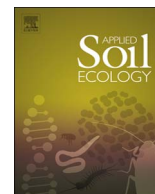




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Varying pyrolysis temperature impacts application effects of biochar on soil labile organic carbon and humic fractions

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ABSTRACT

Limited information is available to understand the effect of biochar (BC) on soil labile organic carbon (C) and humic fractions. A laboratory incubation experiment was conducted with Lou soil (Earth-cumuli-Orthic Anthrosol) to study the influence of BCs produced at 300, 400, 500 and 600 °C (BC300, BC400, BC500 and BC600) on soil labile organic C and humic fractions, respectively. BC products were added at the rate of 2% and incubated at 25 °C over 360 days. Non-amended soil was used as the control (CK). Compared with CK, BCs amendment significantly increased the contents of soil organic C and humin by 167.51%–255.52% and 288.42%–618.11%, respectively. Microbial biomass C (MBC), dissolved organic carbon (DOC), humic acid (HA) and fulvic acid (FA) were lower in BC500 and BC600 amendment after the initial stage of incubation, while MBC, DOC and HA were higher in BC300 and BC400 amendment during the whole incubation in comparison with CK. The color tone coefficients of HA and FA increased by 6.74%, 30.87% in BC300 amendment and by 2.57%, 9.27% in BC400 amendment, while decreased by 0.32%, 5.98% in BC500 amendment and by 3.19%, 12.19% in BC600 amendment at the end of incubation as compared with CK, respectively. Compared with CK, BC300 and BC400 amendment significantly increased the ratios of HA/FA, while BC500 and BC600 amendment showed no significant difference in comparison with CK. Thus, the properties of BC should be individually characterized and the application of BC should be carefully monitored due to the positive and negative effect on soil labile organic C and humic fractions after BCs amendment.

1. Introduction

Soil organic carbon (SOC) is one of the main global carbon (C) pools, storing three times more C than living organisms or the atmosphere (Ji et al., 2016). Strategies to increase SOC sink draw much attention because of the potential to mitigate global climate change (Yin et al., 2014). Biochar (BC) is a solid material obtained from the pyrolysis of plant biomass under low or zero oxygen conditions (Blanco-Moure et al., 2016). Recently, BC is being used for sequestering atmospheric CO₂ due to its contribution to the refractory SOC pool (Keith et al., 2016). In general, higher temperature resulted in a higher C content at a certain temperature range (< 650–750 °C) (Jeong et al., 2015), while lower contents of nitrogen (N), hydrogen (H) and oxygen (O) were also observed (Liang et al., 2016). In addition, the increasing temperature results in higher contents of ash and fixed C, while a loss of volatile materials was also recorded (Tag et al., 2016). Furthermore, pyrolysis temperature can also alter porosity and surface area of BCs

(Suliman et al., 2016). The characteristics of BC are likely to determine its effect on soil properties when used as a soil amendment (Butnan et al., 2015). Therefore, it is important to understand the physico-chemical properties and structure of BC before its application in soil, and the influence of application of BC pyrolyzed at varying temperatures on the fractions and molecular condensation of SOC still remains further concerns.

The addition of BC to soil can increase SOC content (Agegnehu et al., 2016). Due to its predominantly aromatic structure, BC is widely recognized as a relatively stable form of C (Purakayastha et al., 2015). However, a part of BC may be mineralized, especially for BC produced at lower temperatures (Mukome et al., 2013). BC produced at lower temperatures may have aliphatic fractions that are microbial available (Smith et al., 2010) and, when applied to soil, typically undergoes a period of rapid mineralization during the first few days to weeks (Novak et al., 2010). Moreover, either labile or leachable organic C also generate during pyrolysis (Lin et al., 2012), and are incorporated into

Abbreviations: BC, biochar; SOC, soil organic carbon; C, carbon; N, nitrogen; H, hydrogen; O, oxygen; DOC, dissolved organic carbon; MBC, microbial biomass carbon; HS, humic substances; HA, humic acid; FA, fulvic acid; Hu, humin; LHS, humic-like substances; LHA, humic-like acid; LFA, fulvic-like acid; $\Delta \log K$, color tone coefficient; SPAC, stable polycyclic aromatic carbon

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BC-amended soils due to the adsorption of BC. These low molecular weight organic compounds directly increase the pool of soil labile organic C in the soil. Some previous studies indicated that BC amendment increased the content of dissolved organic C (DOC) and microbial biomass C (MBC) compared with un-amended soil (Liao et al., 2016). However, different views exist (Prommer et al., 2014). These contradicting positive and negative effects on soil labile organic C may be attributed to the different physicochemical properties and structure of BC, as well as the soil characteristics.

Humic substances (HS), important fractions of soil organic matters, are usually fractioned into humic acid (HA), fulvic acid (FA) and humin (Hu) depending on their solubility at different pH ranges (Vergnoux et al., 2011). Previous studies showed that application of BC played an important role in the formation of HS (Brodowski et al., 2005). Humic-like acid (LHA) extracted from laboratory-oxidized BC showed remarkable similarities to highly aromatic soil HA in their spectroscopic properties and chemical composition (Haumaier and Zech, 1995). Therefore, BC is considered to be a possible source of the chemically stable, aromatic soil C pool (Cheng et al., 2006). BC in soils may promote the decomposition of labile C compounds rather than stabilize them against degradation (Hamer et al., 2004), the decomposition of labile C compounds may transform into soil HA by special microbial species in soil (Kwapinski et al., 2010). A recent study indicated that humic-like substances (LHS) also generate during pyrolysis (Uchimiya et al., 2013) and the increase in pyrolysis temperature from 450 to 550 °C decreased the relative proportion (with respect to the total DOC of BC) of “humic” fraction (Lin et al., 2012). It has been reported that the humification process during composting could be improved by the addition of BC (Jindo et al., 2016; Zhang et al., 2014). However, the information about the effect of BC addition on the composition and structure of HS in soil is still limited.

In general, different temperatures ranging from 200 to 600 °C were used for BC production. Therefore, four temperatures (300, 400, 500 and 600 °C) were employed to prepare BCs in this study. BC products were added to soil at the rate of 2% (44 Mg ha⁻¹), which was often used for crop productivity (Jeffery et al., 2011). A incubation study was conducted at 25 °C in lab over 360 days with the aim to investigate the effect of BC produced at different temperatures on the contents of SOC, soil labile organic C and humic fractions based on the following hypotheses (1) BCs amendment will enhance the contents of SOC and humin, (2) Lower-temperature derived BCs (≤ 400 °C) amendment will significantly increase the contents of soil labile organic carbons (MBC and DOC), HA and FA, while higher temperatures derived BCs (> 400 °C) have the reverse effect, and (3) Higher temperatures derived BCs (> 400 °C) will lead to the complicated structure of soil HS, while opposite trend may be observed in the treatments amended with lower-temperature BCs (≤ 400 °C).

2. Materials and methods

2.1. BC and soils

Apple tree branches were collected from apple orchards located in Guanzhong Plain, Northwest China (34°35'N, 108°24'E). The apple tree branches were washed with deionized water and then they were dried at 80 °C for 24 h to remove moisture. The dried apple tree branches were chopped to pass through a sieve with a mesh size of 2 mm. Such pre-prepared samples (about 125 g–150 g) were placed into a stainless steel box (Length 20 cm, width 12 cm and height 5.5 cm) and then the four stainless steel boxes were placed into a muffle furnace (Yamato FO410C, Japan). The samples were pyrolyzed at 300 °C, 400 °C, 500 °C and 600 °C. The pyrolysis heating rate was 10 °C min⁻¹ and nitrogen gas was injected at a rate 630 cm³ min⁻¹ (at stp 298 K, 101.2 kPa) to ensure an oxygen-free atmosphere. The temperature of pyrolysis was held for 2 h 10 min. The BC products at different charring temperatures (300 °C–600 °C) were left inside the furnace to cool to

ambient temperature. Then the BC products were milled to pass through a 0.25 mm sieve (#60 mesh) for further analysis and use. The produced BCs are named as BC300, BC400, BC500 and BC600, where BC and numbers denoting biochar and pyrolysis temperatures, respectively. Three batches of BC300, BC400, BC500 and BC600 were produced, respectively.

In September 2013, the top 20 cm soil was collected from agricultural fields (Yangling, Shaanxi Province), which is located at the southern edge of the Loess Plateau (34°36'N, 108°72'E). Soil samples were air-dried and roots and other visible plant remains were removed. Soil samples were sieved (2 mm) for the incubation experiment. The Lou soil (Earth-cumuli-Orthic Anthrosol) is gray-brown, loose and granular with silty sand particles. The physicochemical characteristics of the soil were pH = 8.11, organic carbon = 9.27 g kg⁻¹, total N = 0.65 g kg⁻¹.

2.2. Experiment design

An incubation experiment was conducted over 360 days to investigate the effect of BC on the contents of SOC, soil labile organic C and humic fractions. One hundred grams of air-dried soil (< 2 mm) were weighed into a 100 mL plastic container. BC300, BC400, BC500 and BC600 were added separately at the rate of 2% and mixed thoroughly with soil. A non-amended control soil treatment was also included. Each treatment had 39 replicates. Three replicates of each treatment were destructively collected after 1, 3, 6, 10, 20, 40, 60, 90, 120, 180, 240, 300 and 360 days incubation, respectively. Temperature (25 °C) was kept constant during the entire experiment. The moisture content of each sample was adjusted to 70% of the water-holding capacity and the moisture was kept constant by adding deionized water every sampling time. Soil samples were analyzed for SOC, labile soil organic C (MBC and DOC) and the C contents of HS (HA, FA and Hu).

2.3. BC characteristics analysis

The total elemental content of C, H and N in BC samples were measured by C H N analyzer (Vario EL III, Germany) (Claoston et al., 2014). The oxygen content (%) was calculated by the following equation: O (%) = 100 % - (C % + H % + N %). The H/C and O/C ratios were also calculated.

The method of Brunauer–Emmett–Teller (BET) is commonly used to determine the total surface area of materials (Sun et al., 2014). The BET analysis was carried out using a NOVA 2200e surface area machine (Quantachrome Instruments, USA). BC samples were analyzed for multipoint BET surface area using nitrogen as adsorptive gas at 77 K.

2.4. Analysis of soil organic C and its fractions

Total organic carbon was determined by the dichromate wet oxidation method (Sun et al., 2013). Soil microbial biomass C (MBC) was determined by the fumigation-extraction method (Wu et al., 1994). In brief, fresh soil (equivalent 5 g oven dry weight) was fumigated with ethanol free chloroform for 24 h, and then the fumigated soil sample and another equal weight non-fumigated sample were extracted with 20 mL 0.5 mol L⁻¹ K₂SO₄. The C content of extracts was measured using a TOC Analyzer (TOC-UCPH, Japan). MBC was calculated from the TOC by using a K_{EC}-factor of 0.45. Soil dissolved organic C (DOC) was extracted with a soil: water ratio of 1:10 (w/w), filtered (0.45 μm) with a vacuum extraction set after 30 min shaking at room temperature. The organic C in the extracted solution corresponding to DOC (Blair et al., 1995). The C content of the solutions was measured using a TOC Analyzer (TOC-UCPH, Japan).

A modified method based on the method used by the International Humic Substances Society (IHSS) was adopted to extract the LHS of BC and the HS of soil. Briefly, an equivalent of 10 g oven dry weight BC or soil were extracted with a 0.1 mol L⁻¹ NaOH + Na₄P₂O₄ solution at

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