



Fundamental and molecular composition characteristics of biochars produced from sugarcane and rice crop residues and by-products



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HIGHLIGHTS

- Pyrolysis temperature at 550 °C yields stable biochar characteristics and maximum CEC.
- Lignin decarboxylation and demethoxylation at 450 °C and dehydroxylation at above 550 °C.
- Biochars are dominated with 20-nm pores and high temperatures increased both smaller and larger than 50 nm pores.
- Lower WHC of rice husk biochar dominated with higher hydrophobic composition of BTX and PAH than other biochars.

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ABSTRACT

Biochar conversion of sugarcane and rice harvest residues provides an alternative for managing these crop residues that are traditionally burned in open field. Sugarcane leaves, bagasse, rice straw and husk were converted to biochar at four pyrolysis temperatures (PTs) of 450 °C, 550 °C, 650 °C, and 750 °C and evaluated for various elemental, molecular and surface properties. The carbon content of biochars was highest for those produced at 650–750 °C. Biochars produced at 550 °C showed the characteristics of biochar that are commonly interpreted as being stable in soil, with low H/C and O/C ratios and pyrolysis fingerprints dominated by aromatic and polyaromatic hydrocarbons. At 550 °C, all biochars also exhibited maximum CEC values with sugarcane leaves biochar (SLB) > sugarcane bagasse biochar (SBB) > rice straw biochar (RSB) > rice husk biochar (RHB). The pore size distribution of biochars was dominated by pores of 20 nm and high PT increased both smaller and larger than 50 nm pores. Water holding capacity of biochars increased with PT but the magnitude of the increase was limited by feedstock types, likely related to the hydrophobicity of biochars as evident by molecular composition, besides pore volume properties of biochars. Py-GC/MS analysis revealed a clear destruction of lignin with decarboxylation and demethoxylation at 450 °C and dehydroxylation at above 550 °C. Overall, biochar molecular compositions became similar as PT increased, and the biochars produced at 550 °C demonstrated characteristics that have potential benefit as soil amendment for improving both C sequestration and nutrient dynamics.

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1. Introduction

Biochar, produced from biomass pyrolysis under the partial or total absence of oxygen, has been recently proposed as a soil amendment to sequester carbon (C) in soils through a C negative process, as well as reduce greenhouse gas emissions and improve soil quality (Lehmann, 2007; Spokas et al., 2009). Biochar has been shown to enhance nutrient retention due to its high cation exchange capacity (CEC) and high surface area (Liang et al.,

2006). It can also act as a slow release fertilizer by permeating with other nutrients, and therefore increases fertilizer use efficiency (Chan and Xu, 2009). Furthermore, biochar has been shown to reduce the mobility of contaminants (Yu et al., 2009). For instance, addition of wheat biochar at a rate of 1% (w/w) to soils increased the sorption of diuron by 80–86% compared to non-amended soils (Sheng et al., 2005).

Biochar amendment to soil could create a significant long-term sink for atmospheric CO₂ in terrestrial ecosystems (Spokas et al., 2009). Research on the effect of biochar addition on CO₂ emission showed that only 2.8% of the added biochar C was decomposed, whereas 56% of the added wheat straw C was decomposed during 84 days of experiment (Zavalloni et al., 2011). Research on rice paddy soil also showed that N₂O and CH₄ were decreased by 43%

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and 46%, respectively, under biochar amendment over two cycles of cultivation as compared to the control (Zhang et al., 2012). Besides C stability, the main reasons for greenhouse gas reduction by biochar addition could be increased adsorption of dissolved organic C on biochar surfaces (Thies and Rilling, 2009) and enhanced soil aggregation, which protects labile soil organic matter inside the aggregates from decomposition (Karhu et al., 2011). In addition, increased soil aeration due to increased porosity with biochar addition likely reduces CH₄ and N₂O productions (Van Zwieten et al., 2009).

While biochar is useful for agronomic and remediation purposes (Liang et al., 2006; Yu et al., 2009), its efficiency is influenced by their physicochemical properties, which varies with the source materials and pyrolysis conditions (Chan and Xu, 2009; Cantrell et al., 2012). Studies of biochars derived from various feedstocks such as animal manure (Cantrell et al., 2012), woodchip (Spokas et al., 2009), wheat straw (Zavalloni et al., 2011), rice husk (Maiti et al., 2006), sludge (Lu et al., 2012), and algal biomass (Bird et al., 2011) showed that the C contents increased upon pyrolysis as compared to source materials and varied with source material and pyrolysis temperature (PT). Pyrolysis temperature also influences pH, surface area, and other properties of biochars made from various feedstocks (Hossain et al., 2011; Pereira et al., 2011; Xu et al., 2011; Yuan et al., 2011; Cantrell et al., 2012; Song and Guo, 2012). In general, biochar produced from slow pyrolysis was found to have a considerably higher C (69.6%) content than that by the fast pyrolysis (49.3%) (Bruun et al., 2011).

The conversion of plant-derived biomass as a source material to biochar supports an alternative way of managing crop residues, which have, in most cases, been directly incorporated in soils or subjected to open field burning. In conventional sugarcane production, especially in sub-tropical region, harvest residues (leaves/trash) are generally burned in the field to avoid yield reduction of ratoon crops (Udeigwe et al., 2010). Approximately 70–90% of the dry matter in the sugarcane harvest trash is lost to the atmosphere through burning (Thorburn et al., 2004). Alternatively, these residues could be converted to biochar and returned to the fields for improving soil quality and enhancing C sequestration. Similar to sugarcane production, residue burning practice in rice cultivation is also common in many countries, although it is limited in the U.S. In addition, large quantities of surplus bagasse after sugar extraction are usually stockpiled, landfilled, and/or burnt in open field, creating a growing environmental problem (Xu et al., 2011). Milling by-products such as rice husk/hulls are also available (Zhang et al., 2012). All these harvest residues and milling by-products could be potentially converted to value-added biochar.

While there is increasing attention to biochar studies, the information on the biochars produced from sugarcane residue especially commonly burned out harvesting leaves/trash is lacking. Previous biochar production and property characterization on sugarcane related feedstocks focused primarily on bagasse, the milling waste (Tsai et al., 2006; Inyang et al., 2010; Kameyama et al., 2012; Novak et al., 2013). On the other hand, various studies investigated hydrolyzed products of rice residue and associated by-products for energy and agronomic applications (Maiti et al., 2006; Tsai et al., 2006; Gadde et al., 2007). However, most of the previous studies were limited in elucidating individual molecular composition changes of these feedstock-derived biochars as a function of pyrolysis conditions, which could have significant environmental implication. The biochar molecular composition characteristics associated with the corresponding precursor, such as cellulose, lignin, protein and charred matter provide the information for the stability of biochar as well as enabling the elucidation of possible relationships between the occurrence of partially preserved original biomass surviving the carbonization process and the microbial

mineralization potential (Fabbri et al., 2012). Therefore, the objectives of this study were to evaluate the molecular composition characteristics of biochars produced from sugarcane harvesting leaves/trash (SLB), sugarcane bagasse (SBB), rice straw (RSB), and rice husk (RHB) at different pyrolysis temperatures (PTs) based on individual molecular “fingerprint” using pyrolysis-gas chromatography (Py-GC/MS). In addition, associated elemental and other fundamental physical and chemical properties of these biochars were characterized and compared.

2. Materials and methods

2.1. Residue and biochar production

Sugarcane leaves/residues and rice straw were collected from fields after harvest and bagasse and rice husk, as by-products, were collected after milling process. Raw materials were directly used for charring process. The materials were tightly packed in several stainless steel canisters using a wooden mallet. After flushing packed materials with N₂ gas, each canister was closed with another slightly larger stainless steel canister (to minimize the oxygen content) before placing in the muffle furnace. Biochars were produced using a bench-top slow pyrolysis unit that consisted of an electrical muffle furnace and a pyrolysis canister. The muffle furnace ran a constant temperature environment during biochar production with an average temperature ramp of 20 °C min⁻¹ for targeted temperature (Thermo Fisher Scientific Inc., Suwanee, GA). The canister was made of stainless steel and designed for limited oxygen contamination during the pyrolysis. The biochars were produced at 450 °C, 550 °C, 650 °C, and 750 °C for 2 h.

2.2. Basic characterization

Elemental analysis of biochars and raw materials for C, N, H, and S was performed by dry combustion, followed by gas chromatography using a Perkin Elmer 2400 Series II CHNS Elemental Analyzer (Waltham, MA). Other elements were determined by wet acid digestion (conc. HNO₃ + 30% H₂O₂) followed by analysis with induced coupled plasma atomic emission spectroscopy (ICP-AES) (Spectro CIROS^{CCD}, Mahwah, NJ). Ash contents of biochars and raw materials were determined by overnight combustion at 750 °C in a muffle furnace, and cation exchange capacity (CEC) of biochars was measured based on the ammonium-acetate (1 M neutral NH₄OAc) extraction method (Yuan et al., 2011). The pH of biochar samples was measured using pH meter at a 1:5 solid/water ratio. Specific surface area was determined based on multi-point adsorption of N₂ (Quantachrome, Boynton Beach, FL) and calculated from the BET (Brunauer–Emmett–Teller) equation. Pore size distribution (PSD) was determined based on N₂ adsorption isotherms at 77.35 K (temperature of liquid nitrogen) using BJH (Barrett–Joyner–Halenda) equation (Jagiello and Thommes, 2004) and the non-local density functional model (Neimark et al., 2009). The pore size analysis was performed by Y-Carbon, Inc. (Bristol, PA). Water holding capacity (WHC) was determined based on pre-saturation of biochar with de-ionized water for 24 h followed by removing gravitational water from biochar samples in a Kontes flex glass column at 0.1 bar vacuum (Song and Guo, 2012). Linear regression analysis was conducted between surface area and WHC of all biochars. Water extractable organic C (WEOC) in biochars was estimated by extracting approximately 0.2 g of biochar with 30 mL ultrapure deionized water followed by shaking for 3 days on a horizontal shaker. The extracts were filtered through 0.2 μm glass filter and analyzed for total organic C

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