Pyrolysis temperature had effects on the physicochemical properties of biochar

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Citation: Li X., Liu H., Liu N., Sun Z.T., Fu S.F., Zhan X.M., Yang J.F., Zhou R.X., Zhang H.D., Zhang J.M., Han X.R. (2023): Pyrolysis temperature had effects on the physicochemical properties of biochar. Plant Soil Environ., 69: 363–373.

Abstract: Biochar is the carbon-rich product obtained when biomass is anaerobically heated. In this study, different materials (corn straw and peanut shell) and pyrolysis temperatures (350, 450 and 550 °C) affect the elemental composition, surface structure, and biochar properties. The results showed that the carbon (C) content in biochar increased as the temperature increased, but hydrogen (C) and nitrogen (C) did not change. The alkane overpressure of corn straw and peanut shell increased first and then decreased with the increased temperature; the fatty alkyl chain disappeared, but the ash content increased at 550 °C. At high temperatures, the aromaticity (C) and hydrophobicity (C) at the polarity (C) of biochar become "carbon-rich particles", while the polarity (C) the internal pore structure became larger, and a large number of micropores appeared in biochar. Biochar pyrolysed at 550 °C has much higher C, ash content, pore, and stronger buffering capacity, and thus is more promising to improve soil health.

Keywords: renewable resource; organic material; carbonisation; nutrient availability; absorption efficiency

Crop straw is an important biomass resource in the agricultural production system, with an annual production of nearly 2 billion tons worldwide (Reddy et al. 2003). Corn straw is one of the three major crop straws (Bi et al. 2009). Crop straw is one of the most abundant renewable resources. At present, corn straw is mainly used in organic amendment, household fuels, and livestock feed. The proportion of burning straw on site is large, which leads to atmospheric pollution

(Zhang et al. 2016). Another potential use of crop straw is to convert it into biochar, which can be used as a soil amendment to improve the net photosynthesis and dry matter accumulation rate of crops, as well as fertiliser utilisation efficiency (He et al. 2020, Das and Ghosh 2022, Li et al. 2023). Biochar can be made from a variety of organic materials, including agricultural (corn straw, rice straw, and wheat straw) and forestry waste (shrubs, willow leaves) (Sohi et al.

Supported by the scientific research funding project of the Shenyang Science and Technology Bureau Shenyang Black Protection Science and Technology, Project No. 22-317-2-02; by the Liaoning Provincial Department of Education, Project No. LSNJC202013; by the Liaoning Province Colleges and Universities Innovative Talents, Project No. 202038935, and by the key project of the National Key Research and Development Program "Intergovernmental Cooperation in International Science and Technology Innovation", Project No. 2021YFE0192700.

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2010, Li et al. 2022). In addition, peanut shell is also a common raw material for biochar, and peanut shell biochar improves soil organic C, nutrient availability and peanut kernel quality under different irrigation and fertiliser rate regimes in field conditions (Xu et al. 2015).

Biochar is a carbon-rich, fine-grained and porous material produced by pyrolysis of biomass raw materials under limited oxygen and relatively low temperature (300-700 °C) (Lehmann and Joseph 2009). Generally, the stronger the acidity of the soil, the greater the amount of biochar applied; in soil with high heavy metal content, the greater the amount of biochar applied (Achor et al. 2020). Applying biochar in the soil can increase carbon storage, reduce CO2 release, and be used as a soil amendment to improve physical, chemical, and biological characteristics, soil fertility, and plant productivity (Lehmann 2007). Biochar can also be used for soil water retention due to its large surface area, porous structure, and unique surface composition (Glaser et al. 2002). Since biochar can hold nutrient elements (Fellet et al. 2011, Getachew et al. 2015) adsorb heavy metals (Regmi et al. 2012, Chen et al. 2015, Goswami et al. 2016), biogas and organic pollutants (Angin et al. 2013, Maienza et al. 2017, Sethupathi et al. 2017), and inhibit greenhouse gas release (Cornelissen et al. 2013, Wang et al. 2013), biochar has attracted more attention in recent years. However, biochar's stability and adsorption capacity are related to their properties. The performance of biochar is affected by raw materials (Pellera et al. 2012), processing methods, and pyrolysis temperatures (Masiello et al. 2013, Wang et al. 2013). To achieve this, biochar with a well-developed mesopore structure could be more advantageous. It is necessary to completely assess biochar's physical, chemical, and morphological properties to elucidate their potential value as highquality material. In this study was performed on how biochar type, based on its feedstock, production temperature, and characteristics such as surface area and porosity, affect its physicochemical properties and structural characteristics.

Consequently, the objective of this study was to investigate the impact of feedstock sources and pyrolysis temperatures on several biochar physicochemical properties and structural characteristics. We produced biochar using two feedstocks, corn straw and peanut shells, and three pyrolysis temperatures, 350, 450 and 550 °C for this study.

MATERIAL AND METHODS

Biochar production. Corn straw and peanut shells were obtained from experimental field sites of Tianzhu Mountain of Shenyang Agricultural University, Shenyang, China (latitude 41°48'11.75"N, longitude 123°25'31.18"E) during the growing seasons of 2010, and were pyrolysed at 350, 450 and 550 °C for 4 h using muffle-roaster (TJ-XCT-1). The basic chemical properties of corn straw (peanut shell) were as follows: total N concentration of 1.03% (0.95%), total C concentration of 43.99% (50.84%).

Biochar chemical properties. The elementary composition analysis of biochar (C, N, H) was conducted with Vario EL III (Elementar, Germany). After 24 h in the Blast Constant Temperature Dying Oven (80 ± 1 °C), a 5–6 mg biochar sample was analysed for the element content with Vario EL III (Gao et al. 2012a, b). Biochar pH was measured following (Schomberg et al. 2012). 1.0 g biochar was added to 5.0 mL deionised water and stirred for 2 min on the Magnetic Stirring Apparatus. pH electrode was inserted into the suspension in the beaker after standing for 1 h, and pH data were recorded after the reading of the pH meter stabilising. 0.2000 g samples of biochar were weighed, washed separately 5 times with 100 mL deionised water, and washed with sodium acetate solution (100 mL, pH 8.2, 1 mol/L) 5 times that making the samples Na+ equilibration, then washed the biochar 5 times with ethanol (100 mL, 95%), so that redundant Na+ was cleared away. Finally, the biochar was washed 5 times with sodium acetate (100 mL, pH 7.0, 1 mol/L) in order to exchange the Na⁺. The filtrate was collected with ammonium acetate to a constant volume (100 mL), and the Na+ concentration was measured with a flame photometer and calculated the cation exchange capacity (CEC) of biochar (Schomberg et al. 2012).

The measurement of ash content was conducted according to methods of charcoal and charcoal experiment (GB/T 17664-1999): at first, the crucible, lid, and 1 g biochar (accurate to 0.0001 g) were weighed; then the crucible and lid with 1 g biochar were ignited in the high-temperature electric resistance furnace (800 \pm 20 °C) for 2 h; lastly, the crucible and lid with the crude ash were weighed after cooled and the ash content was worked out.

Biochar physical properties. The specific surface areas and average pore volumes of the corn straw and peanut shell were measured using a surface area meter (Micromeritics ASAP 2020 HD88, AUTOSORB IQ,

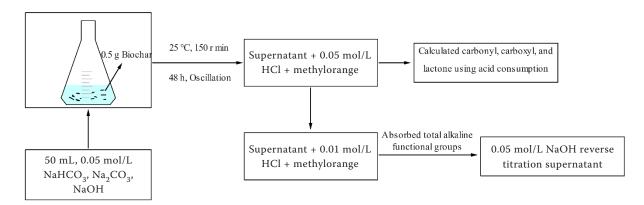


Figure 1. Boehm titration method for the determination of functional groups in biochar

Atlanta, USA) and a prosimetra analyser (Norcross, USA).

Biochar characterisation. Fourier-transformed infrared (FTIR) (Nicolet is5, Thermo Nicolet Corporation, Waltham, USA) spectra of a sample of ground parent biochar and a sample of dried biochar colloids were acquired with a platinum FTIR, Bruker, from wavenumbers of 4 000–400/cm at a spectral resolution of 4/cm (von Gunten et al. 2017).

The Boehm titration method was used to determine the number of acid-base groups on the surface of biochar, as shown in Figure 1 (Zhang et al. 2019). The dry biochar samples were observed, recorded, and micromorphological structure images were saved with the scanning electron microscope (S-4800, Hitachi, Tokio, Japan) (Zhang et al. 2011). 30 g of two raw materials (corn straw and peanut shell) were used to produce biochar at 350, 450 and 550 °C, and the carbon conversion rate was measured using the scheme described by Barros et al. (2010). The equations of carbon conversion efficiency as given below:

$$Rq/R_{\rm CO_2} = -(1-\gamma s/4)\Delta H_{\rm O_2} - \Delta H_{\rm B}~(\epsilon/1-\epsilon)$$
 where: γs – oxidation number of the C source; γs – 0 for glucose; $\Delta H_{\rm O_2}$ – Thornton's constant (Thornton 1917);

 $\Delta H_{\rm B}$ – difference in the heat of combustion of the biomass (Battley 1987, Von Stockar et al. 1993).

Statistical analysis. The data were analysed using IBM SPSS Statistics 20.0 (IBM Corporation, New York, USA), and the plots were created using Origin Pro 2016 (Northampton, USA). The treatments were compared using one-way analysis of variance (ANOVA) and Fisher's least significant difference (LSD) tests, and the differences were considered to be significant at P < 0.05.

RESULTS AND DISCUSSION

Elementary composition of biochar. The elementary composition of two kinds of biochar is presented in Table 1, it showed that C content increased with the pyrolysis temperature rising in both corn straw and peanut shell biochar. The C content of corn straw at three temperatures exceeded 60%, while that of peanut shell at three temperatures exceeded 70% and reached the highest value at 550 °C. Some previous studies demonstrated that the C content in the biochar was high (usually 60%) due to that nutrients were enriched in the pyrolysis process (Liu et al. 2009).

Table 1. The carbon (C), nitrogen (N), and hydrogen (H) content of corn straw and peanut hull biochar under different pyrolysis temperatures

	Corn straw biochar			Peanut shell biochar			
	350 °C	450 °C	550 °C	350 °C	450 °C	550 °C	
C (%)	60.22^{cB}	63.86^{bB}	68.77 ^{aB}	$70.64^{\rm bA}$	71.23 ^{bA}	78.56 ^{aA}	
N (%)	1.69^{aB}	1.72^{aB}	1.29^{bB}	2.59^{aA}	2.61^{aA}	1.55^{bA}	
H (%)	3.27^{aB}	2.51^{bB}	2.16^{cB}	3.58^{aA}	3.16^{bA}	2.82 ^{bA}	

Different uppercase letters indicate significant difference among different biochar within the same temperature, and different lowercase denotes significant difference between different temperature within the same biochar (P < 0.05)

Table 2. The specific surface area, pore volume, and size of corn straw and peanut hull at different pyrolysis temperatures

	Specific surface area (m ² /g)		Pore vol	ume (mL/g)	Pore diameter (nm)	
	BET Multi-point method	BET Single-point method	cylinder pore	parallel-plate pore	cylinder pore	parallel-plate pore
Corn straw biochar						
350 °C	2.58^{cB}	2.53^{bB}	0.0104^{bB}	0.0086^{cB}	28.09^{aA}	48.45^{aA}
450 °C	$4.04^{ m bB}$	2.68^{bB}	$0.0171^{\rm bB}$	0.0140^{bB}	7.12^{bB}	20.76^{bA}
550 °C	6.80^{aB}	5.28^{aB}	0.0323^{aB}	0.0297^{aB}	6.65^{cA}	13.92^{cA}
Peanut shell biochar						
350 °C	5.42^{cA}	5.63^{cA}	0.0171^{cA}	0.0145^{cA}	15.35^{aB}	44.17^{aB}
450 °C	33.80^{bA}	35.40^{bA}	0.0208^{bA}	0.0186^{bA}	8.55 ^{bA}	13.80^{bB}
550 °C	120.39^{aA}	113.25 ^{aA}	0.0502^{aA}	0.0403^{aA}	4.24^{cB}	6.58^{cB}

Different uppercase letters indicate significant difference among different biochar within the same temperature, and different lowercase denotes significant difference between different temperature within the same biochar (P < 0.05)

However, the N and H content of peanut shell and corn straw biochar was lowest at 550 °C. This result was consistent with Zhang et al. (2020) and Jassal et al. (2015), who reported pyrolysis temperature has significant effects on the properties of biochar, demonstrating a negative relationship with N and H content. Comparing corn straws, the C, N, and H content in peanut shell biochar was relatively higher, which can be related to the plant's raw materials and nutrition absorption accumulation. Yuan and Xu (2011) reported that the chemical composition of raw materials has a significant effect on biochar element composition and content.

Biochar physicochemical properties. The specific surface area, pore volume, and pore diameter of the biochar will affect the adsorptive property and the utilisation of base fertiliser (Li et al. 2023). Despite the high specific surface area and pore volume in peanut shell biochar, and they were highest at 550 °C (Table 2). We speculated that increasingly volatile

substances were released gradually with the increase in temperature in the pyrolysis process. In contrast, pore diameter was lowest in peanut shell biochar. The pore volume and pore diameter changed remarkably after carbonisation, which is related to the types and special organisation structure of biomass (Rawal et al. 2016). The pore diameter gradually grew with increasing temperature in the cylinder and parallel-plate pore. The main properties influencing biochar pore composition were increasing pyrolysis temperatures (Brendova et al. 2017). The pore diameter showed a declined trend with the increase of temperature after carbonisation, no matter cylinder pore or parallel-plate pore, and also the average pore diameter is bigger in corn straw biochar (Table 2). The pore size can affect the surface area of the biochar and finally influence the absorption efficiency of the adsorbent and adsorption capacity (Lawal et al. 2021, Li et al. 2023).

Table 3 showed that biochar from corn straw and peanut shells were alkaline generally; the pH value

Table 3. pH, ash, and cation exchange capacity (CEC) content of maise straw and peanut shell biochar at different pyrolysis temperatures

	Corn straw biochar			Peanut shell biochar			
	350 °C	450 °C	550 °C	350 °C	450 °C	550 °C	
рН	8.90 ^{cA}	9.93 ^{bA}	10.23 ^{aA}	7.13 ^{cB}	8.58 ^{bB}	9.11 ^{aB}	
Ash (%)	15.35^{bA}	16.31^{aA}	16.74^{aA}	5.40^{cB}	5.95 ^{bB}	6.69 ^{aB}	
CEC (cmol ₊ /kg)	114.81 ^{aA}	92.14^{aA}	$68.27^{\rm bA}$	77.86^{aB}	72.07^{aB}	63.55 ^{aB}	

Different uppercase letters indicate significant difference among different biochar within the same temperature, and different lowercase denotes significant difference between different temperature within the same biochar (P < 0.05)

appeared to increase with the rising of temperatures, and these differences in pH are significant; moreover, the pH of corn straw biochar reach the highest value (10.23) at 550 °C. This was mainly because the ash of biochar contains oxide or carbonate formation of Na, K, Mg, Ca, H, and Al, which dissolves in the water, leading to an increase in alkalinity (Oueslati et al. 2019). The pH value of biochar was related to the pyrolysis temperature and the species of raw materials (Gao et al. 2012a, b), which was similar to our research results. In practice, biochar could be used to improve soil acidity when added to soil, as the alkaline matters of biochar could neutralise the soil acidity and improve the pH value (Zhang et al. 2013).

The ash content in corn straw and peanut shells increased with the increase in temperature (Table 3). The results were consistent with the results from Zhou and Zhang (2005), which can be related to the raw materials because the raw materials contained stable mineral substance enrichment. Carbonaceous material in the raw materials decomposed and led to loss rising with preparation temperature increas-

ing. Bedmutha et al. (2011) and Nanda et al. (2016) also reported that pyrolysis at higher temperatures leads to greater cracking of organic components, thereby reducing biochar production. The ash content was higher in corn straw than in peanut shells, and biochar ash content was related to raw materials (Łapczyńska-Kordon et al. 2022).

Generally, biochar can absorb nitrate, ammonium salt, phosphorus, and other water-soluble saline ions, and has high CEC, therefore, biochar has fertiliser maintenance capability (Domingues et al. 2020). In our study, the CEC of corn straw biochar was higher than that of peanut shell biochar, in which the CEC of corn straw char reached 114.81 cmol /kg at 350 °C, but the CEC of peanut shell biochar was 77.86 cmol /kg (Table 3). In peanut shell biochar, CEC had no difference in temperature, but the CEC of corn straw biochar was lowest at 550 °C. The research from Yuan and Xu (2011) showed that CEC is related to the ratio of an oxygen atom to a carbon atom (O/C), the higher the O/C ratio, the higher CEC (Xu and Yuan 2011). The higher ratio of O/C is consistent with the carboxyl, hydroxyl, and carbonyl on the surface.

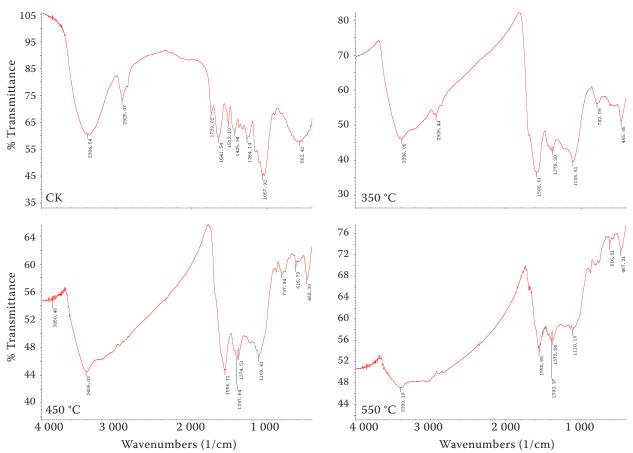


Figure 2. Fourier-transformed infrared (FTIR) spectra of corn straw biochar at temperatures of room 350, 450 and 550 °C. CK – control

The functional group might be oxidised, leading to the quantity of oxygen-containing functional group increases, therefore, with the increase of biochar in the soil, its CEC will also increase (Huff et al. 2018). Similarly, both pH and ash content reached their highest values in corn straw biochar, and reached their highest values at the highest temperatures. Many studies have also shown that the properties of straw biochar were superior to those of peanut shell biochar (Liu 2018, Li and Xue 2022, Su et al. 2022).

FTIR-PAS of the biochar. Adsorptive properties of biochar, the adsorptive property is influenced by the surface chemical property, especially the functional group species and quantities of the biochar surface. The FTIR spectra of corn straw and peanut shell under diverse temperature conditions were shown in Figures 2 and 3. The biochar surface contains an abundant oxygen-containing functional group of -COO-(-COOH), -O-(-OH), aromatics, alkanes, esters, and amine. The character peaks at 3 528/cm, 2 972/cm, 1 507/cm, 1 262/cm, represented the -OH, -CH, -C=C, -C-O; 817/cm and 719/cm are -C-H peak outside of the benzene derivative.

For corn straw biochar, the content of alcohol (including phenol) decreases with the increase of temperature, while the aromatic nucleus increases

(Figure 2). When the temperature exceeds 450 °C, alkanes disappear. Alcohol (including phenols) content of peanut shell biochar decreased when the temperature was increased, while alkanes were not detected at 550 °C due to their heat resistance (Figure 3). In addition, both corn straw and peanut shell biochar produced halides and sulphides after carbonisation. In general, the structure of biochar was stable, with the majority of its structure being aromatic ring skeletons, and its carbon composition mainly being aromatic nuclei. The biochar surface contained oxygen-containing functional groups such as carboxyl, hydroxyl, and carbonyl, which improved the adsorptive property and CEC. The oxygen-containing functional groups such as -COO-(-COOH), -O-(-OH) improved the capabilities of lyophilic, hydrophobicity, and acid-base buffer ability, which is the main reason that the surface usually presents with a negative charge. These properties decide the biochar effect on the soil.

The oxygen-containing functional groups. Boehm titration was used for quantitative analysis of phenolic hydroxyl, focusing, and carboxyl group content for acid functional group analysis (Table 4). The content of the alkaline functional group was higher than the acidity functional group. This may be because biochar was

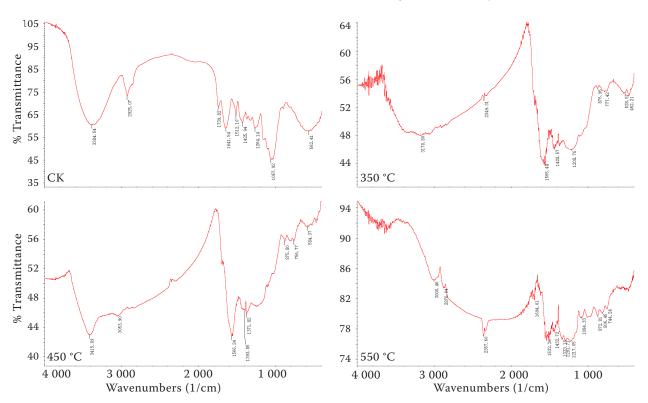


Figure 3. Fourier-transformed infrared (FTIR) spectra of peanut shell biochar at temperatures of room 350, 450 and 550 °C; CK – control

Table 4. The number of oxygen-containing functional groups of maise straw and peanut hull biochar at different pyrolysis temperatures (mmol/g)

_	Acid	functional group (mm	Alkaline functional group	
_	(AROH)	(RCOOCOR')	(RCOOH)	(mmol/g)
Corn straw biochar				
350 °C	0.5397^{aA}	0.2668^{aB}	0.2420^{B}	0.8120^{bA}
450 °C	0.0810^{bA}	0.1553^{bB}	_	1.0564^{aA}
550 °C	0.0703^{bA}	0.0779^{cB}	_	1.1578^{aA}
Peanut shell biochar				
350 °C	0.3363^{aB}	0.4376^{bA}	0.3464^{aA}	0.1517^{bB}
450 °C	0.0761^{bB}	0.6295^{aA}	$0.1445^{\rm b}$	0.5189^{aB}
550 °C	0.0074^{cB}	0.4587^{bA}	0.1124^{c}	0.5518^{aB}

Different uppercase letters indicate significant difference among different biochar within the same temperature, and different lowercase denotes significant difference between different temperature within the same biochar (P < 0.05)

alkaline in nature (Das and Ghosh 2022). At 350 °C, the alkaline functional group was 0.812 mmol/g, with the increase in temperature, the quantity of the alkaline functional group increased; however, the quantity of acidity decreased gradually, and the content of the carboxyl group could not be detected at 450 °C and 550 °C. A report on increasing the pyrolysis temperature to increase the content of alkaline functional groups in biochar (Ulusal et al. 2021). The acidity functional group excluding lactones decreased with temperature increase, and the content of the phenolic hydroxyl group reached a minimum of 0.0074 mmol/g at 550 °C. However, the content of lactone first increased and then decreased (Table 4). The results indicated that low temperatures may promote the formation of acid functional groups. Nevertheless, we do not know whether high temperature influences the formation of an alkaline functional group in this text because of the temperature range.

The microstructure and morphology of biochar. Before carbonisation, the surface structure of corn straw biochar was not obvious, with uneven and disordered edges. However, after carbonisation the main skeleton structure became obvious. At 550 °C, the pore structure became abundant and aligned orderly. Most of the biomass structure remained (Figures 4 and 5). The microscopic surface of the peanut shells changed significantly before and after carbonisation. Before carbonisation, the structure of peanut shell biochar was not obvious and the skeleton structure was not distinct. However, carbonisation made the peanut shell skeleton structures more distinct and prominent. The number of peanut shell biochar surface pores increased, but corn straw decreased,

at 550 °C, and the carbon structure was integral and distinct. This result was different with Krebsbach et al. (2023), they found that the number of corn straw biochar surface pores increased at high temperatures. This could be due to the different high pyrolysis temperature (1 000 °C).

Generally, after carbonisation, the structure was clearly visible, which remained connected to air and water and provided good environmental conditions (Meng et al. 2011). In the process of pyrolysis, microstructure generated gaps, and the unstable parts escaped, leading to the pore size of the raw material for biochar preparation being narrowed, and forming a small pore structure. The abundant pore structure is significant for biochar application, and biochar can adjust the water, fertiliser, air, and thermal environment in the soil (Arthur and Ahmed 2017, Xuan et al. 2022).

The carbon conversion efficiency of biochar. Many factors affect the carbonisation of biomass, mainly because of the raw material properties (Wang et al. 2020). On the other hand, the temperature also affects carbonisation, so the carbon conversion efficiency of biochar changes with the different temperatures (Liu et al. 2018). The carbon conversion efficiency was 42.12% at 350 °C, which was 1.6% higher than corn straw biochar; with a temperature rising, both the carbon conversion efficiency of corn straw and peanut shell raw material showed a declining trend (Table 5). At low temperatures, the cellulose and hemicelluloses began to decompose, but the lignin just decomposed under high temperatures, mainly because the lignin is an aromatic structure, higher content of carbon, thermostability of which is

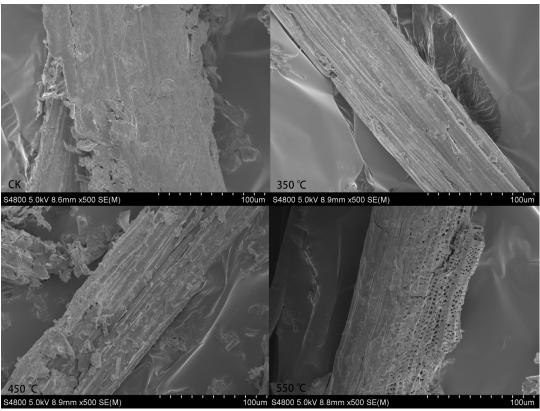


Figure 4. Scanning electron micrographs of corn straw biochar at temperatures of room 350, 450 and 550 $^{\circ}$ C. CK – control

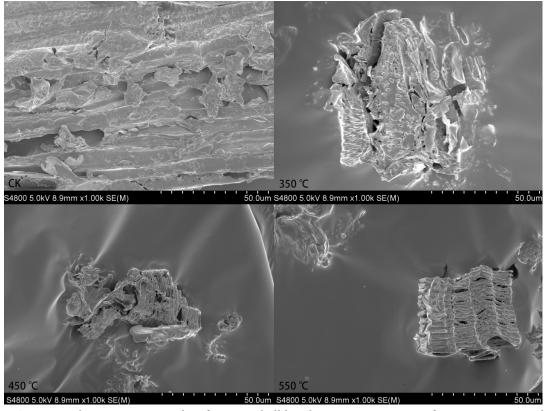


Figure 5. Scanning electron micrographs of peanut shell biochar at temperatures of room 350, 450 and 550 $^{\circ}$ C. CK – control

Table 5. The carbon conversion rate of corn straw and peanut shell at different pyrolysis temperatures (%)

	Corn straw biochar			Peanut shell biochar		
	350 °C	450 °C	550 °C	350 °C	450 °C	550 °C
Carbon conversion efficiency (%)	40.52 ^{aB}	34.36 ^{bB}	30.78 ^{bB}	42.12 ^{aA}	36.90 ^{bA}	33.27 ^{bA}

Different uppercase letters indicate significant difference among different biochar within the same temperature, and different lowercase denotes significant difference between different temperature within the same biochar (P < 0.05)

higher than cellulose and hemicelluloses (heterocyclic structure) (Liu et al. 2018, Jia et al. 2023). With the temperature rising, both corn straw and peanut shell biochar, and volatile substances escaped, the carbon content increased, and the elements such as hydrogen and oxygen declined, and so did the carbon conversion efficiency.

Chemistry of biochar materials varied with the biomass feedstock type and temperature variation. The carbon content in corn straw and peanut shell biochar increased with temperature. Corn straw biochar had stronger alkalinity than peanut shell biochar. At 550 °C, the ash content of corn straw biochar was higher than that of peanut shells. Microstructure scanning showed that the original skeleton structure was retained after carbonisation. The high CEC of biochar was due to the oxygen functional groups on the surface The functional groups of corn straw biochar increased with temperature, while the acidic functional groups decreased.

Acknowledgement. We thank Yajuan Duan (Polytechnic University of Catalonia, Computational Mechanics, Barcelona, Spain) for her valuable help.

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Received: December 21, 2022 Accepted: July 31, 2023 Published online: August 17, 2023