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Pyrolysis methods impact biosolids-derived biochar composition, maize growth and nutrition

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ABSTRACT

Land-applied biosolids (sludge) can improve food production sustainability through nutrient recycling. Biosolids-derived biochar may enhance soil fertility and overall soil health. However, there is little information on the conversion of biosolids to biochar using traditional kilns, or effects on biochar characteristics and plant growth. Biochar was produced from biosolids using two pyrolysis methods: 1) a traditional retort kiln (Top-lid Updraft-TLUD) intended for use by small farmers and gardeners, and 2) a laboratory muffle furnace, with the aim of evaluating biochar characteristics and its effects on *Zea mays* L. (corn) seed germination, growth and nutrition. Biochar produced in a muffle furnace contained 70% more ash, 78% more fixed carbon, and 63% less volatile matter than biochar produced by TLUD, which raised concern regarding TLUD-derived biochar toxicity The TLUD-derived biochar inhibited corn seed germination in a petri dish bioassay at biochar application rates from 2.5 to 100 Mg ha^{-1}. However, germination increased from 29% (control) to approximately 60%, at 60 Mg ha^{-1} or greater rates, with muffle furnace biochar. A greenhouse experiment was conducted to evaluate the growth and nutrition of corn grown in soil treated with 0, 5, 10, 20 and 60 Mg ha^{-1} biochar pre-incubated for two weeks in moistened soil. The muffle furnace biochar had no negative effect on plant growth and N nutrition, whereas the TLUD biochar at a 60 Mg ha^{-1}rate, reduced plant growth and increased plant N concentrations four-fold, compared to the control. Both biochars increased plant P concentrations with increasing application rates. Biosolids biochar produced via TLUD at rates below 20 Mg ha^{-1} may benefit crop production, although an incubation or weathering period may be necessary to limit potential short-term, phytotoxic effects. Future research needs include optimizing TLUD operational parameters and identifying weathering processes that improve biochar product quality for agronomic use.

1. Introduction

Biosolids, formerly known as sewage sludge or wastewater treatment residuals, is a major source of plant nutrients, especially nitrogen (N) and phosphorus (P). Land-applied, carbon-rich biosolids improve soil health (Singh and Agrawal, 2008; Usman et al., 2012). Municipal biosolids have undergone treatments, such as alkaline stabilization and thermal drying, to create a product safe for land application, a cost-effective method of waste disposal (Lu et al., 2012). Even so, fertilizer-grade biosolids (Class A and AA) must minimize human pathogens and inorganic contaminants that the US federal government regulates (EPA, 1999). Organic contaminants, such as pharmaceuticals and health care products may also be found in biosolids, but understanding and regulation of these materials are in their infancy. Furthermore, land-applied biosolids contribute to greenhouse gas emissions (Brown et al., 2010).

Thermally treating biosolids, via pyrolysis, reduces waste volume and mass, therefore, transport costs (Inguanzo et al., 2002). Manure-derived biochar further reduced pathogens and heavy metal bioavailability in soils (Cantrell et al., 2007). Additionally, soil-applied biochars often contribute to C sequestration, due to their inherent stability (Lehmann, 2007).

The chemical and physical characteristics of different biochars, in general, depend on the operating conditions of the pyrolysis unit (Mendez et al., 2013). Depending upon the pyrolysis operational

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conditions, biochar varies considerably in its elemental composition (C, N, H, S and O), ash content, pH, porosity, etc. (Enders et al., 2012).

Biochar effects on crop growth have been extensively reported (Gaskin et al., 2010; Major et al., 2010; Van Zwieten et al., 2010), but much less information is available about biochars derived from biosolids. Hossain et al. (2010) applied biosolids biochar at 10 Mg ha^{-1} to cherry tomatoes and observed a 64% increase in production. The authors attributed their results to increased N and P fertility. They also observed that the biochar mitigated some of the inherent soil acidity. Liu et al. (2014) tested biosolids biochar on Chinese cabbage and reported a significant increase in plant growth. Others have reported that biochars from different feedstocks will promote soil N immobilization and therefore alter N bioavailability (Lehmann et al., 2003; Steiner et al., 2008; Laird et al., 2010). Biochar applications also have been reported to enhance P bioavailability and consequently, plant growth (Xu et al., 2014), but according to Sandeep et al. (2013), the selected soil type may alter biochar's impact.

Despite the potential agricultural advantages and environmental benefits of biochar, its large-scale production under controlled conditions remains a constraint. Many small farmers, especially in developing and undeveloped countries, use conventional ovens and small retort kilns to produce biochar. In addition, it is unclear how well these systems and their products compare to products from more controlled conditions. Furthermore, the effect of biosolids biochar on plant growth and nutrient uptake has seldom been reported. Therefore, the aim of this study was to: 1) characterize and test biosolids biochar produced by two different pyrolysis units (TLUD retort kiln and muffle furnace); 2) evaluate the effect of different rates of the two biochars on corn seed germination using a soilless petri dish bioassay, and 3) evaluate corn growth, N and P nutrition in soil amended with different rates of the two biochars.

2. Materials and methods

2.1. Biochar production and characterization

Biosolids were collected from a tile-lined, drying bed, at a municipal wastewater treatment facility (WWTF), located in Tallahassee, Florida, U.S.A. The biosolids were the end-product of an activated sludge treatment process. Biosolids had the following composition (mean of three replicates ± std): 91 ± 2% moisture, 6.8 ± 0.3 pH units, and total elements (dry mass basis): 57 ± 9 g kg^{-1} N, 13 ± 5 g kg^{-1} P, 2 ± 0.1 g kg^{-1} K, 94.0 ± 21 mg kg^{-1}Cu, 88 ± 21 mg kg^{-1} Zn, 18 ± 3 mg kg^{-1} Mo, 4.0 ± 0.8 mg kg^{-1} As, 0.80 ± 0.26 mg kg^{-1} Cd, 20 ± 9 mg kg^{-1} Pb, and 3.6 ± 0.6 mg kg^{-1} Ni.

Biochars were produced using two types of slow pyrolysis units. The first unit was a Top-Lit Updraft retort unit (TLUD), which is a micro-kiln that uses a reburner to eliminate volatile byproducts of pyrolization (Nsamba et al., 2015). Both, the vapors, as well as the non-condensable gases, are combusted, to provide heat for driving the pyrolysis reaction. The sewage sludge was dried in an oven at 45 °C for 5 days and subsequently 20 kg of the feedstock was pyrolyzed over 3 h, at approximately 550–700 °C, which was measured using a thermal gun aimed at the center of the unit during operation. After cooling, biochar was weighed, ground with a mortar and pestal, sieved to pass through a 2 mm screen, and stored in airtight plastic bags. The second pyrolysis unit was a bench-scale, muffle furnace. The feedstock was oven-dried at 45 °C, ground with a mortar and pestal and sieved to pass through a 2 mm screen. Approximately 32 g of the dried biosolids were placed into ceramic crucibles with loose-fitting ceramic lids and pyrolyzed at 600 °C for 1 h. Subsequently, the oven was turned off and the material was allowed to cool (overnight) before collecting the biochar, in order to avoid auto-ignition when the lids were removed. The biochar was weighed and stored in sealed plastic bags.

Biochar yield was determined according to Gaskin et al. (2008) as the mass ratio of biochar product to oven-dried biosolids feedstock (Eq. (1)):

$$\text{BCyield}(\%) = \frac{W_2}{W_1} X100 \quad (1)$$

Where W1 is biosolids dry mass prior to pyrolysis and W2 is the biochar product dry mass.

Biochar samples were ground in a ball mill to pass a 300 μm sieve and sent to a commercial laboratory (Huffman Labs, Boulder, CO, USA) for proximate analysis (ash content, volatile matter and fixed carbon). The determination of the volatile matter and ash content was conducted according to the American Society for Testing and Materials (ASTM) D1752-84, which is recommended by the International Biochar Initiative. The volatile matter was thus determined by measuring the weight loss that followed combustion of about 1 g of biochar in a crucible at 950 °C. Following the same procedure, the ash content was determined at 750 °C. The laboratory conducted ultimate analysis (elemental C, N, H and S) using a CNHS elemental analyzer, via flush combustion at 1020 °C and oxygen was determined by difference (Mukherjee et al., 2014). Sample caloric value (HHV) was measured by the ASTM bomb calorimeter method, according to ASTM5865.

Biochar pH was determined in a 1:5 (w/w) biochar:water ratio after 1.5 h shaking in a reciprocating shaker and one hour equilibration period (Gaskin et al., 2008). Electric conductivity (EC) was determined in the same extract.

2.2. Soilless germination bioassay

Fifteen corn (*Zea mays*) seeds were sown in petri dishes (8.5 cm diameter) on a layer of 41 mm filter paper moistened with 20 mL deionized water and containing biochar rates of 0, 2.5, 5, 10, 20, 60 and 100 Mg ha^{-1} on a volume basis, with three replications, according to the procedure described by Morrison and Morris (2000). All petri dishes were covered with lids and incubated in the dark at 25 °C for 72 h. The number of germinated seeds was counted and germination percent determined. Root and cotyledon lengths were measured and reported as the sum from each dish (cm per dish). Roots and cotyledons were dried at 60 °C for 48 h and weighed to determine dry mass.

2.3. Greenhouse experiment

The soil used in this experiment was taken from a fallow field at North Florida Research and Education Center (NFREC), Quincy, Florida, from a depth of 0–20 cm (A horizon), air-dried and sieved to pass through a 2 mm screen. The soil was classified as Loamy, kaolinitic, thermic Grossarenic Kandiudults (Soil Survey Staff, 2007), with 90% sand, 6% silt and 4% clay, pH (ratio of 1:5 w/v) of 5.8, 0.72% organic matter, 3.70 Cmolc kg^{-1} CEC, 149 mg kg^{-1} P, 65 mg kg^{-1} K, 345 mg kg^{-1} Ca, and 56 mg kg^{-1} Mg. Cation exchange capacity was determined by the ammonium acetate method (Thomas, 1982); soil organic matter by the Walkley Black method (Nelson and Sommers, 1982); and soil texture by the pippete method (Day, 1965). Concentrations of extractable P, K, Ca and Mg were determined by the Mehlich 3 method (Mehlich, 1984).

The experiment was conducted as 2 × 5 factorial and completely randomized design, with two types of biosolids biochar (TLUD or muffle furnace), five biochar application rates (0, 5, 10, 20 and 60 t ha^{-1}), and four replications. For each observation, 2.0 kg of air-dried and sieved (2 mm) soil was put into a plastic bag and thoroughly mixed with the appropriate rate of biochar and then