A Rapid Test for Plant-Available Water-Holding Capacity in Soil-Biochar Mixtures

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Abstract

One of the benefits of the application of biochar to soil is its ability to increase the plant-available water-holding capacity (PAWC) of soil, and thereby, primary productivity. The increase in PAWC involves both the internal porosity of the biochar as well as new porosity created by gaps between the biochar particles and pre-existing soil particles (inter-particle porosity) and is expected to change over time as the biochar and soil particles adjust to their new neighbors. Multiple measurements over several years are likely to be required to understand these changes. Because measurement of PAWC by standard methods can take days, if not weeks, to complete, it is both difficult and expensive to estimate the changes in PAWC expected when soil is amended with biochar. To help address this situation, we refined and calibrated an inexpensive, rapid method for measuring PAWC of soil-biochar mixtures. The method is based on applying a specific level of water potential to a sample using a centrifuge. The sample is supported by a filter membrane fixed midway in a centrifuge tube, thus allowing drainage into the bottom of the tube to occur. The PAWC is calculated from the amount of water retained by an initially saturated sample after centrifugation for 90 minutes at a relative centrifugal force (RCF) of 1000 *g*. We calibrated the method against standard methods for determining PAWC and then applied it to a suite of 72 binary soil-biochar mixtures. The results show (1) biochar increases the PAWC of soils, but the contribution of biochar is not linearly proportional to the amount of biochar added, (2) soil texture, and possibly soil mineralogy in some instances, have a large impact on the degree to which biochar increases PAWC, and (3) inter-particle effects accounted for the majority of the overall impact of biochar on PAWC.

Introduction

Biochar is a promising material for reuse of organic waste in various industrial and environmental applications (Manyà, 2012). As an amendment to soil, biochar has great potential for carbon sequestration as well as improvement of soil fertility and soil physical properties (Sohi et al., 2010; Agegnehu et al., 2017; Ali et al., 2017). Key physical properties of soils that are affected by biochar amendments are porosity and hydraulic properties, which include water retention, hydraulic conductivity, water infiltration, and water repellency. In general, biochar amendment leads to an improvement of soil hydraulic properties, but the effects of biochar depend strongly on biochar type, its application rates, and on soil texture.

Biochar amendments to soil have been reported to increase water retention (Ajayi et al., 2016; Agegnehu et al., 2016; Zhang et al., 2016; Omondi et al., 2016; Blanco-Canqui, 2017). Higher rates of biochar applications usually lead to a greater increase in water retention, and the effects of biochar are more pronounced in coarse-textured soils than in fine-textured soils (Ajayi et al., 2016; Omondi et al., 2016; Blanco-Canqui, 2017). For instance, Gamage et al. (2016) compared the water retention in a sandy and sandy loam soil, and found that biochar application resulted in a more pronounced increase in water retention in the sandy soil.

The PAWC, which is defined as the amount of water retained by soil between the field capacity and the permanent wilting point, is an important parameter to assess how much water a soil can store and provide for plant use. The field capacity is usually defined as the water content at a volumetric water potential of −100, −300, or −330 hectopascals (hPa), and the permanent wilting point is the water content at a volumetric water potential of −15,000 hPa. Current methods to determine the field capacity and the permanent wilting point are the pressure-plate and the dew-point methods, respectively (Bittelli and Flury, 2009). An evaporation-based method (Schindler, 1980) has recently become popular also with the availability of the commercial HYPROP instrument (UMS, 2015). This method provides the entire soil-water retention curve, and thus allows the determination of the PAWC (Schindler and Müller, 2006; Peters and Durner, 2008; Schelle et al., 2013) as well as estimates of unsaturated hydraulic conductivity. While highly accurate, application of these methods requires lengthy equilibration times, typically 6 to 8 days for pressure-plate measurements, 2 days for vapor-pressure measurements, and 2 to 10 days for evaporation-based measurements, investment in specialized instrumentation, and offers low sample throughput.

Biochar amendment often leads to an increase of the water content at both field capacity and the permanent wilting point, but with a more pronounced increase at field capacity, so that PAWC overall also increases (Abel et al., 2013). This effect is most pronounced in sandy soils, but will also depend on the type of biochar used (Abel et al., 2013). Based on a meta-analysis of 74 datasets, Omondi et al. (2016) found that on average, biochar application led to a relative increase of PAWC by 15%.

Biochar amendments to soil improve PAWC due to two mechanisms: (1) biochar contributes new porosity to the soil because of the biochar's own internal porosity, and (2) interactions between biochar and soil particles lead to better soil structure (Andrenelli et al., 2016). The total porosity and intra-aggregate structure of the soil are usually increased by biochar application (Yu et al., 2016). Considering that coarse-textured soils have less porosity and surface area than fine-textured soils, the coarse-textured soils benefit more from the addition of biochar amendment. This has been widely reported, especially for sandy soils (Omondi et al., 2016; Suliman et al., 2017).

The particle size of biochar is also an important parameter affecting water retention in soils. For instance, Glab et al. (2016) applied three different size fractions of biochar (0– 500, 500–1000, and 1000–2000 μm) to a loamy sand, and found that PAWC was increased the most by the finest biochar fraction. This was attributed to fine biochar particles infilling pore space between sand particles, thus contributing to greater water retention. Esmaeelnejad et al. (2016) compared the effects of two biochar size fractions (0–1000 and 1000–2000 μm) from four different biochars on the PAWC in a sandy loam soil, and found that while all biochar amendments significantly increased PAWC, an effect of biochar particle size was only found for one of the biochars—the <1 mm fraction of a high temperature biochar from wood significantly enhanced PAWC relative to the coarser 1-2 mm fraction. In contrast, Liu et al. (2017) reported that the medium and coarse fractions of biochar (251–853 and 853–2000 μm) improved PAWC of a sand more than did the fine fraction (0–251 μm).

Given the lengthy time and high expense associated with measurement of PAWC by conventional methods, and that the factors controlling the degree to which biochar affects PAWC change with time (thus requiring multiple measurements), a need clearly exists for a rapid, inexpensive, and reasonably accurate method for measuring PAWC of soil-biochar mixtures. One such approach involves the use of centrifugation (Cassel and Nielsen, 1986). Briggs and McLane (1907) recognized early that water will not drain readily from a soil sample taken to the laboratory under normal gravity and proposed a centrifuge method to remove water held under larger gravity pressures. Soil samples were placed into cups with perforated bottoms and then centrifuged at a known acceleration until a constant water content was achieved. Briggs and McLane (1907) called the water content obtained after centrifuging soil samples at a certain relative centrifugal force (RCF) the "moisture equivalent." A constant RCF of 1000 *g* was proposed by Briggs and McLane (1910).

Briggs and McLane (1907) initially used this method to compare the water holding capacity of different soils, and later adapted the method to determine the water content of soils at which permanent wilting of plants occurs (Briggs and Shantz, 1912a; Briggs and Shantz, 1912b). This water content, termed the "wilting coefficient" (determined for different plant species in wax-sealed soil systems), was found to correlate well with the "moisture equivalent" determined at a RCF of 1000 *g*, with the correlation being "wilting coefficient" = "moisture equivalent"/1.84 (Briggs and Shantz, 1912a; Briggs and Shantz, 1912b).

A disadvantage of the centrifuge method of Briggs and McLane (1907), however, is that the water potential at the lower boundary of the soil is undefined. Russell and Richards (1938) improved the centrifuge method by using an inverted ceramic cup and a defined water level in the centrifuge tube to control the lower boundary condition of the soil so that the water potentials in the soil samples could be calculated. Although it eliminates one disadvantage of the centrifuge method, the Russell and Richards (1938) approach requires a fairly sophisticated sample holder. Shortly after their report, the commercial pressure-plate apparatus was developed (Richards, 1938; Richards, 1941; Richards and Fireman, 1943; Coleman and Marsh, 1961) and currently most laboratory-based field-capacity measurements for PAWC determinations are performed with this instrument.

The relatively recent commercial development of disposable centrifuge filter units and highly accurate electronic analytical balances, coupled with the ready availability of laboratory centrifuges capable of handling dozens of samples simultaneously, suggests that a centrifuge-based method patterned on the early work of Briggs and co-workers might provide the high throughput and reasonable accuracy desired for measurement of PAWC of soil-biochar mixtures. Preliminary work on this approach, involving mixtures of biochar with a synthetic soil consisting of 30-50 μm-diameter borosilicate glass beads to simulate a silt-textured soil, was performed in 2012 (J. E. Amonette, unpublished data) as a way of screening different biochar samples, but was never calibrated to the conventional methods. The primary goals of the present work were to further develop and calibrate the centrifuge approach and then apply it to a large suite of representative soil and biochar mixtures to demonstrate its potential use.

Experimental

Experimental design

To achieve the overall goal of the project, we designed an experimental matrix of nine natural soils and four biochars, incorporated at two levels, from which 72 soil-biochar mixtures (treatment combinations) were prepared (Figure 1). The soils and biochars are described in greater detail in the *Materials* section. Thus, we prepared eight different mixtures with each soil, two for each biochar.

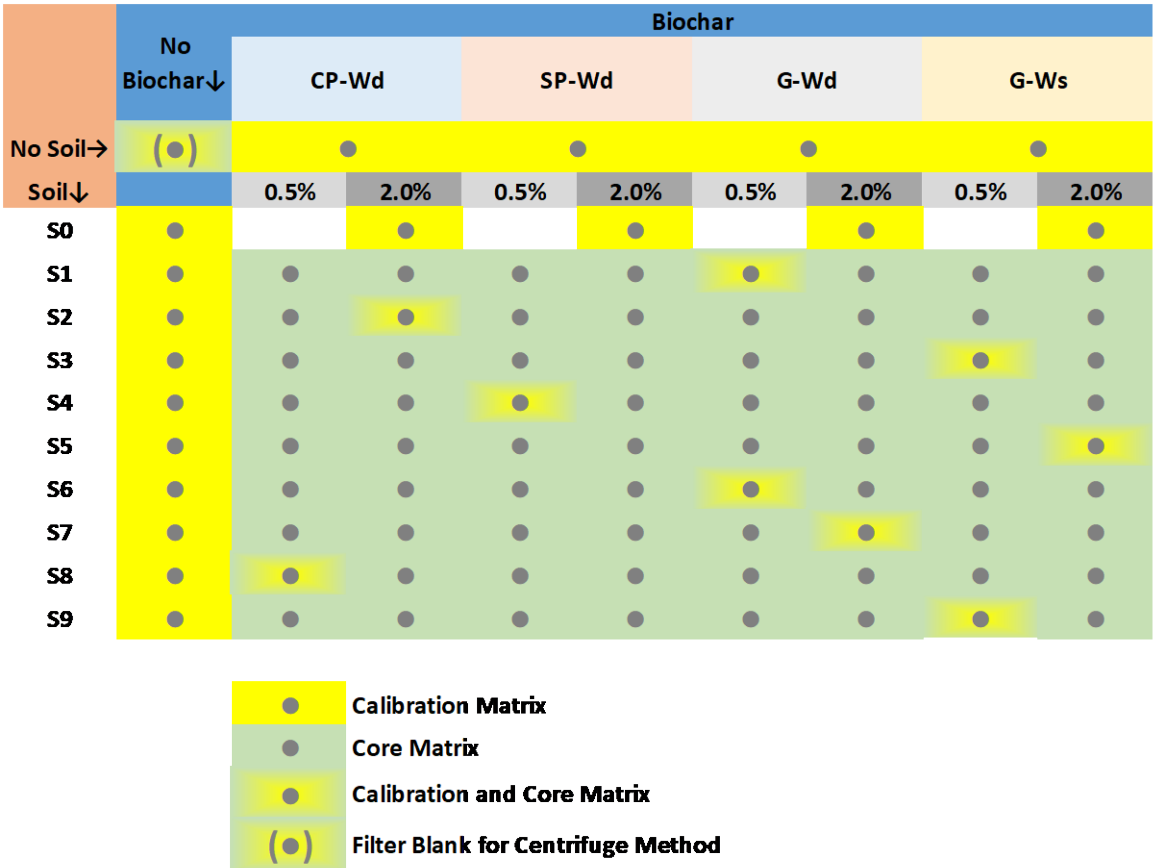

Figure 1: Experimental design matrix showing biochars, soils, and biochar-soil mixtures used during the calibration and application stages of the project

During the calibration stage, we randomly selected a subset of nine soil-biochar mixtures, one for each soil, for analysis by both the reference and centrifuge-based methods. In addition, we analyzed the nine soils and the four biochars that were not mixed, as well as a synthetic soil, alone, and after mixing with each of the four biochars at the highest level. The total number of treatment combinations during the calibration stage was 27. We analyzed three replicates by the reference methods (81 experimental units), and two replicates by the centrifuge method (54 experimental units).

We used the centrifuge method to analyze this core experimental matrix in duplicate (144 experimental units) in the first application of the method after the calibration stage of the project was completed.

Materials

Biochars

We selected four commercially available biochars to represent the types of feedstocks and biochar manufacturing processes most likely to be encountered for large-scale application to agricultural soils. In this report, we identify each biochar by a code that combines information about the production process (CP, SP, or G) in the first syllable with that for feedstock (Wd or Ws) in the last syllable, separated by a hyphen (Table 1, Figure 1). The production processes include one unknown (i.e., CP, but of nominally less importance as the biochar is subsequently "engineered" to achieve the desired properties prior to sale and use), one batch process using slow-pyrolysis (SP) kilns, one continuous large-capacity wood boiler (i.e., gasifier, G) process co-generating electricity, and one continuous stand-alone small-capacity gasifier (G). The feedstocks include three biochars made from woody biomass (Wd) feedstocks and one from wheat straw (Ws). The G-Wd biochar was from the same batch being used in field trials of potatoes and strawberries by Gang et al. (2019).

Table 1: Provenance and selected physical properties of the biochars used in the project

Biochar	Feedstock	Manufacturing Process	Supplier	Contact Angle[1]	Bulk Density	Organic C Content
				(degrees)	(g cm^{-3})	(wt%)[2]
CP-Wd	Wood (pine)	Engineered	Cool Planet, CO	95 ± 5	0.24	82.2
SP-Wd	Wood (pine)	Slow pyrolysis	Biochar Now, CO	93 ± 1	0.17	81.3
G-Wd	Forest wood residuals (Douglas fir, pine)	Gasification	Oregon Biochar Solutions, OR	< 10	0.09	85.0
G-Ws	Straw (wheat)	Gasification	Ag Energy Solutions, WA	< 10	0.19	66.9

[1] The angle formed in the water phase between the water-solid surface and the water-air surface when water contacts a solid in the presence of air. Contact angle is measured to determine the hydrophobicity of a surface. A high contact angle indicates greater hydrophobicity (less interaction of water with the surface).
[2] Reported on an oven-dry basis

Biochars were obtain from suppliers, air dried, and then sieved through a 2-mm sieve. Larger portions were crushed with a glass bottle on a stainless-steel tray and passed through the sieve. The sieved material was then dried at 60 °C in an oven for 48 hours, and finally stored in sealed Corning glass jars. Water content of all biochar samples after processing was determined by drying at 105 °C for 24 hours.

Contact angles (air-water-solid) were measured with a digital goniometer (DSA 100, Krüss, Hamburg, Germany). Contact angle is a measure of how easily water can interact with the surface of a solid. The higher the contact angle, the less likely water will adhere to the solid, and the more hydrophobic the surface. Air-dry biochar samples were crushed with a mortar and pestle to obtain a fine powder of biochar. The biochar powder was then sprinkled onto a glass microscope slide covered with a strip of double-sided tape, until biochar particles formed a smooth surface. Loose particles were shaken off by tapping the slide. The slides were then placed on the goniometer platform and 3 μL of nanopure water were dosed as a sessile drop onto the biochar-covered slide. Contact angle on the sessile drops was determined by the Young-Laplace method (Shang et al., 2008). Measurements were made at 25 °C on 10 to 14 replicates and reported as mean and standard deviation. For some biochars, the water drop spread so rapidly that no contact angle could be measured. For these biochars, the contact angle was reported as <10°.

Bulk densities of the biochars were measured as part of the determination of field capacity by the pressure-plate method. Briefly, air-dried samples were packed into brass cylinders (5.35-cm diameter and 3.0-cm height). Samples were packed in 1.5-cm height increments and compressed with a constant pressure of 2,011 Pa. The pressure was applied with a 50 mL glass beaker and a 256.73 g lead block. At the end of the field capacity determination, the samples were dried at 105 °C for 24 hours and weighed after cooling in a desiccator. Bulk chemical properties (i.e., ultimate and proximate analysis, Table 2) were determined by Huffman Hazen Laboratories (Golden, CO, http://www.huffmanlabs.com).

Table 2: Ultimate/proximate analysis of biochars used in the project

Biochar	Carbon	Hydrogen	Oxygen[1]	Nitrogen	Sulfur	Ash	Drying Loss[2]
	------------------------------------ weight percent (as received) --------------------------------------						
CP-Wd	81.55	2.47	10.02	0.14	0.03	4.93	0.83
SP-Wd	80.61	2.40	8.75	0.15	0.01	7.28	0.81
G-Wd	83.43	0.77	6.85	0.43	0.03	6.64	1.85
G-Ws	66.07	2.13	6.10	0.43	0.08	23.91	1.28

[1]Determined by difference after subtracting drying loss

[2]Dried at 110 °C for 24 h

Soils

We selected nine natural soils from Washington having textures that encompass the range of textures typically found in the top horizon of Washington soils (Table 3, Figure 2). Three silt loams (S1, S2, and S4) were selected because silt loam is the soil texture of major agricultural areas in Washington (e.g., Palouse silt loam (S4) is the major soil for the wheat producing area in eastern Washington, and Skagit silt loam (S1) is the major soil for specialty crop production in western Washington). Two of the silt loam soils (S1 and S2) are from the field plots in Mount Vernon and Puyallup being used to test effects of G-Wd biochar amendments on production of potatoes and strawberries by Gang et al. (2019). The Quincy sand (S3) was also selected because it is a widespread soil used in irrigated agriculture (typically potatoes) in the central portion of the state.

The soils were air-dried and sieved through a 2-mm sieve and stored in sealed plastic buckets. Air-dry water content was determined by drying the soils at 105 °C for 24 hours. Soil texture according to the USDA soil texture classification was determined by sieving and the hydrometer method (Gee and Or, 2002). Bulk density was determined during the field capacity determination as described previously for the biochars. Organic carbon content (C_{org}) was determined by Huffman Hazen Laboratories (Golden, CO, http://www.huffmanlabs.com).

In addition, we used one synthetic soil (S0), consisting of solid borosilicate glass beads having diameters of 30-50 μm (Spheriglass® 3000E, Potters Industries, Valley Forge, PA, www.potterbeads.com), to simulate a silt-textured soil having no organic matter present.

Table 3: Names, typical crop, and selected physical and chemical properties of soils used in test

Soil	Typical Crop	Series Name[1]	Textural Class[2]	Texture[2]			Bulk Density[3]	C_{org} Content[4]
				Sand	Silt	Clay		
				--------- weight % ---------			(g cm^{-3})	weight %
S0[5]	--[6]	--	silt	0	100	0	1.67	0.0
S1	potato	Skagit	silt loam	9	74	17	1.25	1.14
S2	strawberry	Sultan	silt loam	22.3	64.3	13.4	1.26	1.30
S3	potato	Quincy	sand	98	1	1	1.78	0.12
S4	wheat	Palouse	silt loam	13.2	68.6	18.2	1.4	0.50
S5	forest	Salkum	silty clay loam	11.9	59.7	28.4	1.19	2.21
S6	--	Kapowsin	sandy loam	59	32	9	1.32	0.66
S7	forest/ pasture	Salkum B/C-Horizon	clay	9	33	58	1.04	0.49
S8	--	Briscot/Kitsap	loam	45	40	15	1.26	1.07
S9	forest/ pasture	Harstine	loamy sand	82	14	4	1.67	0.06

[1]NRCS-USDA National Cooperative Soil Survey, Official Soil Series Description
[2]USDA Soil Classification
[3]Bulk density of packed soil used for field capacity measurements
[4]Organic carbon content, reported as weight % as received
[5]Solid borosilicate glass beads
[6]--Not applicable

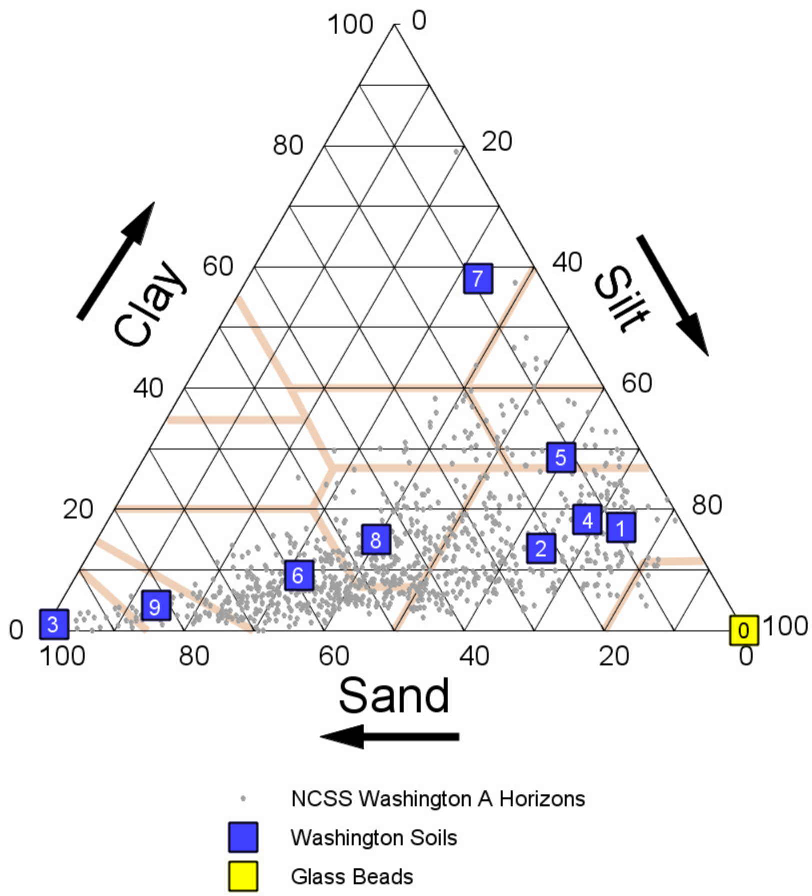

Figure 2: Soil textural triangle showing textural distribution of Washington A horizons in the USDA National Cooperative Soil Survey database, and the nine natural Washington soils and one synthetic soil (borosilicate glass beads) used in this work

Soil-biochar mixtures

For each of the 36 soil-biochar combinations, we prepared two mixtures at nominal biochar-C concentrations of 0.5 wt. % C and 2.0 wt. % C. These levels roughly correspond to biochar amendment rates of 10 and 50 t C ha^{-1} when mixed to the 15-cm plow depth. Amendments at rates higher than 50 t C ha^{-1} are generally not performed due to deleterious effects on plant growth.

For the reference method, we mixed appropriate amounts of each sieved oven-dried biochar (60 °C) with air dried soil in a Nalgene plastic bottle to obtain the different experimental treatments. Collection of data in triplicate for field capacity and permanent wilting point (PWP) measurements required preparation of 300-450 g (depending on soil bulk density) of biochar-amended soil. For the much smaller sample sizes used in the centrifuge method, we mixed a total of 15 g of soil (oven dried basis) with the appropriate amount of biochar, which we crushed to a fine powder to ensure reproducible amounts of biochar in each sub-sample analyzed. The procedure involved weighing out the biochar at a precision of 0.0001 g and gently crushing it (to approximately a coarse silt texture) with an alumina mortar and pestle. We then added the soil to the biochar in the mortar and mixed the two materials with a minimum of crushing until a uniform mixture (determined visually) was obtained. We stored the mixture in a glass vial until needed for the PAWC analysis.

Methods for plant-available water-holding capacity

Reference method

Determination of PAWC by the conventional approach involves separate measurements of the moisture content at field capacity and the PWP. The difference in these two values is the PAWC.

The moisture content at field capacity was measured with a pressure-plate apparatus (Klute, 1986; Figure 3A). Air-dried soil and soil/biochar samples were packed into brass cylinders (5.35-cm diameter and 3.0-cm height). Samples were packed in 1.5-cm height increments and compressed with a constant pressure of 2,011 Pa. The pressure was applied with a 50 mL glass beaker and a lead block of 256.73 g. The bottom of the cylinders was covered with a polystyrene mesh (250 μm mesh size) to contain the soil samples in the cylinder. Initial tests showed that the mesh had no effect on the determination of the field capacity. No significant differences were observed between samples with and without mesh.

The packed cylinders were then placed onto a porous ceramic membrane (No. 0675B05M1, effective pore size 0.5 μm, Soil Moisture Equipment Corp., Santa Barbara, CA; Figure 3B), and saturated for 12 hours with a solution of 5 mM $CaSO_4$, which was allowed to imbibe from the bottom of the samples to ensure no air entrapment during saturation. Samples were then pressurized in a pressure chamber (Soil Moisture Equipment Corp., Santa Barbara, CA; Figure 3A) at 330 hPa. Outflow from the pressure chamber was monitored daily, until no outflow was observed for 2 days, which we considered as criterion for equilibration. This equilibration time differed among samples and ranged from 4 to 6 days. The samples were then removed from the pressure chamber, weighed, and oven dried at 105 °C for 24 hours to determine the gravimetric water content.

Some soils shrank during the pressure plate measurements. For these soils, we estimated the volume of shrinkage by measuring the distance in height between sample ring and soil surface with a caliper. The soil volume was then corrected for this shrinkage, and used for the bulk density calculation. Three replicates were used for each sample.

The moisture content at the PWP was determined with a dew-point psychrometer (WP4 Dew Point Meter, Decagon Devices, Pullman, WA, Figure 3C). Measurements were made at a temperature of 20 °C. Soil, biochar, and soil-biochar mixtures were wetted with deionized water and equilibrated in 500-mL Nalgene bottles for 12 hours. The amount of water for wetting was chosen to obtain a water content which results in a water potential close to the PWP. We estimated the amount of water needed based on the soil texture and tabulated values (Campbell, 2012). Water was added with a syringe to the Nalgene bottle, the soil mixed with a steel rod, and the Nalgene bottle capped and rolled several times to further enhance the mixing. Three replicates were used for each sample.

After equilibration, samples were placed into a plastic WP4 sampling cylinder and the water potential was measured with the WP4 dew-point psychrometer. After the measurement, the water content of the sample was determined by oven-drying at 105 °C for 24 hours. The PWP was then calculated by the following equation (Campbell, 2012):

$$w_{-15,000} = w_{\text{measured}} \{\ln(-10^7 \text{ hPa} / -15{,}000 \text{ hPa}) / \ln(-10^7 \text{ hPa} / \psi_{\text{measured}})\}$$

where $w_{-15,000}$ is the gravimetric water content at the permanent wilting point (−15,000 hPa), w_{measured} is the measured gravimetric water content of the sample, and ψ_{measured} is the measured water potential (hPa). This equation is based on a linear interpolation of the water retention curve (in the logarithmic-form of the water potential) between -10^4 and -10^7 hPa (oven-dry).

Centrifuge method

The measurable quantity in the centrifuge method is the moisture equivalent, i.e., the quantity of water retained per unit mass of oven-dry soil after centrifugation at an RCF of 1000 *g*. Calibration of this result with PAWC results obtained by the reference method allows a value for PAWC to be calculated from the moisture equivalent.

To determine the moisture equivalent, we weighed 1.5 g of air-dry soil, biochar, or soil/biochar mixture (0.0001-g precision) into a tared filter top from a 2-ml capacity centrifugal filter device (SUN-Sri™ 0.2-µm nylon filter membrane in a polypropylene housing, Thermo Fisher Scientific Catalog #10-800-188, www.fishersci.com; Figure 3D). An aluminum foil tube was used to facilitate sample transfer with a minimum of static. The filter top and cap were re-weighed. To saturate the sample, we added 4.5 mL of 0.005 M $CaSO_4$ solution to the receiving tube of the filter device and 2 mL of this solution to the filter top and then assembled the filter device. The sample was allowed to equilibrate with the solution for a minimum of 12 hours, preferably 24 hours, by supporting the bottom of the filter device by the bottom of a 20-mL plastic scintillation vial held in a 100-sample rack (Figure 3E). We then poured off the solution in the receiving tube of the filter device, re-assembled the device, and, after balancing tubes by adding water to the receiving tube as needed, centrifuged it at RCF = 1000 *g*.

During the method development, we used a non-refrigerated Beckman GS15 table-top centrifuge (Beckman Coulter, Brea CA) equipped with a S4180 bucket rotor (capacity of 12 filter tubes) and operated at room temperature for periods ranging from 20 to 90 minutes. In the final run with the core matrix of soil-biochar mixtures, we used a refrigerated Eppendorf 5810 R table-top centrifuge (Eppendorf AG, Hamburg, Germany) equipped with an A-4-81 swinging bucket rotor (capacity of 48 filter tubes) and operated at 20 °C for 90 minutes.

After centrifugation, we determined the final weight of the filter top (precision = 0.0001 g) and calculated the moisture equivalent by the following equation:

$$\text{ME} = [(\text{SF}_{\text{final}} - (\text{S}_{\text{OD}} + \text{SF}_{\text{tare}})) - (\text{EF}_{\text{final}} - \text{EF}_{\text{initial}})] / \text{S}_{\text{OD}},$$

where ME is the moisture equivalent, SF_{final} is the final weight of the filter top containing sample, S_{OD} is the oven-dry weight of the sample, SF_{tare} is the tare weight of the filter top before sample was added, and EF_{final} and $EF_{initial}$ are the final and initial weights of a separate empty filter tops treated in the same manner but containing no sample. We determined S_{OD} values indirectly, based on the moisture contents of the soil and biochar samples at the time of preparing the initial mixture. By avoiding the issues associated with irreversible changes associated with oven drying, this practice allowed us to re-analyze the same specimen multiple times, if need be. After centrifugation, we placed the capped filter tops containing sample in a sealed plastic container and stored them in a freezer until needed. Repeated analyses after thawing and re-wetting showed no significant change in the measured value of the moisture equivalent.

We calculated the PAWC using an equation of the form:

$$PAWC = (ME - b_{regr}) / m_{regr}$$

where b_{regr} and m_{regr} are the intercept and slope, respectively, of the regression equation relating ME as a function of PAWC measured by the reference method. These two parameters vary according to the matrix involved (i.e., natural soil, biochar, synthetic soil).

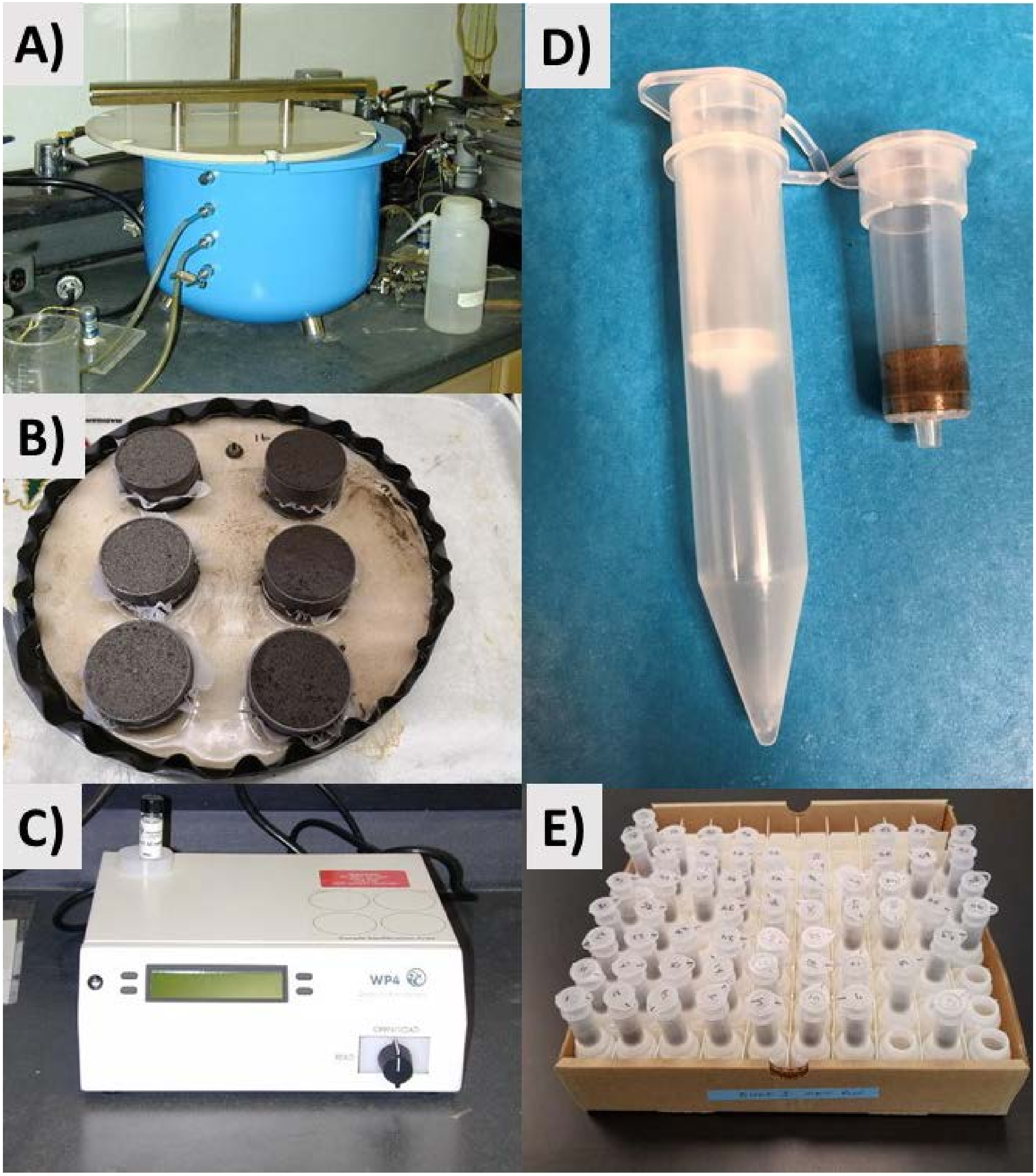

Figure 3: Apparatus used to measure soil moisture holding capacities by conventional and centrifuge methods. A) pressure-plate apparatus for conventional field capacity measurements, B) soils in cups on top of pressure membrane, C) dew-point psychrometer for conventional wilting point measurements, D) assembled centrifuge filter tube showing removable filter top containing soil to right, and E) rack containing large number of assembled and loaded centrifuge tubes

Results and Discussion

Method development

Briggs and McLane (2010) recommended a centrifugation time of 40 minutes to achieve equilibrium in determination of moisture equivalent of soils. To determine whether this time period was adequate for use with biochars and soil-biochar mixtures, we conducted a time study in which a saturated sample was centrifuged for 20 minutes, weighed, and then centrifuged further for an additional 10 minutes, re-weighed, and so on until a total of 90 minutes of centrifugation had been received by the sample. The water content of the sample relative to that after the first 20 minutes of centrifugation was then plotted to determine at what point equilibrium was established. This experiment was conducted with 12 samples representing a subset of the soils, biochars, and soil-biochar mixtures used in the core matrix.

The results (Figure 4) suggest that after 90 minutes, equilibrium was established for the soils, soil-biochar mixtures, and one of the biochars. The G-Wd biochar sample, however, continued to lose water throughout the centrifugation process and a projected equilibration time seemed to be many hours. Given that our main emphasis was on the soil-biochar mixtures and these equilibrated readily, we settled on 90 minutes for the standard centrifugation period.

The different result for the G-Wd biochar sample (Figure 4) likely stems from the presence of long, anisotropically oriented, and poorly connected pores in this wood-derived biochar when compared to the short, relatively isotropic, and well-connected pores typical of most soils and of the G-Ws biochar derived from wheat straw residues.

The moisture release curve obtained for the silt-sized glass beads (S0) was nearly identical to that for S2, the natural silt loam soil from Puyallup, which contained 64% silt. Similar curves were obtained for the two S0-biochar mixtures, which reached equilibration more readily than the natural soil-biochar mixtures tested.

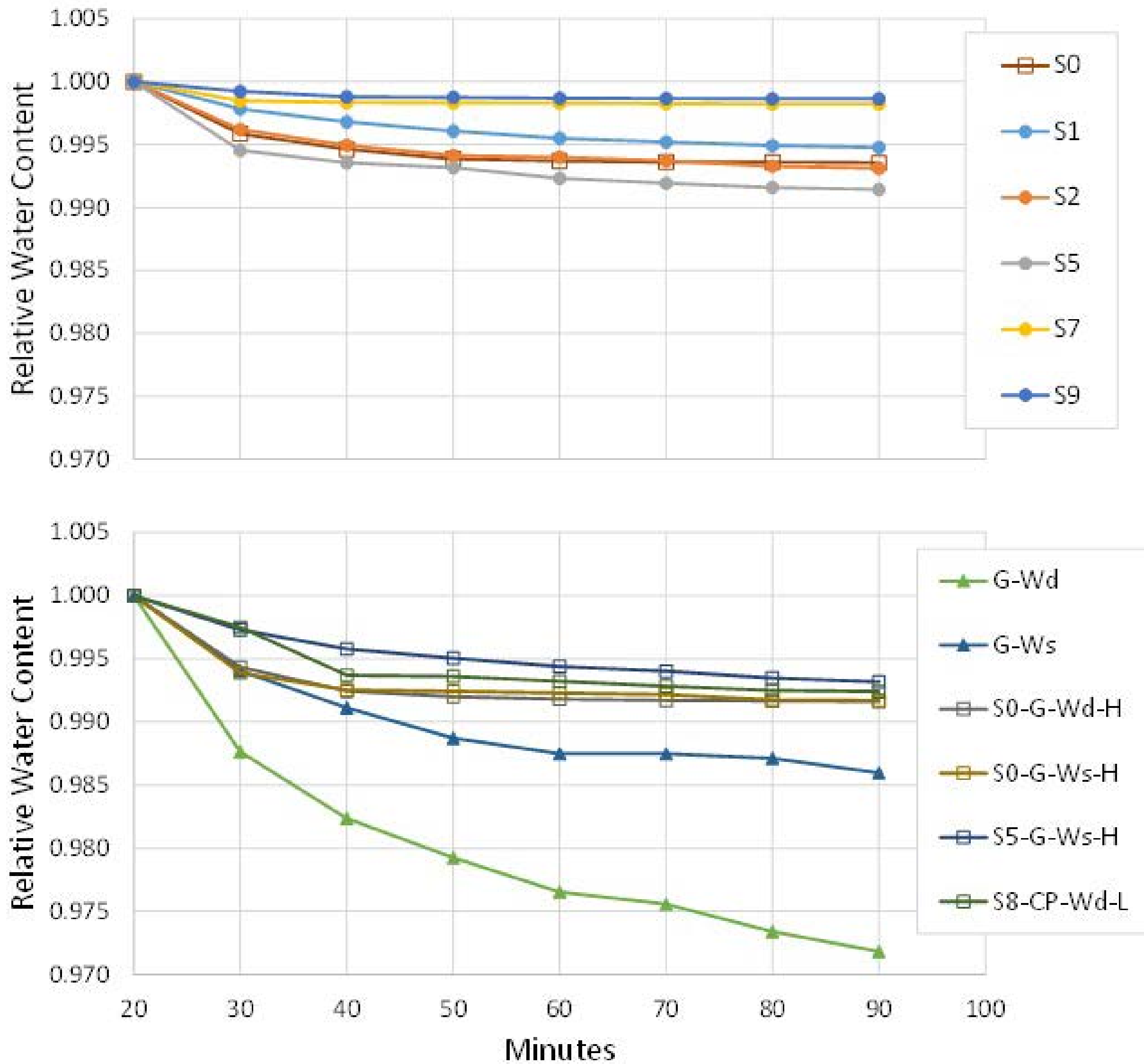

Figure 4: Water content remaining after 30 to 90 minutes of centrifugation at 1000 g RCF in natural and synthetic soils (top) and in biochar and soil-biochar mixtures (bottom). Water content is expressed relative to that present after 20 minutes of centrifugation.

Calibration

The results obtained for the unmixed biochars and soils in the calibration test matrix both yield reasonably strong correlation coefficients, 0.98 for biochar (Figure 5, Table 4) and 0.91 for natural soils (Figure 6, Table 4). Ideally, the slopes of the correlation equations would also be close to 1. The slope for the natural soils (1.13) is reasonably close to 1, whereas that for the biochars (0.50) is half the ideal value. The biochar for which the ME and PAWC values agree most closely is G-Wd, which was one of the two hydrophilic biochars having a low contact angle (Table 1). Conversely, the greatest disagreement between PAWC and ME values is for the SP-Wd biochar, which was one of the two hydrophobic biochars (Table 1). It is possible that the degree of hydrophobicity is a major factor, for which the two methods respond differently. Regardless of mechanism, the high linearity of the correlations obtained means that corrections to the ME values can be easily made to obtain reasonable estimates of the PAWC values.

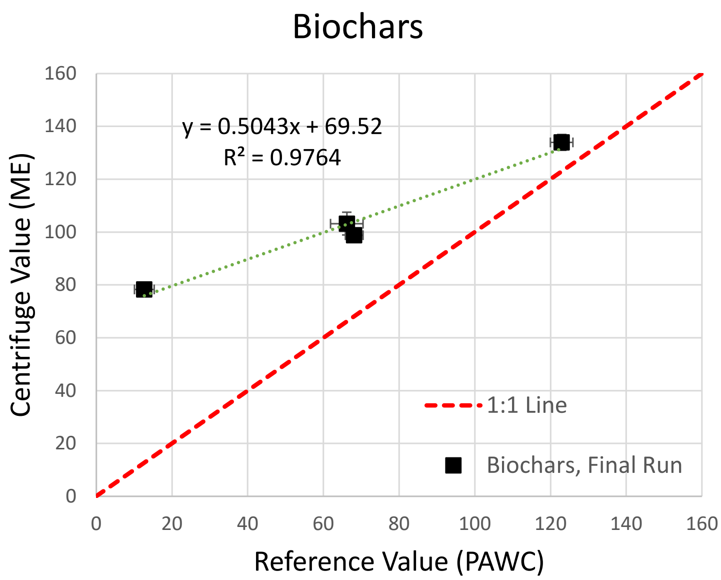

Figure 5: Plot showing correlation between centrifuge-measured moisture equivalent (ME) and conventionally measured value for plant-available water-holding capacity (PAWC) of biochar samples

Table 4: Regression equation parameters and predictive equations for calculation of plant-available water-holding capacity (PAWC) from moisture equivalent (ME)

Material Tested	Regression Equation Parameters			Equation for Calculation of PAWC from ME
	Slope	Intercept	r^2	
Biochar	0.5043	69.52	0.98	PAWC = (ME - 69.52) / 0.5043
Natural Soil	1.133	2.4803	0.91	PAWC = (ME – 2.4803) / 1.133
Synthetic Soil – Biochar Mixtures	0.2047	5.2773	0.48	PAWC = (ME – 5.2773) / 0.2047
Natural Soil – Biochar Mixtures	1.0676	3.9219	0.87	PAWC = (ME – 3.9219) / 1.0676
Natural Soil and Natural Soil – Biochar Mixtures	1.1004	3.1759	0.89	PAWC = (ME – 3.1759) / 1.1004

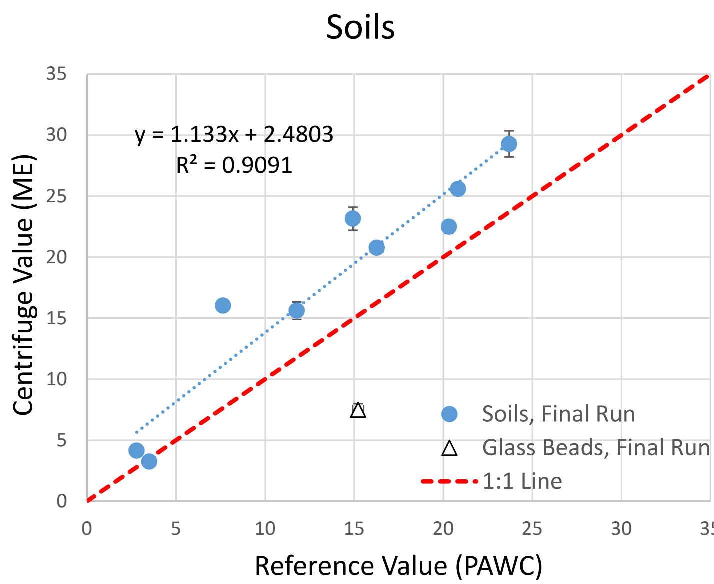

Figure 6: Plot showing correlation between centrifuge-measured moisture equivalent (ME) and conventionally measured value for plant-available water-holding capacity (PAWC) of nine natural soils and one sample of silt-sized glass beads

The correlations obtained for the soil-biochar mixtures differed for the natural soils and S0 (Figure 7, Table 4). The natural soil-biochar mixtures yielded a slightly lower correlation coefficient (0.87) than the unmixed biochars and natural soils, and an excellent slope of 1.07, closer to the ideal value of 1. In contrast, the correlations for the mixtures of S0 with biochars were relatively weak—a correlation coefficient of 0.48 and a slope of 0.20. In part this is caused by the low range in the values of PAWC and ME obtained with these mixtures stemming from the fact that only one glass-bead texture is considered. The mechanism driving the difference between natural soils and S0 probably relates to the relative uniformity and high degree of connectivity of the glass beads in S0, for which removal of water by centrifugation would be more efficient than by the reference methods. Because our primary interest is in natural soils and their mixtures with biochar, we will focus the remainder of the discussion on those matrices.

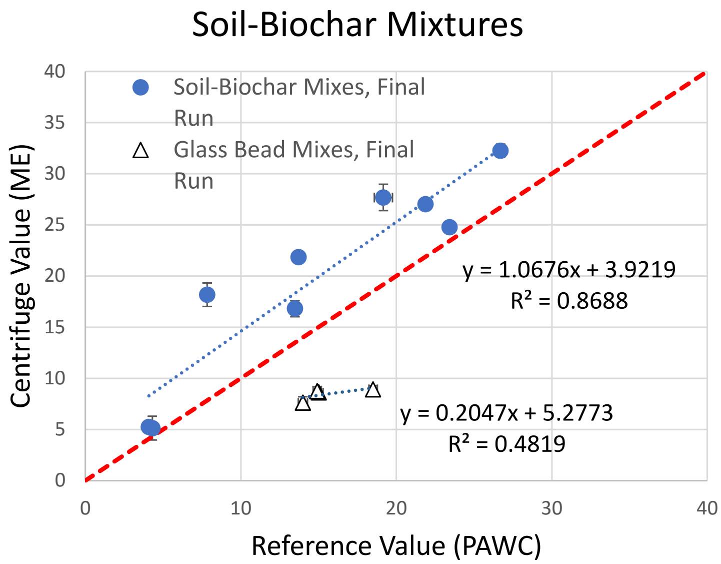

Figure 7: Plot showing correlation between centrifuge-measured moisture equivalent (ME) and conventionally measured value for plant-available water-holding capacity (PAWC) of nine mixtures of biochars with natural soils and four mixtures of biochars with one type of silt-sized glass beads

From a practical standpoint, it would be simpler to be able to use a single correlation equation for measurements of both soils and soil-biochar mixtures, and a comparison of the correlation results for natural soils (Figure 6, Table 4) and their mixtures with biochars (Figure 7, Table 4) suggests that they are very similar. We pooled the two datasets and found that the correlation between ME and PAWC was, as one might expect, very similar to those for soils and soil-biochar mixtures (Figure 8, top; Table 4). The correlation coefficient was 0.89 and the slope was 1.10, both of which are very good. Consequently, we decided to use this correlation equation for determination of PAWC for the core matrix samples by the centrifuge method.

To give an idea of the final calculation (using the equation given for "natural soil and natural soil-biochar mixtures" in Table 4) we have plotted calculated PAWC values obtained for natural soils and soil-biochar mixtures against the reference PAWC values in Figure 8 (bottom). Further, we looked at the change in PAWC (ΔPAWC) predicted for biochar amendments to natural soil for the nine samples analyzed by both the centrifuge and reference methods. This ΔPAWC (Figure 9) is the difference between the water holding capacity of the natural soil and that of the natural soil after amendment with biochar. In general, the results obtained by the two methods agree very well and for eight of the nine soils are not significantly different ($P = 0.05$) from one another. The impact of a hydrophobic biochar surface is clearly demonstrated for S4, amended with the SP-Wd biochar, for which a decrease in ΔPAWC was measured by the reference method. Although the centrifuge value for ΔPAWC did not show a decrease and was significantly different ($P = 0.05$), it nevertheless was the lowest ΔPAWC measured by the centrifuge method for the nine samples tested. The results shown in Figure 8 (bottom) and Figure

9 suggest that the centrifuge method will meet our criteria for reasonable accuracy combined with rapid and inexpensive methodology.

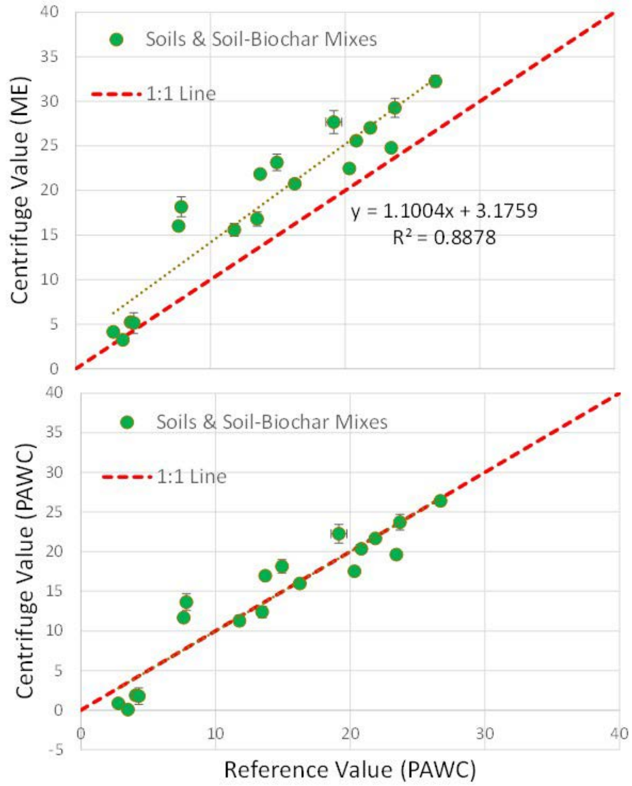

Figure 8: Pooled correlation between centrifuge-measured moisture equivalent (ME) and conventionally measured value for plant-available water-holding capacity (PAWC) of nine natural soils and nine mixtures of these soils with biochars (top); Pooled correlation between centrifuge- and conventionally measured PAWC values for soils and soil-biochar mixes (bottom). Error bars are one standard deviation

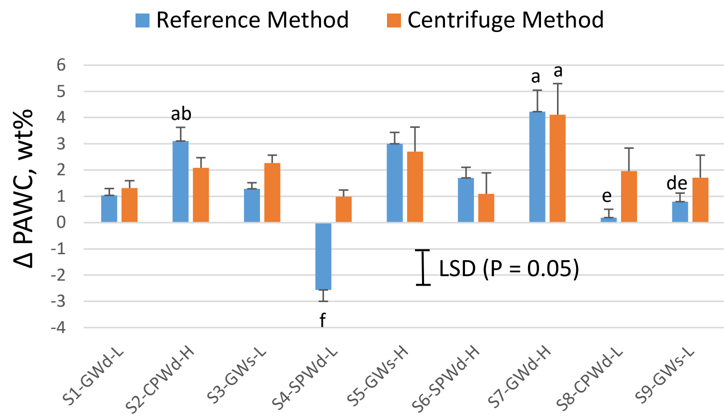

Figure 9: Comparison of the changes in plant-available water-holding capacity (PAWC) for selected biochar-soil mixtures measured by the reference and centrifuge methods. Error bars indicate one standard deviation. Different letters above error bars indicate significant (P=0.05) differences among means. Means having error bars without letters are similar to 3 or more other means. LSD (least significant difference) = 1.20 weight %. Soil and biochar sample codes are defined in Tables 1 and 3; L= biochar added at 0.5 % by weight carbon, H= biochar added at 2.0 % by weight carbon

Application

The final phase of this project focused on demonstrating the utility of the centrifuge method to obtain PAWC data for a large number of samples (i.e., the core matrix of 72 soil-biochar mixtures) and, as a possible consequence, developing further mechanistic insight into the impact of biochar amendments to soils on PAWC. With respect to utility, once the soil-biochar mixes had been prepared, the time needed to collect PAWC data for the complete core matrix dataset (144 experimental units) was on the order of two days to weigh the samples into the centrifuge filter tops, 3 hours to saturate the samples (with a day to equilibrate), 6 hours to load, run, and unload the centrifuge, 3 hours to weigh the samples, 3 hours to enter the data, and 3 hours to perform the calculations. Thus, roughly five days of effort yielded a dataset that would have taken several months to collect by the reference method.

To gain insight into the impact of biochar amendments on PAWC we first looked at the ΔPAWC values obtained, just as for the calibration dataset. When grouped in terms of biochar amendment rate only (Table 5), the mean ΔPAWC for the 0.5% biochar rate was 2.7 wt. % whereas that for the 2.0% biochar rate was 3.5 wt. %. These ΔPAWC means were significantly different (P = 0.05), but not proportional to the amounts of biochar added (i.e., one would expect a roughly 4-fold increase in ΔPAWC in going from the lower rate to the higher rate. Thus, the first insight is that the measured ΔPAWC is not linearly proportional to the amendment rate and most of the benefit seems to be obtained at the lowest rate of addition.

When grouped in terms of soil type alone (Table 5), the ΔPAWC means ranged from a low of 2.0 wt. % for the S3 (Quincy sand) to a high of 3.7 wt. % for S9 (Harstine loamy sand). The S3 was the only soil type that was significantly different from the others. The 7 soil means between the S9 and S3 soils formed a relatively narrow range of 2.7 to 3.4 wt. %; three were not significantly different (P = 0.05) from the S9 soil and the remaining 4 soils were not significantly different (P = 0.05) from each other (the least significant difference among these means was 0.39 wt. %). The ranking of the two coarsest soils (S3 and S9) at opposite ends of the ΔPAWC response spectrum is curious and suggests that, in this instance at least, mineralogy might be more important than texture. The Quincy sand (S3) is of eolian origin and largely derived from basalt, whereas the Harstine loamy sand (S9) is derived from glacial drift with influences from volcanic ash.

Table 5: Means, number of observations contributing to means, non-significant ranges, and least significant differences (P = 0.05) for ΔPAWC values (weight %) obtained for core matrix samples analyzed by centrifuge method

Name	Mean	n	Non-significant Ranges	Least Significant Difference
Overall				
	3.0	144		
Soils				0.39
S1	2.8	16	cd	
S2	3.4	16	ab	
S3	2.0	16	e	
S4	3.3	16	abc	
S5	3.0	16	bcd	
S6	2.7	16	d	
S7	3.4	16	ab	
S8	2.8	16	d	
S9	3.7	16	a	
Biochars				0.26
CP-Wd	2.8	36	b	
SP-Wd	2.5	36	c	
G-Wd	3.4	36	a	
G-Ws	3.3	36	a	
Biochar Levels				0.18
0.5 wt. %	2.7	72	b	
2.0 wt. %	3.3	72	a	

When grouped by both soil type and biochar amendment rate (Figure 10), the least significant difference (P = 0.05) increases to 0.78 wt. % as the number of samples contributing to each mean decreases to 4. A broad spectrum in ΔPAWC values is seen (from 1.4 wt. % for S3 at the 0.5% biochar rate to 3.9 wt. % for S4 and 4.3 wt. % for S9 at the 2.0% biochar rate). None of the remaining means are significantly different (P = 0.05) from each other.

When grouped by biochar type only (Table 5), the two hydrophobic biochars (SP-Wd and CP-Wd) give the lowest mean ΔPAWC values (2.5% and 2.8 wt. %, respectively), whereas the two hydrophilic biochars (G-Wd and G-Ws) yield similar and significantly higher (P = 0.05) mean ΔPAWC values (3.3 wt. % and 3.4 wt. %, respectively). We note that hydrophobic properties are common with fresh biochars and tend to vanish as the biochars age, either in soil or as amendments to compost. These results, therefore, indicate the initial PAWC of the biochar-soil mixtures and underscore the need to be able to measure the PAWC repeatedly and inexpensively over time.

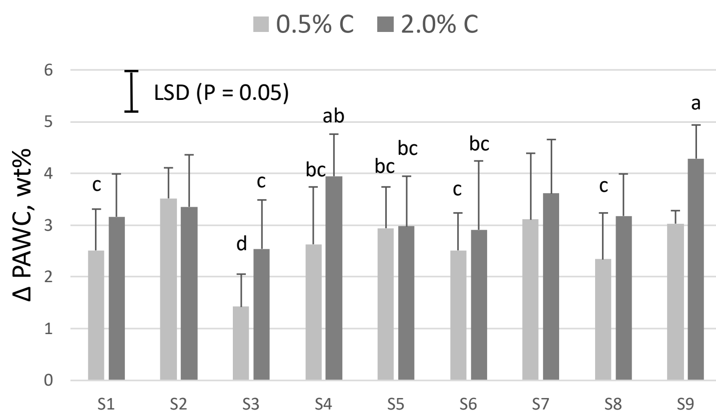

Figure 10: Mean changes in plant-available water-holding capacity (PAWC) measured by the centrifuge method as a function of soil type and biochar amendment rate. Error bars represent 1 standard deviation. Different letters above error bars indicate significant (P=0.05) differences among means. Means having error bars without letters are not significantly different from means labeled with a, b, or c. LSD (least significant difference) = 0.78 weight %

The complete ΔPAWC dataset at the treatment combination level (i.e., means of all 72 treatment combinations) is shown in Figure 11.

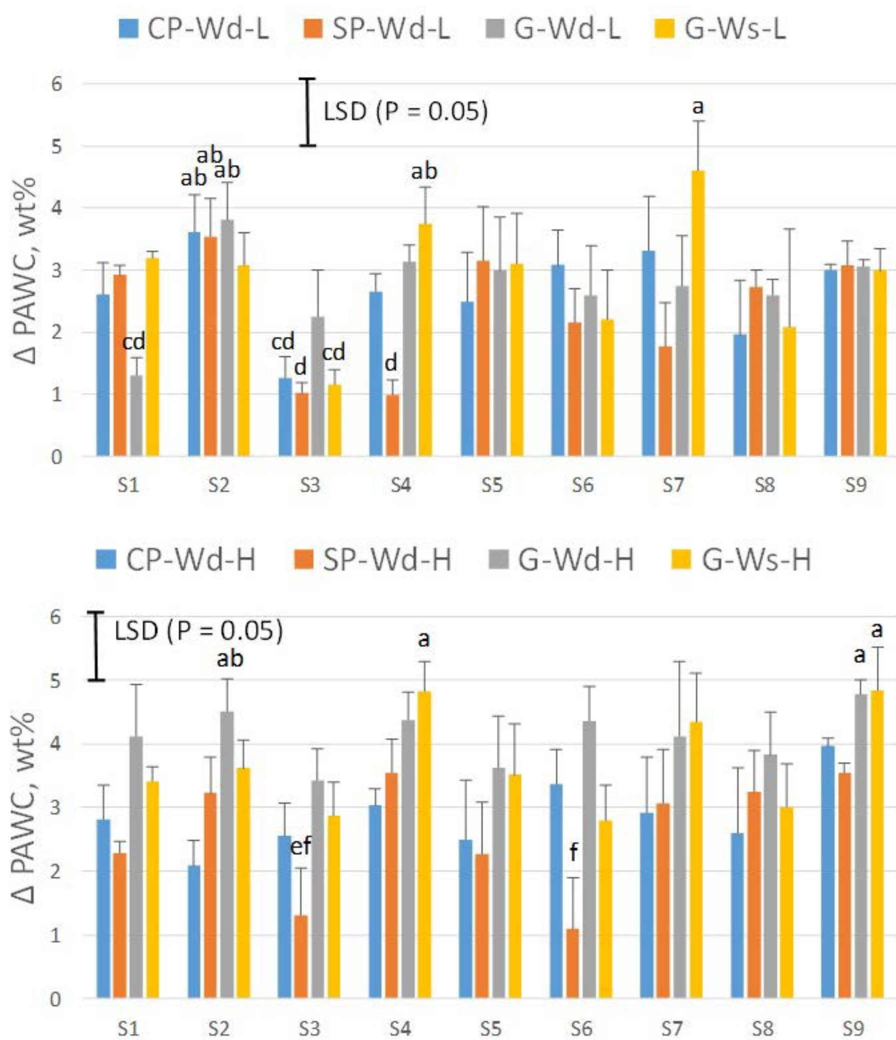

Figure 11: Changes in the PAWC of soils after amendment by different biochars at either 0.5 weight % C (top) or 2.0 weight % C (bottom). Data collected by the centrifuge method. Error bars indicate one standard deviation. Different letters above error bars indicate significant (P=0.05) differences among means. Means having error bars without letters are similar to 2 or more other means (e.g., abc, abcd, or bcdef). LSD (least significant difference) = 1.10 weight %

Having explored the observed ΔPAWC dataset for the soil-biochar mixtures, we next focused on comparing the difference between the observed PAWC value and that for $PAWC_{lc}$, which is the PAWC value predicted from a linear combination of the PAWC values measured for unmixed soil and biochar adjusted by the amounts of each present in the actual mixture analyzed. That is,

$$PAWC_{lc} = f_{soil} \text{ x } PAWC_{soil} + f_{biochar} \text{ x } PAWC_{biochar},$$

where f_{soil} and $f_{biochar}$ are the respective weight fractions of soil and biochar in the soil-biochar mixture, and $PAWC_{soil}$ and $PAWC_{biochar}$ are the respective PAWC values obtained for unmixed soil and unmixed biochar.

If the entire increase in PAWC is due to internal porosity of the biochar added then there should be no difference between PAWC and $PAWC_{lc}$. On the other hand, if inter-particle effects are important, such as creation of new void spaces between biochar and soil particles or physisorption of hydrophobic biochar surfaces to hydrophobic soil surfaces thus decreasing the proportion of hydrophobic surfaces (and increasing the proportion of hydrophilic surfaces) in the mixture relative to the unmixed materials, $PAWC_{lc}$ should be smaller than the actual observed PAWC values. If these inter-particle effects are important, the mixing of biochar with soil could be considered to have a synergetic impact on PAWC.

We found that in every treatment combination, the actual PAWC value exceeded the value for $PAWC_{lc}$. To compare the impacts of soil type, biochar type, and biochar amendment level, on the size of this synergetic effect we calculated the fraction of ΔPAWC not explained by the linear combination approach [i.e., $f_{syn}(\Delta PAWC)$] as:

$$f_{syn}(\Delta PAWC) = (PAWC_{meas} - PAWC_{lc}) / \Delta PAWC$$

where $PAWC_{meas}$ and $PAWC_{lc}$ are the values of PAWC (1) measured on the soil-biochar mixture by the centrifuge method and (2) predicted from a linear combination of unmixed soil and biochar values, respectively, and

$$\Delta PAWC = PAWC_{meas} - PAWC_{soil}$$

where $PAWC_{soil}$ is the value of PAWC measured for the unmixed soil. Because it is a fractional value, $f_{syn}(\Delta PAWC)$ is unit-less.

The overall mean value for $f_{syn}(\Delta PAWC)$ for the core matrix dataset was 74% (Table 6) indicating a very large contribution of factors unrelated to internal biochar porosity to the observed increases in PAWC. A very large significant ($P = 0.05$) difference was seen between the two means for biochar level, with 86% of the effect of the 0.5 wt. % biochar amendment being attributed to this synergy; for the 2.0 wt. % biochar mixtures, the mean level of $f_{syn}(\Delta PAWC)$ fell to 62%, which is still a very significant contribution. Of the nine means for soil type (data not shown), seven ranged between 73% and 80% and were not significantly different from each other; three of these were not significantly different from the 69% value obtained for S8 (Briscot/Kitsap loam). The Quincy sand (S3) was significantly different ($P = 0.05$) from all the other soil types with a relatively low $f_{syn}(\Delta PAWC)$ value of 56%. Further breaking down the $f_{syn}(\Delta PAWC)$ data into means for each soil and biochar level combination (Figure 12), confirms that the dominant effect seems to be the biochar level, with a minor contribution from soil type.

Table 6: Means, number of observations contributing to means, non-significant ranges, and least significant differences (P = 0.05) for $f_{syn}(\Delta PAWC)$ values (% of ΔPAWC) obtained for core matrix samples analyzed by centrifuge method

Name	Mean	n	Non-significant Ranges	Least Significant Difference
Overall				
	74	144		
Soils				5.6
S1	76	16	ab	
S2	78	16	a	
S3	56	16	c	
S4	80	16	a	
S5	80	16	a	
S6	73	16	ab	
S7	80	16	a	
S8	69	16	b	
S9	56	16	c	
Biochars				3.7
CP-Wd	71	36	b	
SP-Wd	96	36	a	
G-Wd	56	36	c	
G-Ws	74	36	b	
Biochar Levels				2.6
0.5 wt. %	86	72	a	
2.0 wt. %	62	72	b	

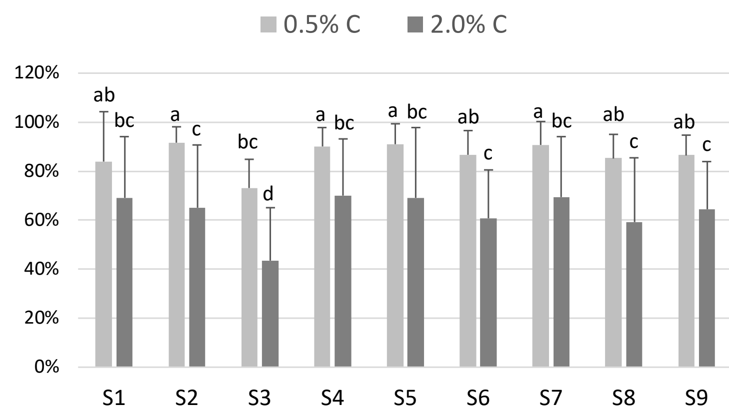

Figure 12: Influence of soil type and biochar level on f_{syn}(ΔPAWC), the fraction of the increase in PAWC that is not predicted by a linear combination of independent data for un-mixed soils and biochars. Data, measured by the centrifuge method, are shown for two levels of biochar amendment to soils. Error bars represent one standard deviation. Different letters above error bars indicate significant (P=0.05) differences among means

Grouping the f_{syn}(ΔPAWC) data by biochar type (Table 6) results in clear effects associated specific biochar types. The SP-Wd biochar yielded a mean f_{syn}(ΔPAWC) value of 96%, suggesting that essentially all of its impact on PAWC is due to inter-particle effects. Of these, the hydrophobic physisorption mechanism may be dominant, given the known hydrophobic properties of this biochar (Table 1). The G-Wd biochar, on the other hand, had a mean f_{syn}(ΔPAWC) value of 56%, consistent with its more hydrophilic nature.

Further grouping the f_{syn}(ΔPAWC) data by biochar type and level (Figure 13), the dominance of inter-particle effects holds for the SP-Wd biochar with little impact of biochar level, whereas for the G-Wd biochar a large drop in mean f_{syn}(ΔPAWC) value occurs in going from 0.5% to 2.0% biochar in the soil mixture. The other two biochars show intermediate behavior, echoing the initial correlation results obtained for the unmixed biochars (Figure 5).

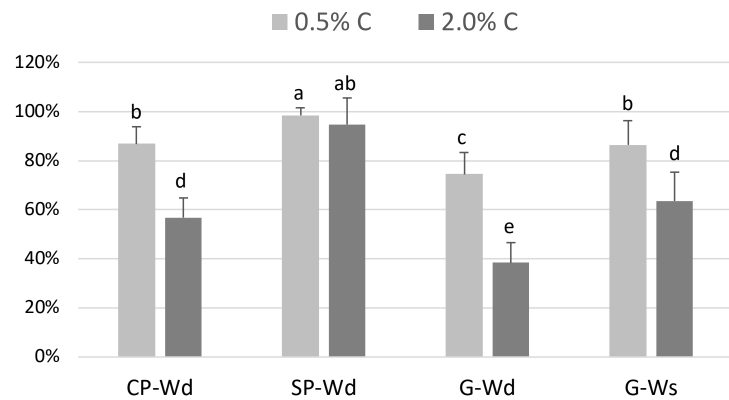

Figure 13: Influence of biochar type and level on f_{syn}(ΔPAWC), the fraction of the increase in plant-available water-holding capacity (PAWC) that is not predicted by a linear combination of independent data for un-mixed soils and biochars. Data, measured by the centrifuge method, are shown for two levels of biochar amendment to soils. Error bars represent one standard deviation. Different letters above error bars indicate significant (P=0.05) differences among means

To summarize, the key insights gained from this first calibrated application of the centrifuge method for PAWC of soil-biochar mixtures are that (1) the contribution of biochar is not linearly proportional to the amount added, (2) in some instances, mineralogical properties of soils may have a large impact on the degree to which biochar increases PAWC, and (3) inter-particle effects represent a large, if not dominant, fraction of the overall impact of biochar on PAWC. Repeated application of this method can track changes in these properties over time as biochar ages.

Conclusions

The centrifuge method is a useful technique to determine the PAWC and allows rapid screening of different soils and biochar types. The calibration dataset using soils of different textures and different types of biochars against measurements made with standard pressure-plate and dew-point-psychrometer methods showed high linearity and a good coefficient of correlation between centrifuge and standard methods. The results we obtained by application of the calibrated centrifuge method to a large set of samples lead to the following conclusions regarding the effects of biochar amendments on the PAWC of soils: (1) biochar increases the PAWC of soils, but the contribution of biochar is not linearly proportional to the amount of biochar added, (2) soil texture, and possibly soil mineralogy, in some instances, have a large impact on the degree to which biochar increases PAWC, with sandy soils receiving proportionally the most benefit from biochar applications, (3) inter-particle effects are the largest contributor to the overall impact of biochar on PAWC. An important question, which is not addressed in this study, is how long the benefits of biochar applications on PAWC will last. As biochar ages in soil, the effects on PAWC will change, and it is unknown whether the biochar benefits on soil water will be short- or long-lasting. With development of the centrifuge method, however, measurements of

PAWC are more readily performed, and monitoring changes in PAWC over time will become accessible to more biochar testing programs.

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