrolysis at 600 °C, whereas the furfural residue biochar resulted in the minimum pH of 3.18 after pyrolysis at 300 °C.

Table 1. Biomass Composition (% Dry Matter)

Biomass Type	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Volatile (%)	C (%)	N (%)
Apple tree branch	34.19	26.73	24.05	3.36	81.65	45.36	1.24
Soybean straw	56.92	15.39	18.68	2.27	80.84	43.93	0.71
Abutilon straw	51.92	16.85	16.40	2.90	81.20	42.98	0.77
Bamboo	46.82	21.45	26.76	0.88	83.10	47.24	0.36
Pine cone	53.24	8.76	30.67	2.47	80.04	46.05	0.75
Wheat straw	47.72	19.02	16.72	9.16	75.26	45.96	0.83
Furfural residue	32.41	1.26	60.87	6.69	62.59	52.82	0.70
Millet husk	44.18	22.90	17.44	8.03	76.79	41.48	1.14
Walnut shell	24.89	32.47	38.28	0.75	82.57	46.58	0.68
Hazel shell	26.73	31.14	41.34	1.18	79.72	47.29	0.65
Peanut straw	44.99	18.23	11.76	5.44	77.79	45.47	1.28
Bean straw	58.03	14.79	17.51	4.97	80.69	43.18	1.42
Rice straw	47.27	18.86	14.05	10.86	76.51	39.15	0.84
Poplar branch	40.01	23.16	22.90	2.67	80.50	45.30	1.13
Buckwheat husk	51.73	16.52	28.67	2.43	77.61	46.01	0.57
Pine sawdust	48.79	18.72	25.72	4.59	82.77	45.70	0.65
Pine needle	37.40	11.09	26.38	6.11	80.09	48.70	0.96
Broomcorn straw	34.83	16.35	13.04	4.94	77.69	41.29	0.76
Soybean pod	48.81	20.18	11.38	7.41	79.69	41.06	0.83
Hemp straw	55.90	18.78	21.65	3.02	81.31	42.69	0.35
Cotton straw	46.64	17.92	22.58	5.65	77.63	43.09	1.72
Grapevine	49.08	21.65	24.73	3.07	78.11	46.22	1.04
Melon seed shell	52.38	23.51	19.28	2.22	79.59	45.91	0.47
Corncob	36.67	22.48	13.93	2.58	82.35	43.33	0.70
Millet straw	41.24	21.08	16.42	12.30	71.24	39.57	1.27
Bagasse	43.51	24.72	24.02	1.86	84.40	45.63	0.31
Wheat husk	37.16	22.34	16.27	9.50	75.01	40.79	1.43
Rice husk	36.70	16.00	21.30	14.82	68.09	39.24	0.60
Maize straw	41.40	25.00	17.60	8.77	74.61	45.45	0.80
Peanut shell	31.10	16.00	27.43	11.33	67.73	45.94	1.00

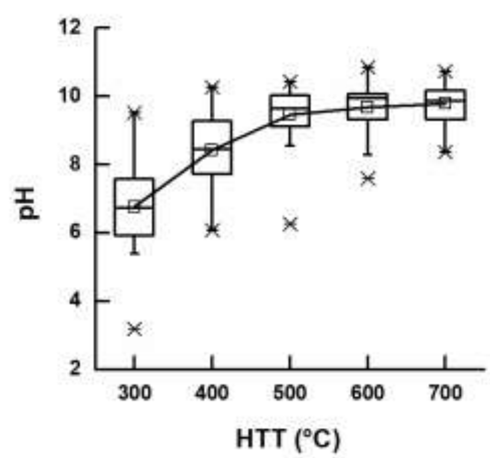

Fig. 1. Variation in the biochar pH with the biomass composition; the line and square within the box represents the median and mean values of the data; the bottom and top edges of the box indicate the 25th and 75th percentiles of the data, respectively; and the bottom and top bars represent the maximum and minimum values of the data, respectively

Several studies have indicated that the yield of biochar decreases with increasing pyrolysis temperature (Angin 2013; Zailani *et al*. 2013). From an economic point of view, optimum pyrolysis temperature should be limited to 500 °C to produce high pH biochar.

Correlation of the Biomass Composition and Biochar pH

An in-depth statistical analysis was performed to characterize the correlation between the cellulose, hemicellulose, lignin, and ash contents and the biochar pH (Fig. 2). Negative correlations were observed between the lignin content and biochar pH for all of the HTTs, while positive correlations with the biochar pH were found for the hemicellulose and ash contents when the HTT was 500 °C and 600 °C to 700 °C, respectively ($P < 0.01$). Also, no significant correlation was seen between the cellulose content and biochar pH at a P less than 0.01. Thus, the lignin content in the biomass was a key factor that affected the biochar pH. A negative correlation was observed between the lignin content and biochar pH (Fig. 3). All of the correlation coefficients reached an extreme significance level at a P less than 0.01 (n = 30). Hence, a decrease in the lignin content resulted in an enhanced biochar pH.

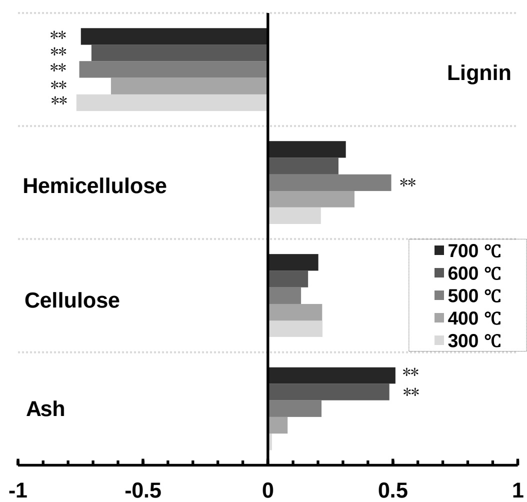

Fig. 2. Correlation analysis between the biomass composition and biochar pH for different HTTs; **significant at $P < 0.01$

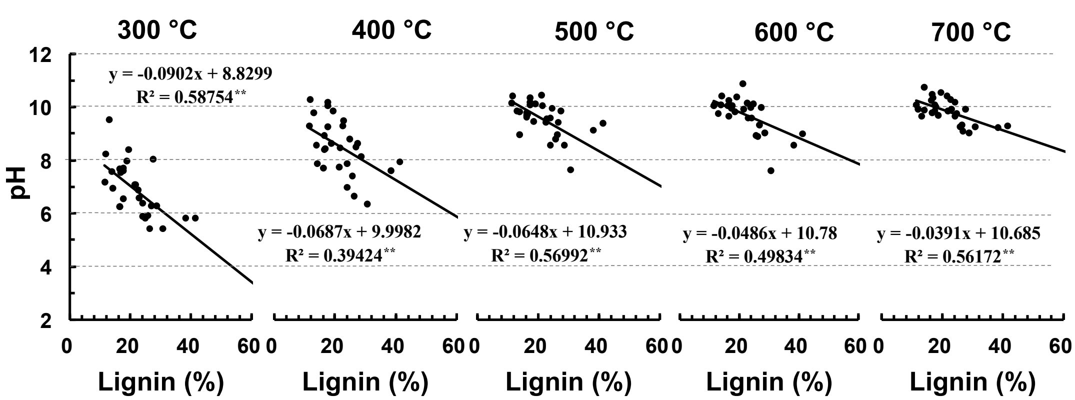

Fig. 3. Correlation analysis between the lignin content and biochar pH (n = 30); **significant at $P < 0.01$

Effect of the Lignin Content in the Biomass on the Acid Functional Groups and Carbonate Contents in the Biochar

To understand the effects of the lignin in the biomass on the biochar pH, the carbonate and acid functional groups contents of the biochar that affect the alkaline content were determined. With an increase in the HTT, the effects of the difference between the biomass lignin content on the biochar carbonate content increased (Fig. 4A) and the effects on the acid functional groups decreased (Fig. 4B). FTIR spectra of biochars were determined to validate the augmentation of acidic groups at low HTT (Fig. 5).

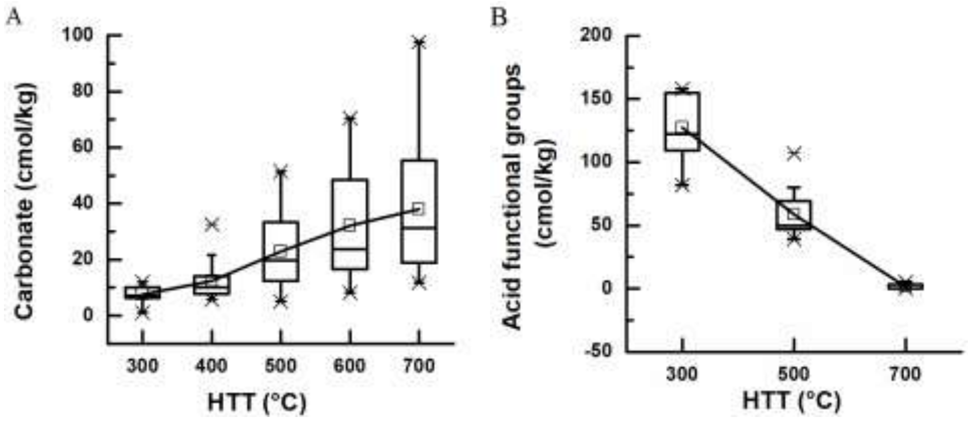

Fig. 4. Variation in the relationships between the lignin content and the carbonates (A) and acid functional groups of the biochar samples (B) (n = 30)

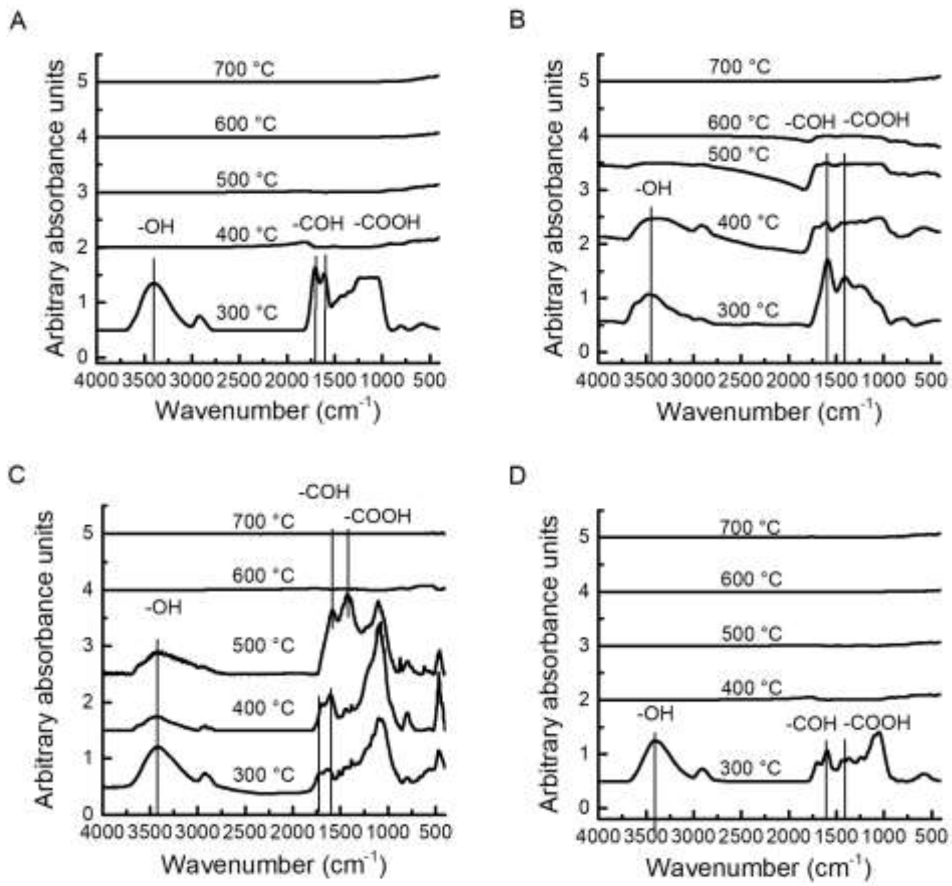

Fig. 5. FTIR spectra of the furfural residue biochar (A), pine sawdust biochar (B), rice husk biochar (C) and maize straw biochar (D)

A statistical analysis was performed to characterize the correlations between the lignin content in the biomass and the carbonates and acid functional groups in the biochar samples. A negative correlation was observed between the biomass lignin content and biochar carbonate content when the HTT was greater than or equal to 400 °C (Fig. 6), and a positive correlation was observed between the biomass lignin content and biochar total acid functional groups content when the HTT was 300 °C (Fig. 7). Despite the lower R^2 values for the correlation equations (0.17 to 0.23), all of the correlation coefficients reached extremely significant levels at a *P* less than 0.01. Although the two correlations showed opposite trends, the significant relationship suggested that the lignin content in the biomass is a key factor that negatively affects the biochar pH.

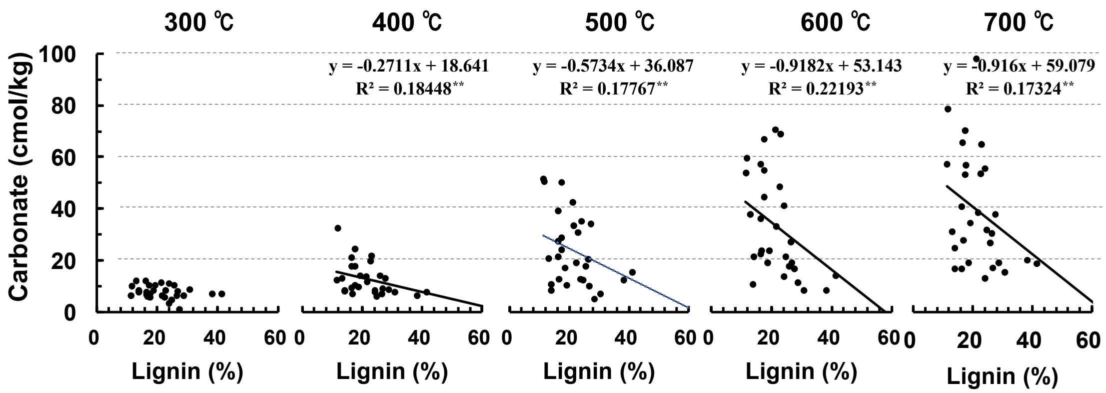

Fig. 6. Correlation analysis between the biomass lignin content and the carbonate content in the biochar samples (n = 30); ** significant at *P* < 0.01

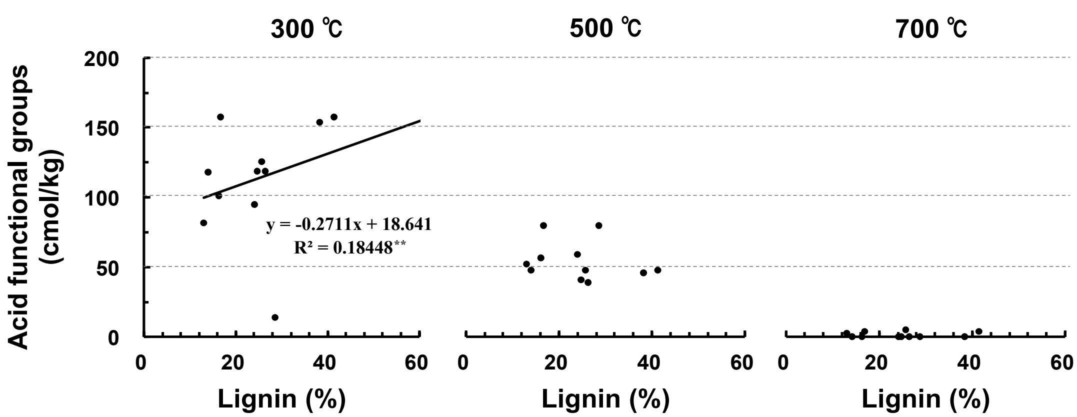

Fig. 7. Correlation analysis between the biomass lignin content and the total acid functional groups content in the biochar samples (n = 12); ** significant at *P* < 0.01

The biomass type is one of the key factors that affects the physicochemical properties of biochar (Pituello *et al*. 2015; Yavari *et al*. 2016). The large difference in the biochar physicochemical properties is because a wide range of biomass types with extremely diverse chemical compositions are available for producing biochar. Therefore, identifying specific types of biomass that are suitable to use as a raw material for biochars using small-scale samples is difficult. Thirty kinds of agro-forestry biomasses, which have considerable natural variations in their chemical composition, were used to determine the biomass composition and pH of the resultant biochars. The diversity in the biomass composition and related variations in the biochar pH allowed for a correlation analysis between the biomass composition and biochar pH to be performed. The correlation results suggested that the biochar pH is fundamentally determined by the biomass chemical composition.

Recently, the effects of the biomass ash content and pyrolysis conditions on the biochar pH have been studied (Enders *et al*. 2012). It has been determined that the carbonates and functional groups are the major alkaline components in the biochar that affect the pH (Yuan *et al*. 2011). In this study, the ash content in the biomass significantly affected the biochar pH when the HTT was 500 °C to 600 °C, but the correlation coefficient was lower than that for the lignin content (Fig. 2). Thus, it was concluded that the lignin content in the biomass is a key factor that affects the biochar pH. Although the effect of the HTT on the biochar pH was consistent with the results of previous studies, a strongly significant correlation between the biomass lignin content and biochar pH indicated that the lignin content fundamentally determines the biochar pH at any HTT. The effect of the carbonates and functional groups on the biochar pH was consistent with the results of previous studies (Yuan *et al*. 2011), but this study determined that the acid functional groups significantly affected the biochar pH when the HTT was 300 °C and the carbonates affected the biochar pH when the HTT was greater than or equal to 400 °C.

The results of this study indicated that the lignin content in the biomass negatively affected the biochar pH. A hypothesis model is summarized in Fig. 8 that indicates the main reasons for this result. The lignin content in the biomass positively affected the surface acid functional groups in the biochar when the HTT was 300 °C. The lignin in the biomass is composed of hydroxyl–phenyl lignin (H-lignin), guaiacyl lignin (G-lignin), and syringyl lignin (S-lignin) (Miller and Bellan 1997). Compared with the S-lignin and G-lignin, the H-lignin could have fewer sites that can combine with acid functional groups because of the breakage of its methoxy groups during pyrolysis.

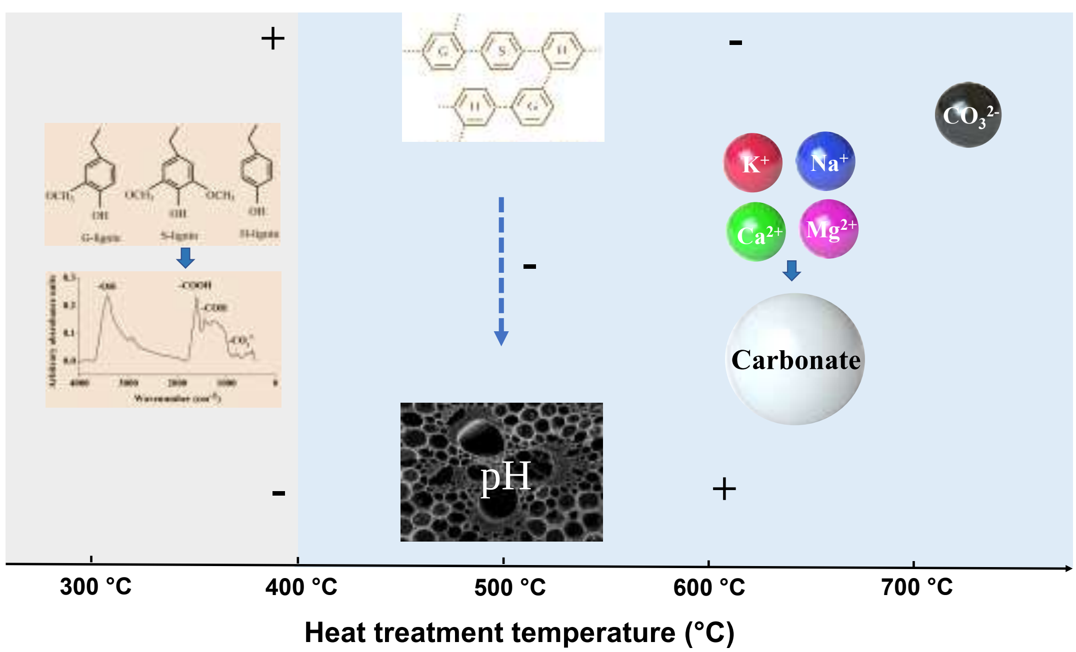

Fig. 8. Hypothesis model for the impact path of the lignin content in the biomass on the biochar pH

Existing studies have reported that the lignin content in the biomass negatively affects the H-lignin to total lignin ratio (Janse *et al*. 2000), and so the lignin might have influenced the pH through the content of surface acid functional groups in the biochar when the HTT was 300 °C. In contrast, the lignin content in the biomass negatively influenced the carbonates when the HTT was greater than or equal to 400 °C. The carbonates in the biochar were attributed to the presence of a certain amount of alkali and alkaline earth metallic (AAEM) compounds, such as K^+, Na^+, Ca^{2+}, and Mg^{2+}, as well as base ions, like CO_3^{2-}. Existing studies have reported that the lignin content in the biomass negatively affects the content of AAEM compounds (Fahmi *et al*. 2008), and so the lignin might have influenced the pH through the carbonates content in the biochar when the HTT was greater than or equal to 400 °C.

The design and oriented preparation of biochar is considered to be a new way to improve soil. The significant correlation of the biomass composition with the biochar pH suggested that the biomass composition fundamentally determines the biochar properties. Hence, this study provided insight into the mechanism of the biochar pH variation and provides guidance for the preparation of biochar for soil amendment applications.

CONCLUSIONS

1. The lignin content in the biomass negatively affected the biochar pH.
2. The analysis of the acid functional groups contents in the biochar samples demonstrated that lignin negatively affects the H-lignin to total lignin ratio and positively affects the content of the surface acid functional groups in the biochar; thus, the biochar pH was ultimately negatively affected when the HTT was 300 °C.
3. The analysis of the carbonate contents in the biochar samples concluded that lignin negatively affects the AAEM compounds content in the biomass and negatively affects the carbonate content in the biochar, and thus ultimately negatively affected the biochar pH when the HTT was greater than or equal to 400 °C.
4. These findings could provide insight into the mechanism of biochar pH variation and provide guidance for the preparation of biochar for soil amendment applications.

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