



Quantitative analysis of biochar in field soil

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ABSTRACT

Biochar is used with increasing frequency as a soil amendment because of its potentially beneficial effects on soil carbon sequestration, crop yield, nutrient leaching and greenhouse gas emissions. Simple methods for the analysis of biochar in soil, however, are currently unavailable. Therefore, we have adapted the “loss on ignition” method for this purpose. The technique requires knowledge of the proportions of both biochar and biochar-free soil that are lost on ignition. One can use values determined prior to the amendment of the soil with biochar, assuming that the values do not change after biochar is incorporated in the soil. We tested these assumptions. Over the course of 15 months, the assumptions proved to be valid under our test conditions. The technique accurately determined a wide range of biochar concentrations in field soil.

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1. Introduction

Thermochemical conversion (pyrolysis) is one route for the production of liquid fuel from biomass (Boateng et al., 2006). One of the byproducts from pyrolysis is biochar, which may be useful as a soil amendment due to its unusual chemical and physical characteristics. Biochar contains high concentrations of carbon that can be rather recalcitrant to decomposition, so it may stably sequester carbon (Glaser et al., 2002). It can increase soil aeration (Laird, 2008) and reduce soil emissions of N₂O, a greenhouse gas (Spokas et al., 2009; Singh et al., 2010). It has increased crop yield through various mechanisms including stimulation of beneficial soil microbes such as mycorrhizal fungi (Warnock et al., 2007), increase of soil base saturation (Glaser et al., 2002; Major et al., 2010a,b), increase in water holding capacity (Glaser et al., 2002; Steiner et al., 2007), and retention of nutrients in the portion of the soil column containing roots, thus improving nutrient use efficiency (Chan et al., 2007; Steiner et al., 2008).

There are multiple situations in which it might be important to measure the amount of biochar in soil. Effective and fair carbon sequestration compensation schemes may require documenting both the temporal and spatial stability of biochar. Biochar is capable

of migrating in soils (Major et al., 2010a,b) and, while it is generally assumed to be recalcitrant, a portion of most biochars, particularly fresh biochars, is “easily decomposable” (Lehmann et al., 2009). Therefore, repeated analyses of biochar in time and space become necessary. Moreover, because biochar has the potential to improve crop growth and reduce leaching and greenhouse gas emissions (see above), its cost-effective use will require knowledge of the relationship between biochar quantity and the magnitude of these benefits.

Despite the clear need for a simple and routine way to quantify biochar in soils, the complex chemistry of biochar and the difficulty in distinguishing biochar from other forms of organic matter has resulted in methods that are either extremely labor intensive or require specialized instrumentation, both of which are impractical for routine analysis by most scientists (Manning and Lopez-Capel, 2009). For example, biochar in soils has been quantified by hand-sorting (Zackrisson et al., 1996), *in situ* analysis via scanning calorimetry, NMR spectroscopy or infrared spectroscopy (Schmidt and Noack, 2000; Manning and Lopez-Capel, 2009; Nguyen et al., 2009), analysis of molecular markers (Manning and Lopez-Capel, 2009), or by preferential removal of inorganic and non-biochar organic C by selective oxidation or acid treatment followed by analysis of residual organic material (presumably equivalent to biochar) by NMR, optical, or mass spectroscopy, or thermal conductivity (Schmidt and Noack, 2000; Elmquist et al., 2004; Manning and Lopez-Capel, 2009). None of these methods is suited to routine analysis of biochar in soils by most agronomists or

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ecologists engaged in soil amendment studies. Our goal, therefore, was to adapt the familiar “loss on ignition” method for use in the routine analysis of biochar in soil.

2. Material and methods

2.1. Development of the method

Our method for analysis of biochar in soils is based on the “loss on ignition” method for the analysis of soil organic matter (Heiri et al., 2001). Loss on ignition is considered to be superior to wet combustion for the estimation of soil organic matter because it accounts for a greater fraction of the organic matter present in the sample, but it can overestimate soil organic matter if there is substantial carbonate present in the sample (Byers et al., 1978). A temperature of 550 °C is generally assumed to oxidize the conventional (non-thermally altered) organic matter in most soils (Ball, 1964; Heiri et al., 2001). In preliminary tests we determined that 550 °C also thermally oxidized the organic portion of biochar produced from hardwood via conventional charcoaling (slow pyrolysis) methods. We therefore subsequently used 550 °C in all experiments of this study.

Because both conventional soil organic matter and biochar are oxidized in the same process, the weight that is lost on ignition from a soil sample containing biochar can be given in the following equation:

$$LI = OM + CH_{LI} \quad (1)$$

where:

LI is the total weight of the sample that is lost on ignition,

OM is the weight of the native, non-biochar organic matter resident in the soil (frequently referred to as thermally unaltered organic matter, Schmidt and Noack, 2000) that is lost on ignition, and

CH_{LI} is the weight of the biochar in the soil sample that is lost on ignition.

The portion of the biochar in the soil sample exposed to 550 °C that is not lost on ignition is the mineral component of biochar or ash. Our soil, described below, did not contain measurable concentrations of carbonate.

In the absence of biochar, OM is operationally defined as that portion of the soil that is lost on ignition (Heiri et al., 2001). It is assumed to be the non-biochar organic matter resident in the soil. Therefore, in a soil sample containing biochar:

$$OM = q(W - CH) \quad (2)$$

where:

q is the proportion (by weight) of pure soil (without biochar) that is lost on ignition,

W is the weight of the soil sample containing biochar, and

CH is the weight of the biochar in the sample (including that which is lost on ignition plus the ash component of the biochar that remains after combustion).

CH_{LI} , the weight of biochar lost on ignition, can be given by the following equation:

$$CH_{LI} = y CH \quad (3)$$

where:

y is the proportion (by weight) of biochar that is lost on ignition. We assume this is the organic fraction of the biochar.

Substituting Eqs. (2) and (3) into Eq. (1), we obtain:

$$LI = q(W - CH) + y CH \quad (4)$$

Rearranging the equation to solve for CH , we obtain:

$$CH = (LI - qW)/(y - q) \quad (5)$$

Thus, the weight of biochar in a soil sample can be calculated from the weight that is lost on ignition from the soil sample containing biochar, the weight of the soil sample containing biochar, the proportion of pure soil (not including the biochar) that is lost on ignition (q), and the proportion of the biochar that is lost on ignition (y).

2.2. Testing the assumptions of the method

The proposed method for biochar analysis assumes knowledge of q , the proportion of the pure soil (without biochar) that is lost on ignition, and y , the proportion of the pure biochar (without soil) that is lost on ignition. We imagine that in most cases it will be difficult to separate biochar from a soil sample, especially if the biochar is fine, in order to routinely determine either q or y . However, in cases in which biochar is experimentally added to soil, samples of pure biochar can be analyzed to determine y and samples of pure soil can be analyzed to determine q . In order for that approach to be useful, however, one must assume that y and q do not change as a consequence of residence of the biochar in the soil. We tested these assumptions by adding biochar to soil in the field and, 15 months later, collecting field samples and separating the biochar from the remaining soil (see below) in order to determine whether q and y remained unaltered.

In June 2009, we rototilled to a depth of 25 cm a portion of a field formerly planted to corn at the Russell Larson Research and Education Center. Its location is 40°42'45.65"N, 77°57'26.21"W. This field was not the source of the soil described in Section 2.3, but it possesses the very same soil type. The soil is Hagerstown soil: fine, mixed, semiactive, mesic Typic Hapludalfs (USDA–NRCS, 2010). The surface texture is a silt loam with subsurface textures of a silty clay loam and silty clay. After rototilling, we laid out 4–1 m² plots in each of three blocks. Two plots in each block were amended with biochar while two plots were not and served as controls. Plots were separated from each other by 2 m. The biochar plots received approximately 5.7 kg (12.5 lbs) biochar, equivalent to approximately 56.8 tonne ha⁻¹. The biochar was produced from domestic hardwood via conventional, slow pyrolysis and obtained from Humphrey Charcoal (Brookville, PA, USA). In blocks 2 and 3 we used mesh size #6 biochar; the pieces of biochar were smaller than 3.4 mm in the smallest dimension. In block 1 we used mesh size #10 biochar; the pieces of biochar were smaller than 1.7 mm in the smallest dimension. Other than size, the biochars were identical. After spreading the biochar uniformly over the plots, we mixed it into the soil with spades to a depth of approximately 25 cm, turning the soil 3–4 times for thorough mixing. The area was then planted to sweet corn (Delectable, Rupp Seeds Wauseon, OH, USA), and the corn was harvested later that season. In May 2010, the same area was planted to soybean (FS H535A90, Growmark, Bloomington, IL, USA). In September 2010, when the biochar had been in the soil for 15 months, we collected soil samples to a depth of 15 cm from each of the 12 plots using a standard 2.5 cm diameter soil sampler (Oakfield Apparatus, Inc., Oakfield, WI, USA). Two cores were taken from each control (no biochar) plot and four cores were taken from each biochar plot.

We then separated out approximately 15 ml of biochar pieces from each biochar plot soil sample. The separation was facilitated by the fact that the biochar pieces were relatively large (see above). We employed a 5 mm soil sieve to perform the initial separation of biochar from soil. Most of the biochar in the sample remained on the sieve. The biochar pieces and soil aggregates remaining on the sieve were easily separated. The soil aggregates were retained as part of the pure soil fraction. The smaller aggregates of soil passing

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