Properties and applications of biochars derived from different biomass feedstock sources

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Abstract: In China, substantial agricultural and garden wastes are burned yearly. This practice not only wastes resources but also pollutes air. Corn straw and poplar leaves are typical agricultural and garden waste in China. In this study, corn straw and poplar leaves were used to prepare biochars with different pyrolysis temperatures (250°C, 350°C, 450°C, 550°C and 650°C) and were labeled as CC (corn straw) and LC (poplar leaves), respectively. The biochars were characterized through elemental analysis, Brunauer-Emmett-Teller surface area analysis, scanning electron microscopy and Fourier transform infrared spectroscopy. Yield, ash content and biochar pH were also measured. Results showed that the two biochars possessed some similar characteristics with increasing pyrolysis temperature. These attributes included increased carbon content, biochar hydrophobicity, alkaline pH; decreased hydrogen and oxygen contents and polar functional group content; and enlarged surface area. The biochars also displayed some different characteristics, such as the obviously larger surface area of CC than that of LC at high pyrolysis temperatures and the regular holes of CC and irregular and disordered holes of LC. When biochars CC-650 and LC-650 were used as soil conditioners, the soil pH increased by 0.3 and 0.4 units, respectively, and the soil cation exchange capacity increased by 12.7% and 21.5%, respectively, with respect to those of the blank controls.

Keywords: biochars, corn straw, poplar leaves, pyrolysis, thermochemical property

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1 Introduction

Compared with the activated carbon production, biochar production is simple and low costs. Biochars also exhibit massive surface areas and are suitable for soil improvement as soil amendment. Biochar is a promising alternative for traditional soil amendments because its production location can be very near the place of use, and transportation costs are hence minimized. Biomass resources are highly substantial in China. In 2013, the corn straw yield was 240 million t, which was the highest crop straw amount in China[1]. Meanwhile, the total area of poplar plantation in China has reached more than 7 million hm^2[2]. In the “Eleventh Five-Year” plan, China intended to build timber forest bases of 9.2 million hectares[3]. However, considerable straw and garden waste would be burned annually in such case. This burning would then result in air pollution and resource wasting. However, converting these biomasses to biochar and applying the biochar to soil can realize resource recycling. Concurrently, biochars can improve soil structure and increase plant production[4]. Biochars also can increase the water retention rate of the soil and protect microbes, enlarge the soil carbon sink, and

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diminish greenhouse gas emissions[5].

Physicochemical biochar properties can change within a wide range depending not only on pyrolysis conditions[6] but also on the substrate type used to produce biochar. Biochar properties are important and enable effective and proper use as soil conditioner. Generally, biochar can be used directly as soil conditioner[7-9]. Studies indicated that adding biochars to soil can increase crop yield[10,11] and significantly augment soil pH and ion exchange capacity[12]. Biochars can also be used to remove pollutants from the water as adsorbent[13-16]. However, studies on new fertilizers produced by biochars combined with liquid digestate that contains substantial nutrients (nitrogen, phosphorus and potassium) are rare. In this work, corn straw and poplar leaves were chosen to produce biochars. These two kinds of material represent agricultural and garden wastes, respectively. Biochar properties were analyzed to observe their variations with process parameters to evaluate effects of different materials. Biochars combined with liquid digestate were applied to soil as conditioner, and the biochar effects on soil pH and soil cation exchange capacity (CEC) were investigated.

2 Materials and methods

2.1 Materials

This research chose two kinds of waste as raw materials, namely, corn straw and poplar leaves. Corn straw was obtained from a farmland at Yanqing District (Beijing, China). Poplar leaves were collected from the University of Science and Technology Beijing and naturally became yellow and fell. The two materials were first dried naturally. Then, prior to analysis, the samples were dried in an oven at around 105°C. Liquid digestate was collected from an anaerobic fermentation plant at Hegezhuang Village in Beijing. This plant uses human feces as raw material to produce biogas. The thermal regime of the plant was mesophilic.

2.2 Pyrolysis experiments

The pyrolysis temperature range of 250°C-650°C was chosen because pyrolysis temperature greatly affects the physical and chemical properties of biochars[15]. The dried materials were each placed in a ceramic crucible with lid, compacted, and then covered by the lid. Subsequently, these samples were pyrolyzed under oxygen-limited conditions in a muffle furnace at different temperatures for 2 h. Then, the biochars were milled to pass through a 0.15 mm sieve.

The biochars derived from corn straw at 250°C, 350°C, 450°C, 550°C and 650°C were labeled as CC-250, CC-350, CC-450, CC-550 and CC-650, respectively. Meanwhile, the biochars derived from the poplar leaves at 250°C, 350°C, 450°C, 550°C and 650°C were labeled as LC-250, LC-350, LC-450, LC-550 and LC-650, respectively.

To evaluate the statistical significance of the data from analytical experiments, each measurement was repeated twice or three times.

2.3 Biochar characterization

Elemental analysis of the biochars was conducted using a Vario EI Elemental Analyzer (Elementar Company, Germany). The ash content of the biochars was determined by heating at 750°C for 4 h in a muffle burner. The surface characteristics of the biochars were determined under field-emission scanning electron microscopy (SEM; EVO 18, ZEISS, Germany), whereas the surface areas were determined by a surface area analyzer (NOVA4200e, USA). Biochar pH was measured after adding 3 g biochars to 60 mL deionized water and vibrating the mixture for 5 h. The surface chemistry of the biochars was determined by a Fourier transform infrared (FTIR) spectrophotometer (Nexus 670, USA). The spectrum scope was recorded from 4000 cm^{-1} to 400 cm^{-1} with a resolution of 4 cm^{-1}.

2.4 Biochar as soil conditioner

The properties of the liquid digestate used are shown in Table 1. Biochar (10 g) was added to 10 mL of liquid digestate to produce the soil conditioner. To study the influence of biochar combined with liquid digestate on soil properties, we used soil pH and CEC as the evaluation indices. Changes in these indices were measured after the biochar was applied to the soil.

Test devices were 3 L wide-mouthed bottles labeled as blank group and test groups 1 and 2. The soil was collected from University of Science and Technology, Beijing. After the stones, glass and other debris from the soil were removed, the soil was sieved with a 2 mm screen. The soil was divided into three parts, and each part weighed 500 g (25% moisture content). One part soil was placed in a blank group bottle as control. One part soil with 20 g CC-650 biochar and liquid digestate were mixed, and then the mixture was added to test group bottle 1. Meanwhile, one part soil with 20 g of LC-650 biochar combined with liquid digestate was added to test group bottle 2. All of the bottles were exposed to the sun and placed under room temperature. Soil samples (20 g each) were obtained every 20 d, and the experiment lasted for 100 d. The pH and CEC of the soil samples were measured to investigate the effect of biochar on the soil.

Table 1 Physical and chemical properties of the liquid digestate

Parameter	COD/mg·L^{-1}	NH_4^+/mg·L^{-1}	TP/mg·L^{-1}	K/mg·L^{-1}	pH
Value	2753±462	3839±365	138±21	685±48	8.1±0.2

3 Results and discussion

3.1 Biochar properties

3.1.1 Biochar yield

The biochar yields from different pyrolysis temperatures and raw materials are shown in Table 2. The CC and LC yields declined with rising pyrolysis temperature. This result is similar to those of some previous studies[17,18]. The yield of CC-650 is 24.66% and less than half of the yield of CC-250 (60.88%). The yield of LC-650 is 27.25% and almost one third of LC-250 (76.30%). In Table 2, CC yield declines dramatically from 250°C to 450°C, then decreases slowly from 450°C to 650°C. LC yield declines dramatically from 250°C to 550°C, then decreases slowly from 550°C to 650°C. Biochar yield first declines quickly then changes slowly with increasing pyrolysis temperature.

The CC yield is lower than LC yield under the same pyrolysis temperatures. One possible reason was the higher ash content of LC than that of CC. If the ash content is deducted from the biochars, the difference in yield between CC and LC will decrease. High ash content could increases biochar yield. Another reason is the difference in structure and elemental content of the raw materials between corn straw and poplar leaves.

Although increased temperature is well documented to be linked with decreased biochar yield, biochar quality is also thoroughly reported to increase with increasing temperature[19]. Other studies have found increased carbon content and calorific value of biochar with rising temperature[20]. Biochar recalcitrance was also observed to rise with increasing temperature[21].

Table 2 Physicochemical characteristics of CC and LC at different pyrolysis temperatures

Properties	CC-250	CC-350	CC-450	CC-550	CC-650	LC-250	LC-350	LC-450	LC-550	LC-650
Ash/%	4.62	9.66	13.43	18.26	15.78	10.69	17.20	24.58	27.75	33.30
pH	6.53	7.27	8.32	10.45	11.02	7.06	9.17	10.03	10.58	11.38
Yield/%	60.88	38.87	29.09	24.73	24.66	76.30	50.18	39.02	29.84	27.25
Yield deducting ash/%	58.06	35.13	25.18	20.21	20.77	68.14	41.55	29.42	21.56	18.18
Surface area/m^2·g^{-1}	3.52	38.43	130.06	311.06	465.02	11.60	11.88	64.57	92.34	184.06
C/%	56.38	65.32	70.02	72.42	76.73	52.31	55.94	53.33	52.29	54.57
H/%	4.93	3.79	3.02	2.56	1.97	5.77	4.62	3.19	2.53	1.77
N/%	0.75	0.92	1.01	0.89	0.70	1.15	1.54	1.53	1.47	1.30
O/%	33.32	20.31	12.52	5.86	4.81	30.08	20.70	17.37	15.97	9.06
H/C	1.05	0.70	0.52	0.42	0.31	1.32	0.99	0.72	0.58	0.39
(O+N)/C	0.45	0.25	0.15	0.07	0.05	0.45	0.30	0.27	0.25	0.14
O/C	0.44	0.23	0.13	0.06	0.05	0.43	0.28	0.24	0.23	0.12

3.1.2 Biochar pH

The pH of different temperatures and raw materials are shown in Table 2. The pH increased with rising pyrolysis temperature in both CC and LC, and pH rose from 6.53 (CC-250) to 11 (CC-650) and from 7.06 (LC-250) to 11.38 (LC-650), respectively. The alkaline of biochars was enhanced with rising pyrolysis temperature. This result may be due to the biochar ash content, which presented similar variation trends with pH changes (Table 2). Moreover, alkali metal salts (Na, K, Ca and Mg) were separated out from organic matter. Many studies showed that biochar pH increases with rising pyrolysis temperature[17]. Yuan et al.[22] demonstrated that the main form of alkalis in the biochars derived from crop straw was carbonate. Carbonates substantially influence biochar alkalinity with rising pyrolysis temperature. Some organic functional groups also partially contribute to alkalinity. The organic functional groups can neutralize acidic substances and increase biochar pH. Research also found that these organic functional groups slightly influence biochars at high pyrolysis temperatures. The increase of ash components may account for the increase of biochar pH[23].

3.1.3 Thermochemical properties of biochar products

The elemental content of biochars is shown in Table 2. C, H and N contents can be determined by an elemental analyzer directly, and the O content is calculated by the method: O = 100%–C–N–H–Ash. The elemental content was substantially distinctive from the biochars of different pyrolysis temperatures and raw materials. The C content of CC increased with rising pyrolysis temperature, whereas that of LC showed no obvious rules. This discrepancy may be due to the influence of ash content, which was high in LC. Ash does not contribute to the comparison of elemental content among different biochars; thus, ash-content-deducted elemental content was calculated (Table 3). Accordingly, C content increased and H and O contents declined with rising pyrolysis temperatures (both CC and LC) (Table 3). This result indicates that carbon was enriched with the increasing temperature, which is consistent with a previous report where carbon in pitch-pine biochar was enriched when the temperature rose from 300°C to 500°C[23]. Generally, H content is mainly related to the organic matter content of biochars. The results implied that organic matter declined with the rising pyrolysis temperature (in both CC and LC). The degree of biochar carbonation can be measured by the atomic ratio H/C. Lower atomic ratios of H/C denote higher degrees of carbonation. Carbonation degree appears to increase with rising pyrolysis temperature (both CC and LC); hence, biochar carbonation degree depends on temperature. Concurrently, the atomic ratio H/C can be used to evaluate biochar aromaticity. Low atomic ratio of H/C indicates high aromaticity[24] and may exist stably in soil[23]. The results showed that aromaticity increases gradually with rising pyrolysis temperature (both CC and LC). Higher atomic ratios of (O+N)/C indicate greater surface poplar functional groups. The findings showed that surface poplar functional group decline with rising pyrolysis temperature (both CC and LC). The atomic ratio of O/C can represent biochar hydrophobicity. The ratio was reduced as pyrolysis temperature increased because of O reduction and increased C content, indicating that the produced biochar samples became more hydrophobic and polar functional groups were reduced[25]. In addition, the cation exchange capacity was high when the O/C ratio was high, which facilitates soil nutrient retention[26]. High atomic ratios of O/C denote low hydrophobicity[27]. Results showed that hydrophobicity increases with rising pyrolysis temperature (both CC and LC).

Compared with these two kinds of biochars, the C content of CC was higher than LC in the same pyrolysis. The C contents of CC-250 and LC-250 were almost the same; however, the C content of CC-650 was significantly higher than that of LC-650. The H content of CC was lower than that of LC in the same pyrolysis temperature. Changes in elemental content resulted in increased carbonation degree and aromaticity for CC. Concurrently, the O/C and (O+N)/C of CC and LC in the same pyrolysis temperatures, CC holds a lower value of O/C and (O+N)/C. This aspect means that CC involves additional hydrophobicity and low surface poplar functional groups.

Table 3 Elemental compositions of CC and LC at different pyrolysis temperatures

Properties	CC-250	CC-350	CC-450	CC-550	CC-650	LC-250	LC-350	LC-450	LC-550	LC-650
C/%	59.11	72.30	80.89	88.60	91.11	58.57	67.56	70.71	72.37	81.81
H/%	5.17	4.20	3.49	3.13	2.34	6.46	5.58	4.23	3.50	2.65
N/%	0.79	1.02	1.17	1.09	0.83	1.29	1.86	2.03	2.03	1.95
O/%	34.93	22.48	14.46	7.17	5.71	33.68	25.00	23.03	22.10	13.59

3.1.4 Ash content

Biochar ash contents at different pyrolysis temperatures and raw materials are shown in Table 2. Ash content increases with the rising pyrolysis temperature (both CC and LC). Only the ash content of CC-650 is slightly lower than CC-550. The ash content of LC is significantly higher than that of CC at the same pyrolysis temperature. These results could be due to the fact that the leaves per unit mass accumulated more dust than that accumulated by corn straw because of the wider superficial area and larger capacity to carry impurities. Ash content typically increases with augmenting pyrolysis temperature in biochar derived from various waste materials, such as bagasse, rice husk, activated sludge, or cow biosolids[27]. This observation was contradicted by Zhang's research[15], where ash content declined with rising pyrolysis temperature.

3.1.5 FTIR analysis

The FTIR spectra of the biochars (CC and LC) are shown in Figure 1. For CC and LC, the spectra were characterized by three principal bands at wave numbers 3405-3426 cm^{-1}, 2923-2969 cm^{-1} and 1589-1624 cm^{-1}. The bands at 3405-3426 cm^{-1}, 2923-2969 cm^{-1} and 1589-1624 cm^{-1} were assigned to the stretching vibration of –OH, CH_2, and aromatic C=O ring stretching, respectively.

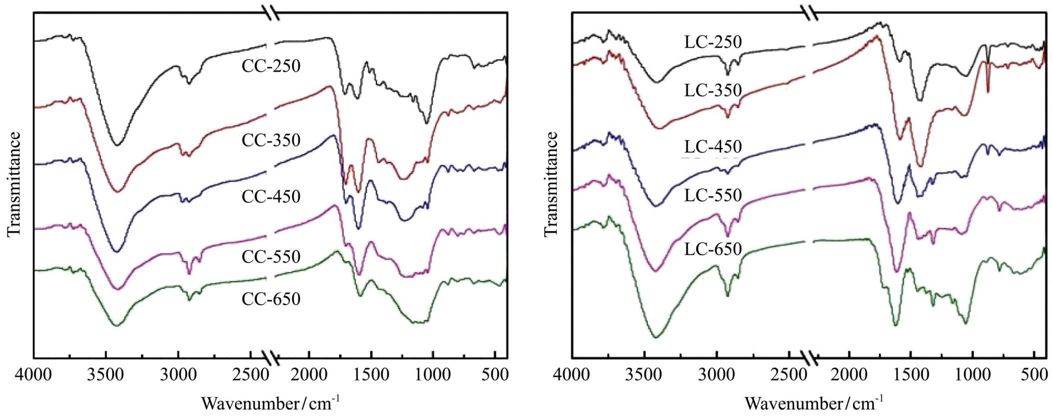

Figure 1 FTIR spectra of CC and LC under different pyrolysis temperatures

The CC spectra hold a special band at 1161-1239 cm^{-1}. The bands at 1161-1239 cm^{-1} were assigned to the phenolic hydroxyl group. CC-250, CC-350 and CC-450 acquired additional two special bands at 1704-1713 cm^{-1} and 1045-1052 cm^{-1}. The band at 1704-1713 cm^{-1} were assigned to ester C=O. The band at 1045-1052 cm^{-1} was assigned to the stretching C–O stretching vibration and in-plane O–H vibration[28]. CC-250 and CC-350 generated other bands at 1440 cm^{-1} and 1512 cm^{-1}, respectively. The peak at 1440 cm^{-1} may originate from the stretching vibrations of C=C in the aromatic ring framework.

The LC spectra held a special band at 1411-1444 cm^{-1}. The bands at 1411-1444 cm^{-1} and 1050-1086 cm^{-1} were assigned to phenolic hydroxyl group. The band at 1411-1444 cm^{-1} was assigned to COOH and CHO[29]. LC-450, LC-550 and LC-650 possessed an additional special band at 1319-1325 cm^{-1}. LC-650 also acquired a band at 1161 cm^{-1}. The FTIR spectra of the biochars showed the obvious difference in functional group in CC and LC. The functional group quantity of CC declined with rising pyrolysis temperature. However, the functional group quantity of LC increased with rising pyrolysis temperature.

The intensity of the more important bands was observed to decrease with increasing pyrolysis temperature in relation to the both of feedstock. During pyrolysis, aromatic compounds may decompose or undergo different transformations. Graphite-like carbon structure, which is not visible in the spectra, can be formed during pyrolysis as a result of condensation of undergraded rings[30]. The increase in the aromaticity of the carbon material with increasing temperature was confirmed by the decreasing value of the H/C mole ratio for individual samples.

3.1.6 SEM analysis

The biochar surface characteristics are shown in Figure 2. All biochars possess porous structures.

Temperature strongly influences the structure of different biochars. However, CC-250 and CC-350 only exhibit slightly porous structure. Although biochar contains some holes, these holes are disconnected. This structure may be attributed to the insufficient carbonization under low temperature. By contrast, CC-450, CC-550 and CC-650 contain a numerous inter-connected holes. The hole wall is thinner than that in CC-250 and CC-350. The hole walls in CC-550 and CC-650 began to collapse, and the smaller holes appeared in the walls of former holes. This phenomenon immediately increased biochar surface area. Compared with the biochars in CC, the biochars in LC possessed fewer holes, which were irregular and messy. Obvious difference existed between CC and LC. Many small pores appeared in the char because of devolatilization when the pyrolysis temperature was 650°C. This trend could be explained by the release of volatiles and intermediate-size organics with increasing temperature[31]. The cracks and pores in the chars increased in number with temperature rise, as observed previously[32].

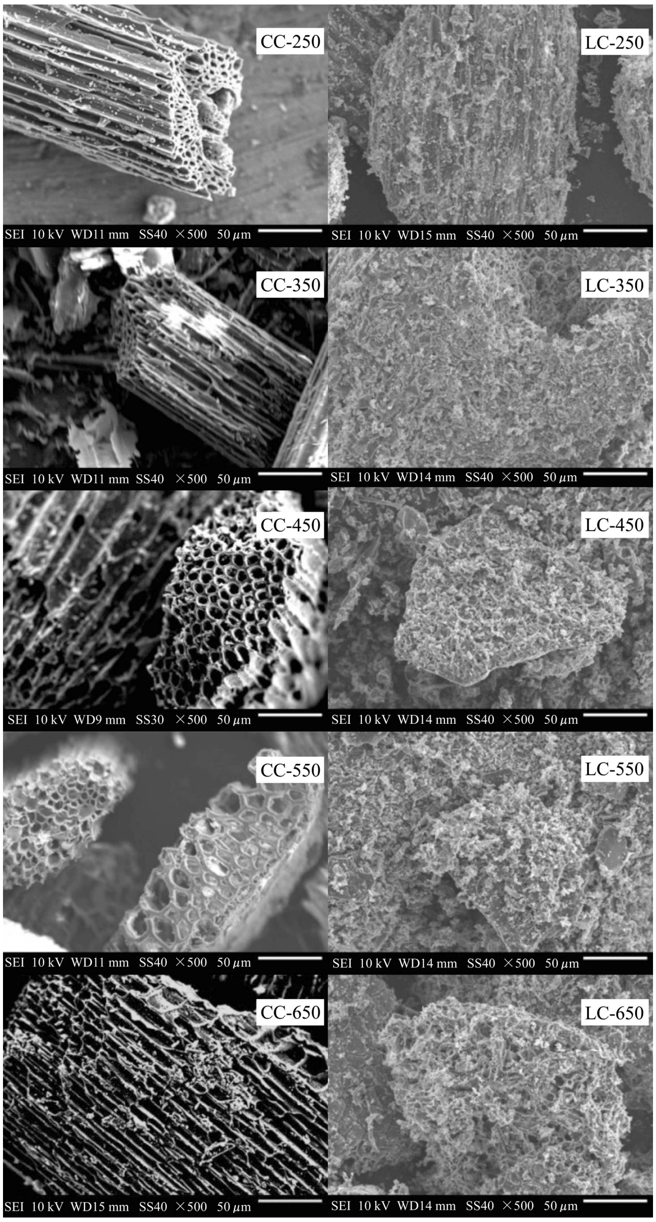

Figure 2 SEM images of CC and LC under different pyrolysis temperatures

3.1.7 Surface area

Biochar surface area changed obviously with different raw materials and pyrolysis temperature[33,34]. Biochar surface area is shown in Table 2. The total surface area increases with increasing pyrolysis temperature. CC-250, CC-350, LC-250 and LC-350 possessed a relatively low surface area. When the pyrolysis temperature is 450°C, the surface areas of CC-450 and LC-450 increased to 130.06 m^2/g and 64.57 m^2/g, respectively. Afterward, the biochar surface area rapidly increased. The surface areas of CC-650 and LC-650 reached 465.02 m^2/g and 184.06 m^2/g, respectively. Meanwhile, the surface areas of CC-650 and LC-650 were 132.2 and 15.9 times those of CC-250 and LC-250, respectively. Pyrolysis temperature is a highly important influence on surface area. This finding could be due to increasing biochar C content and the removal of carbonyl and phenolic hydroxyl of the biochars, leading to cavity growth. Concurrently, LC surface area was relatively smaller than the CC surface area under all the pyrolysis temperatures except 250°C. This finding could be due to the positive effects of corn straw structure on biochar shape and the complex cavity formation in the thin leaf structures. Maximum biochar surface areas were identified by Uchimiya et al.[35] at production temperatures between 500°C and 900°C. In this study, the Brunauer-Emmett-Teller surface areas were achieved within the optimum temperature range for the maximum surface area and indicate the upper surface area levels achievable for the feedstock assessed.

3.2 Biochar as soil conditioner

3.2.1 Soil pH

Alkaline biochar exhibits potential to buffer excess soil acidity. Biochar application to soils has been found to exert an overall liming effect, increasing soil pH[5]. However, knowledge of the initial pH of both biochar and soil is necessary to amend soil pH using biochar. Increasing the pH of the acidic soil organic matter mineralization increases nutrient availability to plants. In some circumstances, these phenomena may induce the priming effect that results in increased CO_2 emission from soils and a short-term effect on microbial activity[36].

The effect of biochar on soil pH is illustrated in Figure 3. After the biochar was added to soil, the soil pH increased with time progression. After 100 d, the pH of test groups 1 and 2 increased by 0.3 and 0.4, respectively, relative to those of the blank group. Test group 2 showed a high pH increase because of the high pH of LC-650. Hence, adding biochar to neutral soil can increase soil pH. The effect becomes notable when biochar is added to neutral soil; the increased soil pH may influence plant growth at certain soil pH. Fu et al.[24] found that the pH increased by 0.1-0.46 after the rice straw-biochar was applied to soil. This trend was mainly due to the following two reasons. First, the pH of biochar increased after pyrolysis reaction; thus, the biochar soil pH increased. Second, the CEC of biochar increased after the biochar was applied to soil and numerous alkaline ions adsorbed on the soil colloids and consequently increased soil pH.

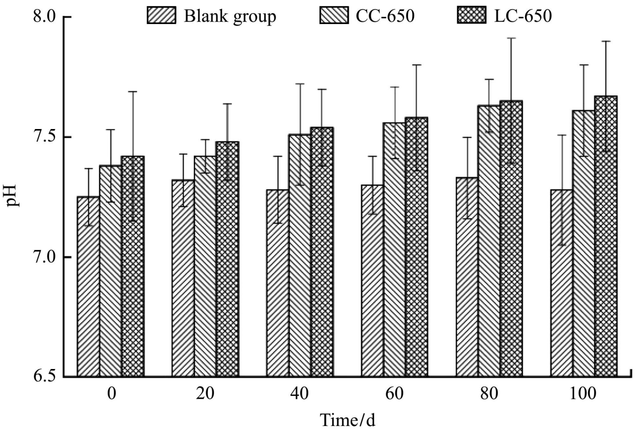

Figure 3 Effect of biochar on soil pH

3.2.2 Soil CEC

The effect of biochar on soil CEC is shown in Figure 4. After 20 d, the soil CEC of added biochars CC-650 and LC-650 increased to 11.21 cmol/kg and 11.24 cmol/kg, respectively. The soil CECs in the succeeding 80 d fluctuated but basically remained at around 13 cmol/kg. The CEC of the blank group also increased from 8.4 cmol/kg to about 10 cmol/kg. However, the CECs of test groups 1 and 2 were about 12.7% and 21.5% higher, respectively, than those of the blank group at the end of experimentation. Test group 2 holds a high CEC increase because LC-650 achieved a high ash content. CEC was related to temperature, soil particle size and moisture. According to Huang's recommendation[37], when soil CEC is above 20 cmol/kg, the soil exhibits a strong nutrient retention and supply capacity. When the soil CEC is between 10 cmol/kg and 20 cmol/kg, the soil offers medium nutrient retention and supply capacity. When the soil CEC is under 10 cmol/kg, the soil possesses weak nutrient retention and supply capacity. The soil CEC of the blank group in this experiment was about 8.4 cmol/kg at the beginning and corresponds to weak nutrient retention and supply capacity. The results showed that biochar could increase the soil CEC and improve nutrient retention and supply capacity of the soil. Fu et al.[24] found that the CEC increased by 4%-17% after the soil was supplemented with biochar-rice straw. In general, high surface area is regarded as a positive attribute in biochar used as soil amendment. The high surface areas and porosities observed in some of biochars in this work indicate good water retention properties and nutrient retention and supply capacity[37]. The soil physical properties, such as structure and water holding capacity, and the biological properties, such as microbial communities and earthworm presence, may also be altered by biochar application[5].

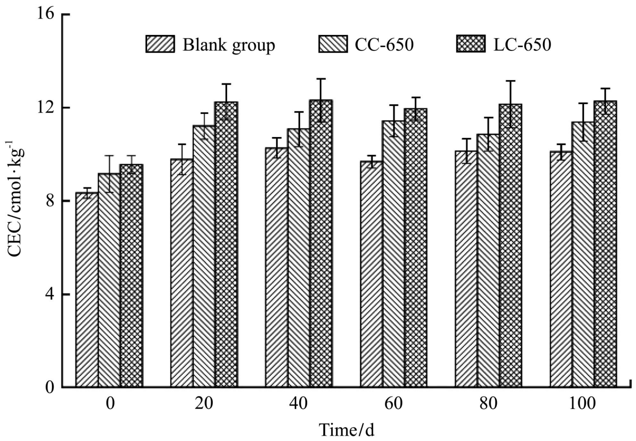

Figure 4 Effect of biochar on CEC of the soil

4 Conclusions

The C contents of CC and LC increased with rising pyrolysis temperature, but the H and O contents decreased. By calculating the O/C and (O+N)/C, the hydrophobicity of CC and LC was strengthened, and the content of their polar functional group decreased. The pH of CC and LC increased with rising pyrolysis temperature, and CC and LC were alkaline. These samples are suitable as a modifier of acidic soil. Concurrently, the surface areas of CC and LC enlarged with rising pyrolysis. However, the surface area of CC was larger than that of LC and increased more rapidly with rising pyrolysis. The surface area of CC-650 was 2.5 times larger than LC-650. SEM analysis revealed the vesicular structure of CC and LC surfaces. The holes on CC are regular and holes on LC are irregular and messy. The number of holes on CC and LC increased with rising pyrolysis temperature. Meanwhile, Holes on the surface of CC-250 and LC-250 were obviously fewer than those in the biochars with higher pyrolysis temperature.

After the addition of biochars CC-650 and LC-650, the soil pH increased by 0.3 and 0.4, CEC increased by 12.7% and 21.5% over those of the blank group, respectively. The soil nutrient retention and supply capacity also increased.

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