



# Characteristics and nutrient values of biochars produced from giant reed at different temperatures



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## HIGHLIGHTS

- ▶ Available N and P in biochar decreased with increasing temperature but K increased.
- ▶ Less-soluble crystalline P minerals were formed in high-temperature biochar.
- ▶ More  $\text{NH}_4^+$ ,  $\text{PO}_4^{3-}$  and  $\text{K}^+$  were released from the biochars at low pH ( $\leq 5$ ).
- ▶ Biochars released  $\text{NH}_4^+$  slowly but released  $\text{PO}_4^{3-}$  and  $\text{K}^+$  fast.
- ▶ Low-temperature biochars could be a good amendment for improving soil fertility.

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## ABSTRACT

To investigate the effect of pyrolysis temperature on properties and nutrient values, biochars were produced from giant reed (*Arundo donax* L.) at 300–600 °C and their properties such as elemental and mineral compositions, release of N, P and K, and adsorption of N and P were determined. With increasing temperatures, more N was lost and residual N was transformed into heterocyclic-N, whereas no P and K losses were observed. P was transformed to less soluble minerals, resulting in a reduction in available-P in high-temperature biochars. A pH of  $\leq 5$  favored release of  $\text{NH}_4^+$ ,  $\text{PO}_4^{3-}$  and  $\text{K}^+$  into water. Low-temperature biochars ( $\leq 400$  °C) showed appreciable  $\text{NH}_4^+$  adsorption (2102  $\text{mg kg}^{-1}$ ). These results indicate that low-temperatures may be optimal for producing biochar from giant reed to improve the nutrient availability.

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## 1. Introduction

Biochar refers to the carbon-rich product from heating biomass in a closed system under limited oxygen supply. It is distinguished from charcoal by its use as a soil amendment (Lehmann and Joseph, 2009). It is a multifunctional material with environmental and agricultural applications (Atkinson et al., 2010; Beesley et al., 2011). Biochar is recognized as a high-efficient and low-cost sorbent for pollutants (Silber et al., 2010; Sun et al., 2011; Wang and Xing, 2007). Application of biochar to soil has been proposed as an approach to sequester carbon (Lehmann and Joseph, 2009) and to possibly reduce or suppress  $\text{CO}_2$ ,  $\text{CH}_4$  and  $\text{N}_2\text{O}$  emissions (Spokas et al., 2009). Most importantly, biochar may improve soil

quality and nutrient availability to plants (Atkinson et al., 2010). Although information on biochar nutrient properties are available (Atkinson et al., 2010; Chan et al., 2009; Lehmann and Joseph, 2009; Silber et al., 2010), the mechanism of nutrient release from biochar is not fully understood. Furthermore, total N, P and K (TN, TP and TK) in biochars may not necessarily reflect the actual availability of these nutrients to plants (Spokas et al., 2012). The influence of pyrolysis temperature on the production of biochars that are suitable as soil fertilizer still needs to be elucidated. Although it has been shown that  $\text{NO}_3^-$  and  $\text{NH}_4^+$  leaching was reduced from soils amended with biochars (Novak et al., 2010), the influence of time and pH on nutrient release from biochars produced at different temperatures remains to be explored.

Giant reed (*Arundo donax* L.) (GR), a perennial grass, is widespread in many aquatic ecosystems in China (Yan et al., 2005). Due to its fast growth rate and good resistance to drought and floods, GR may yield up to 45 tons per hectare and life cycle

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assessments indicate that it could be a suitable feedstock for biochar production (Roberts et al., 2010).

Biochars were prepared from GR at different temperatures using oxygen-limited pyrolysis. The aims of the present study were to (1) investigate the influence of pyrolysis temperature on nutrient composition of biochars and the mechanisms of nutrient release from biochars; (2) evaluate the influence of time and pH on the release of plant-available nutrients from biochars alone; and (3) examine the sorption behavior of  $\text{NH}_4^+$ ,  $\text{NO}_3^-$  and  $\text{PO}_4^{3-}$  onto biochars.

## 2. Methods

### 2.1. Production of biochars

Two batches of biochar were produced at 300, 350, 400, 500 or 600 °C for 2 h from GR stems (1.0 kg) without leaves using a vacuum tube furnace (O-KTF1200, China) under a  $\text{N}_2$  flow of 500  $\text{mL min}^{-1}$ . The pyrolysis temperature was raised to the desired values at a ramp rate of 10 °C  $\text{min}^{-1}$ . Biochar yields were recorded and the samples were milled to pass a 0.12 mm (120 mesh), and the biochars produced at the same temperature were mixed and homogenized for further analysis. The biochars are hereafter referred to as BC300, BC350, BC400, BC500 and BC600, respectively. BC0 is the raw material. Ash (BCA) was produced by placing the raw material into a ceramic crucible and heated at 750 °C for 4 h under air.

### 2.2. Characterization of samples

Total C, N, H, and O were determined in duplicate with an elemental analyzer (MicroCube, Elementar, Germany). Brunauer–Emmett–Teller (BET) surface areas ( $S_{\text{BET}}$ ) were obtained from  $\text{N}_2$  adsorption at 77 K using Quantachrome Autosorb-1 (Autosorb-1, Quantachrome, USA). Ash contents of biochars were measured by the production of BCA. The pH values of all samples were measured at a ratio of 1:20 (w/v) in water after being shaken for 24 h at 130 rpm. X-ray diffraction (XRD) patterns were obtained using a Macscience-M18XHF instrument (UK) with Cu K $\alpha$  radiation at 40 mA and 40 kV. Scanning electron microscopy (SEM) imaging analysis was conducted using a HITACHI S-4800 Scanning Microscope. Zeta potentials were measured by a zeta potential analyzer (90Plus, Brookhaven, USA) (Yuan et al., 2011). The total acidic oxygen-containing groups of the biochar samples were determined using Boehm's titration method (Boehm, 1994).

### 2.3. Nutrient content in biochars

Water-soluble mineral elements released from biochars were determined in a background electrolyte solution with 0.01 M NaCl (Silber et al., 2010) to maintain a constant ionic strength and 200  $\text{mg L}^{-1}$   $\text{NaN}_3$  to inhibit microbial activity. A biochar sample (1.0 g) was placed in a 40-mL glass vial and 36 mL of background electrolyte solution was added. After being shaken at 130 rpm for 72 h at room temperature ( $22 \pm 1$  °C), the suspension was centrifuged at 1742 g for 30 min.  $\text{PO}_4^{3-}$ ,  $\text{NH}_4^+$  and  $\text{NO}_3^-$  in the supernatant were determined using a Flow Injector Autoanalyzer (QuickChem 8500, Lachat, USA) and  $\text{K}^+$  was measured with an ion chromatograph (ICS-3000, Dionex, USA). Acid-soluble and KCl-extractable  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ , and  $\text{PO}_4^{3-}$  were determined in 0.5 M HCl and 1 M KCl solutions, respectively, and acid-soluble  $\text{K}^+$  was also measured using the ion chromatograph. TP in biochars was measured after dry-ashing using a colorimetric method (Page et al., 1982). For determination of TK, biochar was digested in concentrated acid (0.15 g samples in 10 mL 70%  $\text{HNO}_3$  at 120 °C for 24 h) and K concentration was quantified using ICP (Silber et al., 2010).

### 2.4. Effect of time and pH on water-soluble nutrients release

A biochar sample (1.0 g) was placed in a 40-mL glass vial and 36 mL of background electrolyte solution (see Section 2.3) was added. After being shaken at 130 rpm, 30-mL supernatants were removed using a glass pipette without any biochar loss and the vials were refilled with the same amount of background solution and re-extracted at 2, 6, 12, 24, 48, 72, 96, 120, 144 and 168 h. Each sample was run in triplicate. The concentration of  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$  and  $\text{K}^+$  were determined as described in Section 2.3. Triplicate samples with no biochars were concurrently run as controls.

The effect of pH on water-soluble  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$  and  $\text{K}^+$  released from biochars was studied in background electrolyte solution adjusted to pHs from 2 to 12. The suspension pH was adjusted to the predetermined values and kept constant throughout the experiment by adding small volumes of 0.1–1.0 M HCl or NaOH. After being shaken at 130 rpm for 72 h, the concentrations of  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$  and  $\text{K}^+$  in supernatants were determined as described in Section 2.3. Each sample was run in duplicate. Duplicate samples with no biochars were concurrently run as controls.

### 2.5. $\text{NH}_4^+$ and $\text{NO}_3^-$ sorption

Sorption isotherms were obtained using a batch equilibration technique. Appropriate amounts of samples were placed into vials so that the solid/solution ratio would result in 20–80% uptake of  $\text{NH}_4^+$  and  $\text{NO}_3^-$ . The solid/solution ratios for sorption of  $\text{NH}_4^+$  by BC300 and BC350 were 0.35 g/12 mL and 0.8 g/12 mL for BC400, BC500 and BC600. A ratio of 0.3 g/8 mL was used for sorption of  $\text{NO}_3^-$  by all biochars. The test solutions of 0–200  $\text{mg L}^{-1}$   $\text{NH}_4^+\text{-N}$  and 0–100  $\text{mg L}^{-1}$   $\text{NO}_3^-\text{-N}$  were prepared from stock solutions of 1000  $\text{mg L}^{-1}$   $\text{NH}_4^+\text{-N}$  prepared from  $\text{NH}_4\text{Cl}$  and 1000  $\text{mg L}^{-1}$   $\text{NO}_3^-\text{-N}$  prepared from  $\text{NaNO}_3$  in background solutions containing 0.01 M KCl and 200  $\text{mg NaN}_3$ . During the shaking period of 72 h, the pH of suspension was maintained at  $7.0 \pm 0.1$  by adding small volumes of 0.1–1.0 M HCl or NaOH. The samples were centrifuged at 1742 g for 30 min and the concentrations of  $\text{NH}_4^+$  and  $\text{NO}_3^-$  in supernatants were determined as described in Section 2.3. All samples were run in duplicate along with blanks. Because the mass loss of solute was negligible, sorption of solutes by biochars was determined by mass balance.

### 2.6. Data analysis

The nutrient release results were examined for  $\text{NH}_4^+$ ,  $\text{PO}_4^{3-}$  and  $\text{K}^+$  solubilized from biochars using pseudo-first-order (Kasozi et al., 2010), pseudo-second order (Kasozi et al., 2010) and power models (Sparks, 2003), which can be presented as

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (1)$$

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad (2)$$

$$q_t = at^b \quad (3)$$

respectively, where  $q_e$  and  $q_t$  ( $\text{mg kg}^{-1}$ ) are the release capacities at equilibrium and at time  $t$ , respectively;  $k_1$  ( $\text{h}^{-1}$ ) is the rate constant of pseudo-first order release,  $k_2$  ( $\text{h}^{-1}$ ) is the rate constant of pseudo-second order release,  $a$  ( $(\text{mg kg}^{-1} \text{h}^{-1})^b$ ) and  $b$  ( $(\text{mg kg}^{-1})^{-1}$ ) are constants that relate to the initial concentration of nutrients released and the rate of change in nutrients release over time, respectively.

With respect to sorption, nonlinear Freundlich and Langmuir models were used to fit the  $\text{NH}_4^+$  and  $\text{NO}_3^-$  sorption isotherm data:

$$Q_e = K_F C_e^n \quad (4)$$

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