

ABSTRACT

JUDD, LESLEY ANN. Physical and Chemical Analyses of Two Biochars Produced from Pine Wood Chips and Rice Hulls and Their Effects on Container Substrates. (Under the direction of Dr. Brian E. Jackson and Dr. William C. Fonteno).

The use of biochar as a soil amendment has garnered much attention due to its alleged potential in increasing plant growth, soil fertility, soil water holding capacity and carbon sequestration. However, the available literature on this topic is mixed, and suggests that biochar can have either a negative, positive or neutral effect on plant growth in soils and greenhouse substrates. What does this mean and why is it happening? One of the major issues presented from the literature is that biochar is a term given to any charred organic material, whether made from poultry litter or palm trees. Each feedstock could produce a different biochar product physically and chemically. Also, the processes to produce biochar can range from small dirt kilns to large industrial reactors. Certain parameters of the biochar process need to be examined in order to produce biochar consistently. All types of biochar should be characterized for their physical and chemical attributes. For the nursery and greenhouse industry, biochar has several potential uses in substrates that need further investigation. To investigate these potentials, biochar was produced at NC State using a top-lit updraft gasifier with pine wood chips and rice hulls as feedstocks. The biochars were produced in three replicated batches in order to determine the consistency of the gasifier and charring parameters. The biochars were tested for their physical properties, including particle size distribution and porosity, and chemical properties such as cation and anion exchange capacity, elemental composition and available nutrient concentrations. All batches of biochars were consistent in particle size distribution, pH, water/acid extracted nutrients and total elemental composition. All biochars were basic in pH, and the rice hull biochar had

similar pH buffering compared to the feedstock material. Cation exchange capacities were similar or greater than the original feedstocks, however anion exchange capacity was greater for the rice hull biochar. Charring increased plant available potassium (K) from both biochars. However, the acid extractions for total elemental analysis and Mehlich-III analysis produced values hundreds of times higher than the same nutrients measured in the water extracts. Using Mehlich-III test would grossly overestimate the contribution of biochar to the plant available nutrients. These biochars were then used as amendments to peat for comparison to peat:perlite substrates by their physical and chemical properties, as well as the biochars' effect on pH of peat substrates. These biochars match perlite in total porosity and air space, due to particle size, while having increased container capacity to hold more water for container-grown plants. Levels of phosphorus (P) and K rose 1.5 to 10 fold with additions of pine wood biochar and rice hull biochar, respectively. Both biochars had high pH values (8.8-9.5) but when amended to peat as an aggregate, had little direct effect on substrate pH. Even at a smaller particle size (<2.0-mm), these biochars still had little effect on peat pH with rates as high as 14.25 kg·m³. However, when peat is amended with these biochars as an aggregate (at 10%-30% v/v) and dolomitic lime is added from rates of 1.19-7.13 kg·m³, biochar can enhance the effect of lime on pH in fallow containers. From the results of these studies, the two feedstocks produced profoundly different biochars that would impact substrates differently, but the biochars were consistently produced and characterized using a consistent feedstock (especially particle size) as well as consistent production parameters.

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Physical and Chemical Analyses of Two Biochars Produced from Pine Wood Chips and Rice
Hulls and Their Effects on Container Substrates

by
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DEDICATION

I dedicate this work to my grandparents, Herb & Joan Judd and Leslie & Dorothy Warren.

My grandparents spent their lives working hard on farms, instilling a love of land and agriculture in my parents and then on in me. This expanse of work is just a portion of how much you mean to me.

“I had rather be on my farm than be emperor of the world.”

- George Washington

BIOGRAPHY

Lesley Ann Judd grew up in Mason, Michigan and graduated from Mason High School in June 2006. Lesley's family have always been Spartan fans and in August 2006, Lesley moved on to campus and joined her brother at Michigan State University. Lesley started as a freshman majoring in Mathematics with plans to be a high school math teacher. Lesley worked at a local garden center during high school and college and after careful consideration, decided her main passion was for plants and switched her degree to Horticulture. Lesley graduated from Michigan State University in December 2010, receiving a Bachelor of Science degree in Horticulture with a specialization in Landscape Design. After several different jobs in the horticulture industry, Lesley realized she liked the science aspect of Horticulture more than designing landscapes and decided to pursue a Master's degree. In May 2013, Lesley graduated with a M.S. from North Carolina State University and her experiences during her M.S. not only increased her love for horticulture but lead her to stay at NC State to continue graduate studies and pursue a Doctor of Philosophy degree in Horticultural Science. Following the completion of her PhD in 2016, Lesley will continue to promote horticulture and learning at Eastern New Mexico University as an Assistant Professor.

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“Promise me you’ll always remember: You’re braver than you believe, stronger than you seem, and smarter than you think.” – A.A. Milne (Winnie the Pooh)

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CHAPTER 1: Literature Review

History of biochar

The term 'biochar' was first used to describe charcoal used as a soil amendment for agriculture in 2005 by Peter Read from New Zealand (Bates, 2010), however the use of charcoal as a soil amendment was not a new idea at this time because Amazonian Dark Earths were discovered during the 19th century (WinklerPrins, 2014). The dark-colored soil found around the Amazon Basin was not from naturally occurring accumulation of organic matter, but was the result of human activity and termed *terra preta*. These *terra preta* soils contained high levels of stable soil organic matter and were high in phosphorus (P), nitrogen (N), calcium (Ca), magnesium (Mg) and especially carbon (C), with total C stored in these soils 100 to 200 times greater than adjacent soils (Bates, 2010; WinklerPrins, 2014). Researchers found that the carbon in the *terra preta* soils were aromatic carbon that was a likely consequence of the incorporation of charcoal into the soil, not caused by natural forest fires, but due to slow, cooler burns associated with the use of fire as a management tool in sedentary agriculture, such as slash and char, that the early Amazonian cultures could have employed (WinklerPrins, 2014). When this char was mixed into the native Oxisols, it converted these relatively poor soils into rich soils capable of producing food for local communities. Biochar addition to soil has been demonstrated to increase soil pH (Mikan et al., 1996), water holding capacity (Karhu et al. 2011), reduce bulk density (Case et al., 2012), contribute to an increase in cation exchange capacity (CEC; Glaser et al., 2002; Liang et al., 2006; Mao et al., 2012), and improve mycorrhizal response in soils (Warnock et al. 2007). Thus, biochar addition to soil can provide many benefits which contribute to soil fertility

with long lasting effects as the *terra preta* soils continue to be fertile some 800 years after charcoal application (Glaser et al., 2001; Glaser, 2007; Lehmann et al., 2003).

Recent studies have determined that many soils throughout the world contain charcoal and various authors refer to it as carbon black or simply biochar. This charcoal is from natural wildfire (Kuhlbusch, 1998; Rodionov et al., 2010; Schmidt and Noack, 2000) or anthropogenic origin (Cunha et al., 2009). Nevertheless, charcoal, carbon black, and biochar are a significant part of the carbon (C) pool in soil (Fang et al., 2010; Skjemstad et al., 1999; Solomon et al., 2007) and persist due to resistance to oxidation and hence are recognized as a means of potentially modifying soil (Laird, 2008) with long term effects and of sequestering C, which may help to mitigate climate change (Day et al., 2004; Fowles, 2007).

Currently, the term biochar refers to any charred material made from renewable and sustainable biomass under anaerobic conditions (e.g. pyrolysis or gasification; Spokas et al., 2012) made for the purpose of an amendment to improve soil or substrate properties and/or increase plant growth (Lehmann and Joseph, 2009; Schulz et al., 2013). This definition may evolve and serves as a starting point for future development (Lehmann and Joseph, 2009). It encompasses a wide variety of materials as well as multiple methods of production, such as gasification or pyrolysis. An entire range of organic materials can be charred, including poultry litter and sewage sludge, and the resulting biochar will have trace minerals and a pH that reflects the differences of original feedstock (Bates, 2010). Differences can also occur from the production method and parameters set, including temperature, air flow and residence time. There is a lack of consistency over the terminology (Spokas et al., 2012), as well as a lack of clear-cut boundaries according to physical and chemical properties (Glaser

et al., 2002). Biochar materials are diverse and can have varying properties that are dependent on the nature and particle size of the original feedstock, pyrolysis conditions, and their storage or other post-production processes applied (Spokas et al., 2012).

Biochar production

Pyrolysis refers to the process of thermochemical decomposition of organic material at elevated temperatures in an oxygen-free or oxygen-limiting atmosphere (Bridgewater, 1994). During the pyrolysis process, the natural constituents of the biomass (i.e. lignin, cellulose, and starches) are thermally broken down into three different fractions: bio-oil (condensed vapors), char (solids), and non-condensable gases (Boyette et al., 2012; Mohan et al., 2006). Therefore, the biomass undergoes a variety of physical, chemical and molecular changes with pyrolysis, including alterations in carbon : nitrogen, oxygen : carbon, and hydrogen : carbon ratios, porosity, surface area, CEC, crystallinity, and functional groups (Kloss et al., 2012).

There are three main methods to produce biochar: pyrolysis, torrefaction or gasification. Usually, classification of pyrolysis processes is according to the heating rate applied to the biomass particle in order to reach the intended pyrolysis temperature, creating a distinction between fast and slow pyrolysis (Ronsse, 2013). Fast pyrolysis involves high heating temperatures (~500°C), low vapor residence times and oxygen-free; these process conditions are selected to maximize the yield of the bio-oil fraction which may serve as a liquid fuel or be further upgraded into biofuels (Ronsse, 2013). Slow pyrolysis is characterized by lower heating levels (> 400°C) and oxygen-free or oxygen-limiting environments, which result in maximum yields in biochar (Ronsse, 2013). Gasification is the

process where the primary aim of pyrolysis is the production of a flammable gas, not char (Boyette et al., 2012). The design of a gasifier and its operating parameters may be adjusted to produce more gas or more char as desired. Gasification uses the highest temperatures compared to the other methods (600 - 1800°C) in an oxygen-limited environment (Ronsse, 2013). Torrefaction is a mild pre-treatment of biomass at a temperature between 200 - 300°C, where the properties of biomass are changed to obtain a much better fuel quality for combustion and gasification applications (Bergman and Kiel, 2005). Torrefaction has lower heating rates and an oxygen-free environment with the greatest yield (by weight) being the char/solid product (Ronsse, 2013). Another method is hydrothermal carbonization (HTC) which uses water at high temperatures (up to 250°C) and high pressures (up to 35 bar) to convert biomass into a char-like product often termed 'hydrochar' (Ronsse, 2013). The macromolecular breakdown and dehydration reactions inherent in HTC cause a different biochar product than the other methods mentioned.

These different methods can either be a batch system or a continuous operation. A batch system involves having a reactor that is operated in a sequence where it is fully loaded with fresh biomass, then heated after which pyrolysis/gasification takes place. Once the reactor is cooled down, the remaining char is unloaded. Batch operation is simple to implement, but has a number of drawbacks if compared to continuous systems, such as product heterogeneity may exist between different batches (Ronsse, 2013). A continuous operation allows for fresh biomass to be transported through a heated reactor zone, which allows for proper control of biomass residence time, then pyrolysis occurs and char is

produced, moved through the system and allowed to cool before leaving the whole system (Ronsse, 2013).

The consumption of agricultural wastes and other biomasses to produce biochar is environmentally beneficial due to pyrolysis rendering the carbon in the feedstock inert, which significantly reduces the amount of carbon dioxide released into the atmosphere compared to the autotrophic respiration that would occur if the biomass were allowed to decompose (Blackwell et al., 2009; Boyette et al., 2012). There are a lot of process parameters that affect the end-product of biochar, including feedstock, temperature, time of pyrolysis, and particle size. These factors in turn affect the physical and chemical properties of biochar and how the biochar responds in soil or soilless substrates.

Biochar in soils

The influence of biochar in mineral soils has been studied and reviewed extensively, as biochar has been applied to soils for hundreds of years. From early civilizations using fire pits built on soil to the early 1600s in Japan and China where biochar was reportedly used in agriculture (Spokas et al., 2012). Some of the most commonly cited beneficial impacts of biochar are improved crop growth in sandy soils (Lehmann et al., 2003; Novak et al., 2009; Schulz et al., 2013; Prendergast-Miller et al., 2014), increased soil pH (Novak et al., 2009; Ahmad et al., 2012), increased water retention (Abel et al., 2013), alleviated *Phytophthora* spp. or peach replant disease progression and physiological stress (Zwart and Kim, 2012; Atucha and Litus, 2015), induced systemic resistance to foliar fungal pathogens (Elad et al., 2010), increased mycorrhizal associations (Warnock et al., 2007), and improved nutrient retention (Knowles et al., 2011; Clough and Condon, 2010). In 2011, Jeffery et al.

compared 177 individual studies that revealed the beneficial effects of biochar amended to soils outbalance the negative (decreased plant growth/yield) and neutral effects (no effect on plant growth or yield); an average increase of crop productivity of around 10% was noted with the utilization of biochar.

Some of these effects on properties such as soil nutrition, water retention or microbial activity can be immediate, but vary depending on soil type. Biochar can pose other long-term effects as well, it could potentially influence soil-forming processes that govern the accumulation and translocation of soil constituents (Richter, 2007). However, for biochar to serve a beneficial role in revitalizing nutrient-poor soils, there should be a noted increase in the quantity of plant available nutrients and effects on soil microbial communities (Spokas et al., 2012). Research has suggested that soil applications of biochar can provide equivalent amounts of nitrogen, phosphorus and potassium (K) as fertilizers, as well as have a significant impact on microbial carbon metabolism and population dynamics (Spokas et al., 2012; Warnock et al., 2007).

Biochar in substrates

Potential exists for horticultural use of biochar in soilless substrates used for container production of greenhouse crops (Gu et al., 2013; Northup, 2013; Peterson and Jackson, 2014; Vaughn et al., 2015b), although the influence of biochars on soilless substrates used in greenhouse and nursery container substrates has been studied less than in soils (Altland and Locke, 2012; Steiner and Harttung, 2014). The first reported use of a product similar to biochar was by Santiago and Santiago (1989), who recommended charcoal as a container

substrate for potting mix because it acts as a sponge absorbing and retaining water, gases and solutions.

More recently, researchers have investigated amending biochars to types of container substrates and its effects on plants. A pot experiment with soil collected in Cambodia found that rice-husk biochar can increase both plant and root biomass of lettuce (*Lactuca sativa*) and Chinese cabbage (*Brassica chinensis*) plants (Carter et al., 2013). Another alternative substrate, coconut fiber, was amended with wood-derived biochar and was shown to increase plant growth and yield of pepper (*Capsicum annuum*) plants and increase the plant height and leaf size of tomato (*Solanum lycopersicum*) plants (Graber et al., 2010). Red oak biochar amended to peat or peat-vermiculite substrates has been found to increase shoot biomass with hybrid poplar cuttings (Headlee et al., 2014).

Biochar can also impact the physical and chemical environment. Dumroese et al. (2011) reported that amending peat with biochar produced from agricultural/forestry residue could improve substrate hydraulic conductivity and water retention, as well as increase the amount of water available at permanent wilting point within the amended substrate. Cao et al. (2014) reported similar findings using a different substrate: urban green waste-derived biochar amended to scoria green roof substrates. Beck et al. (2011) also investigated green roof substrates and used biochar produced from 70% agricultural waste (rice hulls, pecan shells, walnut shells and coconut shells) and 30% rubber car tires. The researchers reported this particular substrate decreased N, P and organic C in runoff. Nemati et al. (2015) reported similar findings of decreased nutrients in runoff collected from hardwood and softwood bark biochar amended to peat and peat-perlite substrates. Reports of red oak biochar have shown

significantly higher cation exchange capacity (CEC) and exchangeable nutrients when amended to peat and peat-vermiculite substrates (Headlee et al., 2014) and biochar produced from urban greenwaste has shown inherent nutrient content (Kaudal et al., 2015). Different biochars can have ranges of pHs, from 5.4 to 10.3 (Nemati et al., 2015; Fornes et al., 2015; Spokas et al., 2012) and may have the potential to neutralize acidity caused by both peat and root activity (Bedussi et al., 2015). Gasified rice hulls amended to a commercial soilless mix of peat and perlite have shown potential for increased available phosphate and K, as well as potential for decreasing nitrate leaching from substrates (Altland and Locke, 2013a; Altland and Locke, 2013b). This may be due to the anion exchange capacity (AEC) of biochar, although the mechanism is unknown (Lawrinenko, 2014).

There are several global advantages to using biochar in soilless substrates, including decreasing the use of peat for those environmentally conscious, using local and sustainable feedstock materials for lower cost and marketing purposes, and using the other products of gasification such as biofuel or biodiesel. Lehmann and Joseph (2009) state: biochar provides a unique opportunity to improve root zone fertility and nutrient-use efficiency using locally available and renewable materials in a sustainable way. These are similar reasons for the use of other alternative substrate components as well, however most alternatives have low structural stability and cause nitrogen immobilization, contain too many nutrients (e.g. compost) or have a low water holding capacity (Reinhofer et al., 2004; Steiner and Harttung, 2014). Using biochar in substrates and reporting any improvements to plant growth or substrate properties potentially increases the value biochar by promoting its usage while also creating an opportunity for carbon sequestration (Dumroese et al., 2011).

Current issues with biochar

Even with all the reported benefits of biochar, some issues still remain. When reviewing the literature regarding the physical and chemical characterization of biochar, care should be taken not only because experimental conditions are highly variable, but also because the details are not always sufficiently reported (Table 1; Downie et al., 2009). These variable properties are due to the net result of production (e.g. feedstock and pyrolysis conditions) and postproduction factors such as storage or activation (Spokas et al., 2012). Reviewing scientific literature surrounding biochar in soilless substrates, details including feedstock source or production method have been left out, adding to the confusion of what defines “biochar” and if the results can be compounded to the whole population of “biochars” (Table 1). While many various studies have reported improvements to plants and root environment, there are just as many reports showing neutral or decreased improvement to plant growth in biochar amended soils and substrates (Cao et al., 2014; Conversa et al., 2015; Gu et al., 2013; Steiner and Harttung, 2014; Vaughn et al., 2015a), creating a summary of reports showing inconsistent benefits of biochar (Spokas et al., 2012).

The inconsistencies of biochar are similar to the situation with compost when used as an alternative substrate for greenhouses/nurseries. Compost was deemed to have many benefits, such as increased germination and plant height of vegetable plants and increased nitrogen for plant use (Sterrett, 2000). However, not every compost produced had the same findings and did not always have positive impacts on plants (i.e. little or no improved plant

growth). This was due to the initial feedstock material, composting process/handling and storage (Sterrett, 2000). Rosen et al. (1993) reported that the primary limiting factor for the general use of municipal solid waste compost in horticultural crop production at that time was the lack of consistent, high-quality compost. This can also be said for biochar, as each feedstock creates a unique biochar which is affected by production methods, handling and storage. Future research will need to explore the impact of the vast range of biochar properties on their potential use in greenhouse and nursery container production (Altland and Locke, 2012).

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Table 1. Comparison of results from biochar (types and production) when amended to or compared against soilless substrates, reported in scientific literature.

Year	Author	Feedstock	Production and temperature	Substrate base or comparison	Results
1989	Santiago & Santiago	Wood	Kiln	Charcoal chips versus charcoal chunks	Charcoal chips act as a sponge absorbing and retaining water, gases and solutions
2002	Islam et al.	Carbonated rice husks	-- ^z	Coconut coir	Increased yield of tomatoes (<i>Lycopersicon esculentum</i>)
2010	Graber et al.	Citrus wood	Traditional charcoal pit	Coconut fiber and tuff	Increased leaf area, buds, flowers and fruit of pepper (<i>Capsicum annuum</i>) and increased plant height of tomato but no effect on yield
2011	Beck et al.	Agricultural and rubber waste	Pyrolysis	Pro-Gro extensive green roof media	Increased water retention and decreased discharge of N, P, and organic C
2011	Dumroese et al.	Agricultural waste	Pyrolysis	Peat moss (PM)	25% pelletized biochar-wood flour recommended for enhanced hydraulic conductivity
2013	Altland & Locke	Rice hulls	Gasified	PM, perlite, dolomitic limestone and gypsum	Increased shoot dry weight of geranium (<i>Pelagonium x hortorum</i>) in 10% biochar with ammonium nitrate and micronutrient fertilizers
2013	Gu et al.	Pine wood	Flash pyrolysis	Pine bark (PB) or PM with perlite	No interaction between substrate and biochar on plant width of gomphrena, and 20%-25% biochar in PB increased plant height
2013	Northup	Hardwood	--	PM	Biochar had no effect or decreased plant biomass of pepper, tomato, cucumber (<i>Cucumis sativus</i>), marigold (<i>Tagetes patula</i>) and petunia (<i>Petunia x hybrida</i>) compared to PM. Only impatiens (<i>Impatiens walleriana</i>) had equal or greater plant biomass than PM

Table 1 continued.

2014	Cao et al.	Urban green waste	Continuous-flow slow pyrolysis at 550°C	Scoria-based green roof substrates	Improved water holding capacity and increased plant available water. Biochar did not affect plant growth of wheat (<i>Triticum aestivum</i>)
2014	Carpenter	Hardwood	--	PM-based (Jiffy Mix [®])	Had no effect or increased germination of pepper seed depending on cell tray size
2014	Headlee et al.	Red oak	Pilot scale bubbling fluidized bed fast pyrolysis at 500°C	PM and vermiculite	Biochar mix was associated with lower root biomass and higher shoot/root ratio, poplar (<i>Populus</i> spp.) trees had higher concentrations of K in shoots
2014	Zhang et al.	Coconut husk fiber	Pyrolysis at 450°C	Composted green waste	Increased macro- and micro-nutrient contents, and 20% biochar improved plant growth of <i>Calathea insignis</i>
2015	Bedussi et al.	Poplar and spruce	Gasification	PM	Poplar biochar more efficient in neutralizing peat acidity and both biochars increased water soluble K
2015	Copley et al.	Maple bark	Pyrolysis at 700°C for 4 h	AgroMix G10 (Fafard Ltd.)	Increased Rhizoctonia damping-off severity in 11 plant species tested
2015	Conversa et al.	Whole fir tree (<i>Abies alba</i>)	Pyro-gasification at $\geq 1000^\circ\text{C}$	PM – 1:1 mixture of light and dark peat	30% biochar did not affect geranium growth but increased N and chlorophyll leaf concentrations, leaf and flower number
2015	Dunlop et al.	Tomato crop green waste, including stem clips and string	Pyrolysis by wood-fired kiln at 550°C	Pine sawdust (<i>Pinus radiata</i>)	Biochar did not affect plant growth of tomato, yield or fruit quality compared to plants grown in pine sawdust

Table 1 continued.

2015	Fornes et al.	Forest waste (FW) or olive mill waste (OW)	FW purchased as waste from pyrolytic charring in traditional kiln of hardwoods for barbeque purposes. OW purchased as waste from firebrick oven at 500°C for 3 h, used to extract oil	Coconut coir fibre	Forest waste biochar was poor in nutrients and olive mill waste biochar was saline and rich in soluble cations
2015	Kaudal et al.	Urban biosolids and green waste	Continuous pyrolyzer at 650°C for 40 min.	Growing media	Biochar was highly porous and nutrient rich with high salts compared to potting substrate
2015	Mendez et al.	Deinking sludge	Pyrolysis by placing feedstock in steel cup and placing in electric furnace for 2 h, at max. 300°C	Brown PM and coconut coir	Biochar increased air space, water holding capacity and total porosity of PM but not for coir. Increased lettuce yield in PM substrate
2015	Nemati et al.	Hardwood logs (<i>Acer saccharum</i> ; <i>Betula alleghaniensis</i>), softwood bark (<i>Abies balsamea</i> ; <i>Picea glauca</i> ; <i>Picea mariana</i>) or hardwood waste by-products	Pyrolysis between 350 and 600°C	PM or PM with perlite	Biochar can decrease total porosity, container capacity and available water but increased relative gas diffusivity

Table 1 continued.

2015	Vaughn et al.	Wood pellets, pelletized wheat straw, and pennycress presscake	Top-lit updraft cookstove	1:1 PM and vermiculite	Combining potato anaerobic digestate with acidified wood pellet biochar increased tomato growth, while digestate:acidified wheat straw pellet biochar had no effect on plant growth and digestate:pennycress presscake biochar had less growth
2016	Nieto et al.	Commercial charcoal and two biochar samples of pruning waste	Pyrolysis at 300 or 500°C	PM and pruning waste	Mixtures had lower water holding capacities than PM, and pruning waste biochar amended to peat increased lettuce biomass

²Dashed line represents lack of production method and parameters mentioned.

CHAPTER 2

Physical and Chemical Characterization of Two Biochars Produced by Gasification with Effects of Charring Process and Consistency of Production Method

(In the format appropriate for submission to HortScience)

**Physical and Chemical Characterization of Two Biochars Produced by Gasification
with Effects of Charring Process and Consistency of Production Method¹**

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Physical and Chemical Characterization of Two Biochars Produced by Gasification with Effects of Charring Process and Consistency of Production Method

Additional index words: elemental composition, pine wood chips, plant nutrients, pyrolysis, rice hulls

Abstract. Increasing interest in biochar has caused increased production and research of different feedstocks for use in soils or soilless substrates. Wood chips produced from loblolly pine trees (*Pinus taeda* L.) and harvested rice hulls were used to produce biochars from a top-lit updraft (TLUD) gasifier. The objectives of this study were to 1) to create biochar from two different feedstocks using a TLUD gasifier; 2) to test the consistency of the process by replicating the production of both biochars multiple times; and 3) to ascertain differences in physical and chemical characteristics of biochars relative to their respective feedstocks. The production system used, TLUD gasifier, produced consistent physical and chemical results over the batches for both biochars. The batches of both PBC and RBC were consistent in particle size distribution, pH, water/acid extracted nutrients, and total elemental composition. Measurements of cation and anion exchange capacities for RBC were similar between each run, indicating a consistency of process. All batches of biochars were consistent in particle size distribution, pH, water/acid extracted nutrients and total elemental composition. The acid extractions for total elemental analysis and Mehlich-III analysis produced values hundreds of

times higher than the same nutrients in the water extracts. The Mehlich-III analysis would grossly overestimate the contribution of biochar to the plant available nutrients. Charring increased plant available potassium from both biochars, however the biochars also showed having strong hydrophobicity issues with low hydration efficiencies. From the results of this study, the two feedstocks produced profoundly different biochars that could likely impact substrates differently if incorporated as an amendment, but the biochars can be consistently produced using the TLUD method.

Introduction

Currently, the term biochar refers to any charred material made from renewable and sustainable biomass under anaerobic conditions (e.g. pyrolysis, gasification or torrefaction; Spokas et al., 2012) made for the purpose of an amendment to improve soil or substrate properties and/or increase plant growth (Lehmann and Joseph, 2009; Schulz et al., 2013). A broad range of organic materials can be charred and the resulting biochar will have trace minerals and a pH that reflects the differences of original feedstock (Bates, 2010). Biochar materials are diverse and can have varying properties that are dependent on the nature and particle size of the original feedstock, production method and parameters, and their storage or other post-production processes applied (Spokas et al., 2012). There is a lack of consistency over the terminology (Spokas et al., 2012), as well as a lack of clear-cut standards for physical and chemical properties (Glaser et al., 2002).

There are three main methods to produce biochar: pyrolysis, torrefaction and gasification. Fast pyrolysis involves high heating levels (~500°C), low vapor residence times and oxygen-free; these process conditions are selected to maximize the yield of the bio-oil fraction which may serve as a liquid fuel or be further upgraded into biofuels (Ronsse, 2013). Slow pyrolysis is characterized by lower heating temperatures (~400°C) and oxygen-free or oxygen-limiting conditions, which result in maximum yields in biochar (Ronsse, 2013). Gasification is the process where the primary aim of pyrolysis is the production of a flammable gas, not char (Boyette et al., 2012). The design of a gasifier and its operating parameters may be adjusted to produce more gas or more char as desired. Gasification uses the highest temperatures compared to the other methods (600 - 1800°C) in an oxygen-limited

environment (Ronsse, 2013). A particular design of gasification, termed the top-lit updraft (TLUD) gasifier, has been investigated for the potential to produce biochar and synthesis gas (syngas) from biomass residues and has been found to produce relatively high yields of biochar (Rivas, 2015). Syngas, a mixture of carbon monoxide, carbon dioxide and hydrogen with potential use as a fuel source, is generated using TLUD gasifiers which aids in reducing pollution released to the environment when compared to traditional pyrolysis units (Birzer et al., 2013; Rivas, 2015). Torrefaction is a mild pre-treatment of biomass at a temperature between 200 - 300°C, where the properties of biomass are changed to obtain a much better fuel quality for combustion and gasification applications (Bergman and Kiel, 2005). Torrefaction has lower heating rates and an oxygen-free environment with the greatest yield (by weight) being the char/solid product (Ronsse, 2013).

Today, biochar production has greatly increased, due mostly to the increased use of biomaterials as fuel sources in industrial systems, including the production of bio-oils and gases (Renner, 2007). This increase in production has expanded the availability of different biochars for agricultural and horticultural use. With field soils, the use of biochar as a soil additive has been in practice for hundreds of years internationally, due to the potential benefits of increasing plant growth, yield, and important plant nutrients of poor soils (Spokas et al., 2012). The use of biochar in horticultural substrates has been gaining interest over the last decade, however there are references from as early as 1989 that suggest the use of a similar biochar-type product. The first suggestion of a biochar-type material being used as a horticultural substrate was reported by Santiago and Santiago (1989) recommending charcoal as a substrate for potting mix because it acts as a sponge absorbing and retaining water, gases

and solutions. However, the influence of biochars on soils has been studied markedly more than when amended to soilless substrates used in greenhouse and nursery container substrates (Altland and Locke, 2012; Steiner and Harttung, 2014). The research that has been conducted over the years with biochar and horticultural substrates has produced high variability of effects on plant growth or available nutrients. In some cases, little or no benefit has been reported, as well as decreased improvement to plant growth (Cao et al., 2014; Conversa et al., 2015; Gu et al., 2013; Steiner and Harttung, 2014; Vaughn et al., 2015). These issues seem to stem from the fact that “biochar” has been considered as one material, whereas it is actually many different materials with the same name. Each of these biochars researched have highly variable experimental conditions and feedstocks, and these details are not always sufficiently reported (Downie et al., 2009)

The inconsistencies of biochar are similar to the situation with compost when used in the horticultural industry. Compost was deemed to have many benefits, such as increased germination and plant height of vegetable plants and increased nitrogen for plant use, but individual composts were not the same (Sterrett, 2000). This was due to the initial feedstock material, composting process/handling and storage (Sterrett, 2000). Rosen et al. (1993) reported that the primary limiting factor for the general use of municipal solid waste compost in horticultural crop production at that time was the lack of consistent, high-quality compost. This can also be said for biochar, as each feedstock creates a unique biochar which is affected by production methods, handling and storage.

Therefore, measuring physical and chemical properties of biochar is important to first characterize the biochar made from a particular feedstock and charring process with

parameters, and secondly to characterize how the biochar will affect the substrate/root zone/plant growth. It is important to determine total elemental composition and plant available nutrients to show which nutrients would exchange in the substrate solution as well as the potential to decrease the amount of fertilizer applied to the crops. The cation exchange capacity (CEC) and anion exchange capacity (AEC) of biochar has great potential benefit that would aid in keeping cations and anions important to plant growth in the root zone and would decrease nutrients lost through leaching. If stable anion exchange sites exist on biochar surfaces and are capable of reversibly exchanging anions, then substrates amended with high AEC biochars should exhibit reduced leaching losses of nitrate and phosphate (Lawrinenko, 2014). Other reports have speculated about different biochars having an effect on the moderation of nitrate and phosphate in substrate solution and potentially reducing the amount lost through leaching (Altland and Locke, 2012; Altland and Locke, 2013b). Another benefit of the addition of biochar to peat-based substrates is the potential for neutralizing the peat acidity and stabilizing the pH over time. This would create a better root environment for the plant to grow and possibly decrease the necessity or amount of other additions (e.g. lime, acidic/basic fertilizers) to the crop during production.

The objectives of this study were to 1) to create biochar from two different organic feedstocks; 2) replicate the production of both biochars multiple times to determine consistency of the process; and 3) to physically and chemically characterize each material both before and after charring.

Materials and Methods

Gasifier. The TLUD gasification unit was constructed from a 2.5-m high and 1-m diameter black iron tube (Figure 1A). The flow of air was forced into the gasifier with an air fan/electric motor (Dayton Model #7E253, Grainger, Raleigh, NC) attached at the bottom of the reactor (Figure 1C & 1D). Air flow for all runs was set at $12\text{-m}^3\cdot\text{min}^{-1}$ for uniformity. Weight of the reactor and feedstock was determined with four load cells placed on adjustable jacks and the entire gasifier with frame was lifted so weight rested on scales, in order to determine the weight of the feedstock materials and record weight at the end of charring. The unladen weight of the whole gasifier was tared from the scales so weight of feedstock could be determined from the start, throughout charring process and also at the end of the process to determine the percent of biomass lost for each run, and check for consistency. Internal temperature in the middle of the reactor was recorded with a data logger (Measurement Computing, model: USB-5201, Norton, MA), and external temperature was recorded with a hand-held infrared thermometer (Westward IR Therm #2ZB46; Grainger, Raleigh, NC) to determine speed of the flame front and to ensure the flame front was, in essence, level. Data collected included temperature, residency time, initial and final biomass and percent biomass lost.

A conveyor was used to fill the reactor and the feedstock was leveled every 5 min to ensure even placement of the total 1.5-m^3 of feedstock material loaded in the reactor. The feedstock reached approximately 1.8-m high inside the reactor and was ignited with lighter fluid placed in three concentric circles around the center of the reactor. Lit pieces of cardboard were then placed atop the feedstock to ignite the lighter fluid at the top of the

reactor, then the lid was quickly placed on top and sealed to control the gasification of the material. Combustion and speed of burn were sustained by regulating the amount of air entering from the bottom and passing up through the material ($12\text{-m}^3\cdot\text{min}^{-1}$), air flow was calculated by area of fan inlet and air velocity, using a handheld air velocity meter (Turbo Meter Wind Speed Indicator, Davis Instruments, Hayward, CA). A vent at the top of the reactor allowed combustible gas from the process to leave the system (Fig. 1B). This gas was flared (ignited) to reduce the amount of smoke and odor produced. The location and speed of the flame front was monitored on the exterior of the chamber with the infrared thermometer. Once the flame front reached the bottom of the gasifier, the air flow was shut off and compressed nitrogen gas was then fed through from the bottom for 24 h to prevent any flare up as the biochar cooled. Once cooled the pine wood biochar (PBC) or rice biochar (RBC) were removed from the reactor and stored in 1.5-m^3 industrial bags under shelter.

Biochar feedstocks. Two feedstocks were obtained and processed into biochar for evaluation in these studies. Pine wood chips (PWC; loblolly pine; *Pinus taeda* L.) were purchased from a local supplier (West Farms Wood Products LLC, Newton Grove, NC) who had processed the chips to 6.35-mm size (Fig. 2A). Wood chips were stored in three 1.5-m^3 bags on pallets under shelter for protection from the weather. The second feedstock, non-parboiled rice hulls (RH; Fig. 2B; Riceland Food Inc., Stuttgart, AR), were purchased in compressed bales (4.5-m^3) and stored on pallets under shelter. The compressed rice hulls were de-compacted by loosening and placing the rice hulls in 0.03-m^3 boxes in order to measure out 1.5-m^3 for each batch processed in the gasifier.

Processing and sampling. Both feedstocks were processed through the TLUD gasifier in three separate batches each in order to determine consistency of the gasifier/parameters. Each batch of pine wood chip feedstock had a moisture content of 14% while every batch of the rice hulls had a moisture content of 12%. A portion of both feedstocks, PWC and RH, were reserved to test chemical and physical properties for comparison to the biochar produced.

Sampling procedures for analyses were as follows: feedstock was collected from the top of the storage bag, the bottom of the bag when the material was lifted above the conveyor belt and lastly from the middle of the bag. This was repeated for each batch before the charring process. Once the biochar was produced, it was placed into new storage bags. Samples were collected within 30 to 60 days after production by gently and completely rolling the bags a full 360° five times to undo any particle settling and three 0.15-m³ samples were collected from the top, middle and bottom of the bags and placed in individual plastic bags (#5DTW5 H28MET; Grainger, Raleigh, NC).

Physical properties. To determine particle size distribution of the biochar batches and feedstock material, three samples of each aggregate from each batch run were dried at 105° C for 48 h and placed in a Ro-tap Shaker (Model B, W.S. Tyler, Mentor, OH) fitted with six sieves; 6.3 mm, 2mm, 0.71 mm, 0.5 mm, 0.25 mm, and 0.106 mm for five min. The sample from each sieve was weighed, and particle size was expressed as a percentage of the total weight of the sample.

The feedstock materials of either PWC or RH had the same moisture content for each of the three batches, respectively, before being placed in the gasifier and charred. The

resulting biochar was also at the same moisture content after the charring process. Due to the consistency of the particle sizes for each batch (discussed later), samples for each PBC batches were mixed together, as well as samples for each RBC batch, for testing wettability and physical properties.

Hydration efficiency measurements were conducted following the procedures described by Fonteno et al. (2013) and Fields et al. (2014). Three samples of PWC and RH were tested at a moisture content (MC) of 12% (RH) or 14% (PWC); these samples were removed from the bulk feedstock before the charring process. Three samples of PBC and RBC were tested at a moisture content of 8%; these samples were removed from the reactor after the charring process was completed. Each material had its hydration efficiency described with wettability curves as determined by Fields et al. (2014). A measure of initial hydration was the percentage of container capacity (CC) that was attained in a sample after one hydration event.

Physical properties including air space (AS), CC, total porosity (TP) and bulk density (BD) were determined for each original feedstock and biochar type, using the North Carolina State University Porometer method (Fonteno, 1996). Properties were determined using three representative samples of each material, with samples pulled from a mixture of all batches since particle size distribution was not different between the batches.

Chemical properties. Three samples of PWC, RH, WBC and RBC were tested for pH to determine the effects of charring. pH and electrical conductivity (EC) was measured with a pH/EC meter (HI 9811, Hanna Instruments, Ann Arbor, MI) using the 1:1 dilution method; one part material (100 mL) was mixed vigorously with one part deionized (DI) water and

allowed to equilibrate for 15 min (Lang, 1996). Another six samples from the biochar materials were tested for pH buffering capacity. Three of these samples were tested as is, the other three were placed in separate containers to hydrate the particles. Particles were hydrated by placing biochar in cups with degassed DI water and placed inside a vacuum for 15 min. All samples (1.5 g equivalent dry weight) were diluted to 50 g with degassed DI water and were titrated with 0.1 N hydrochloric acid (HCl) or 0.1 N sodium hydroxide (NaOH) using an automatic titrator (Titralab[®] 856, Radiometer Analytical SAS, Villeurbanne Cedex, France). The feedstock or biochar suspensions were taken through two titration cycles between pH 3 and 11; the first cycle aimed to hydrate the material and expose cation exchange sites and involved increasing the pH from its ambient level near pH 9 up to pH 11 and then decreasing the pH to 3. The second cycle was used to generate the pH buffering curve. The parameters of the titration process follow the outline proposed by Barnes (2013).

Samples of PWC, RH, WBC and RBC were submitted to the North Carolina Department of Agriculture and Consumer Services (NCDA), Agronomic Division (Raleigh, NC) for nutrient analysis. Plant available nutrients were determined at NCDA by saturated media extract and vacuum filtration, the solution was analyzed for NO₃-N by nitrate-hydrazine reduction (Kempers, 1988); NH₄-N was determined by a modified Berthelot reaction (Krom, 1980); and urea concentration was determined with the diacetyl monoxime thiosemicarbazide colorimetric method (Sullivan and Havlin, 1991) with an auto-flow spectrophotometric analyzer (San++ Segmented Flow Auto-Analyzer, Skalar Instruments; Breda, The Netherlands). Total concentrations of phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron

(B), and sodium (Na) were determined from the solution of saturated media extract with Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES; Optima 8000 PerkinElmer, Waltham, MA). Total exchangeable nutrients were determined at NCDA using Mehlich-III extraction [reagents include ammonium nitrate, ammonium fluoride, acetic acid, nitric acid and ethylenediaminetetraacetic acid (EDTA); Mehlich, 1984]. Samples were placed in bottles and mixed with Mehlich-III extractant, then shaken for 5 min and vacuum filtered. The solution was then analyzed by molybdate blue colorimetric for P, flame emission for K and atomic absorption for Ca, Mg, Mn, Zn and Cu (Mehlich, 1984).

Total elemental composition and total C, N, and H were determined by the Environmental and Agricultural Testing Service lab at NC State University, on both feedstock and biochar materials after being ground to a particle size of <2-mm. Total elemental composition was determined by dry ash procedure (P, K, Ca, Mg, Cu, Fe, Mn and Zn) and wet digestion method (B, Mo, S, Si) and ICP-OES was used to determine elemental concentrations. Total elemental C, N, and H was determined by total combustion method in the CHNS/O Analyzer (2400 Series II PerkinElmer, Waltham, MA).

The CEC of the biochars and original feedstocks were measured based on procedures described by Kloss et al. (2012) and Dumroese et al. (2011). Initially, 2 g of biochar and 40 mL of degassed DI water were mixed and allowed to saturate for 12 h. Samples were then vacuum filtered (Whatman[®] Qualitative #1) and the biochar was placed in a flask with 20 mL of 0.2 M barium chloride (BaCl₂) and mixed on an oscillating shaker (G10 Gyrotory[®] Shaker; New Brunswick Scientific Co., Edison, NJ) for 2 h. The final solution was filtered with 0.45- μ m nylon filters (25mm syringe filters; Fisher Scientific, Waltham, MA). The

concentrations of Na, K, Mg, Ca, Al, Fe and Mn were measured via ICP-OES (Perkin Elmer 8000, Waltham, MA). The AEC of the biochar samples was determined according to the procedure described by Lawrinenko (2014). One gram samples of biochars and feedstocks were mixed with 40 mL of degassed DI water and 2 mL of 1 M potassium bromide (KBr) and placed on an oscillating shaker for 48 h. Biochar/feedstock residue was then vacuum filtered and rinsed with DI water two or three times until conductivity of the solution was $\leq 5\mu\text{S}$ (HI 9813-6, Hanna Instruments, Ann Arbor, MI). Next, 2 mL of 2.5 M calcium chloride (CaCl_2) was added to the biochar slurry which was then mixed for 48 h. The concentrated biochar was diluted with 200 mL of DI water; 10 mL of this solution was then filtered with 0.45- μm nylon filters. Bromide was detected from the final solution using an Ion Chromatograph (Dionex 500, Thermo Fischer Sci, Sunnyvale CA). AEC and CEC calculations were performed following the methods described by Coleman et al., (1959).

Statistical analyses. To determine consistency of the batches for both biochar types, statistical analyses were run on data from each physical and chemical tests for between batch analysis; where the data was divided based on sample collection (either from the top, middle or bottom of a batch) and compared to the same sample collection area on the next batches. Therefore, each batch is a true replication and data was subjected to the general linear model (GLM) procedures. The standard deviation (s.d.) from this analysis for each physical and chemical test was then used as the range for consistency, and the mean across all the data of a single batch must be within the s.d. range of a particular test to be determined as consistent.

To determine the effect of charring, data from all physical and chemical property analyses were subjected to the general linear model (GLM) procedures when all batches of

biochar were compared to all batches of their respective feedstock and means were separated by least significant differences (LSD) at $P \leq 0.05$ (SAS Institute Version 9.3, Cary, NC).

When the three batches of biochar were compared to their feedstock Least Significant Differences (LSD) was used for means separation at $P \leq 0.05$.

Results and Discussion

Biochar production. Parameters for producing biochar in the TLUD gasifier are listed in Table 1. For the pine wood chips, the maximum temperatures across the three runs averaged 744.6°C and was within a $\pm 6\%$ difference. The time it took for the entire amount of feedstock to be charred (residency time) averaged 4.7 hours, and was within $\pm 10\%$ for run #1 and #2. The temperature for each run of pine wood chips was similar to temperatures used to gasify wood chips in a similar TLUD-designed gasifiers, Rivas (2015) saw temperatures from 648 to 815°C, and Vaughn et al. (2015) reported a temperature range of 720 to 755°C for 13 different species of trees and shrubs. Runs #1 and #2 were completed for PWC with a full 1.5-m³. Run #3 used the remaining volume of PWC available, which was 1.3-m³, slightly less than 1.5-m³. However, the final mass of biochar for all three runs was within $\pm 5\%$ of each other, indicating a similar amount of biochar was produced for comparison in the physical and chemical evaluations. The charring process caused an average loss of 30% of the PWC material by mass and an approximate loss of 50% by volume. This was a higher yield of wood biochar produced compared to another study using a similar TLUD design and gasified different wood species (Vaughn et al., 2015). This could be due to the particle size of the wood species used and the TLUD gasifier was smaller than the one used in this study.

Across all runs for the RH material, the maximum average temperature was 704.5°C compared to 744.6°C for PWC. However, all three runs of the rice hulls were within $\pm 6\%$, the same as the pine wood chips. These temperatures were in the same range but on the lower side as those by Rivas (2015) with a TLUD-designed gasifier and gasified rice hulls with temperatures of 700-862°C. The residency times averaged just under 3 hours (2.98 h). This was considerably faster than the pine wood chips (4.7 h). The starting mass for rice hulls averaged 644 kg, considerably less (100 kg) than the pine wood chips at 746 kg. However, the final mass after charring was almost the same between the two biochars (542 kg for RBC and 528 kg for PBC). Final mass for each run of rice hulls were within $\pm 5\%$. The charring process for rice hulls saw an average loss of 16% by mass (compared to 30% for PBC) and an approximate 50% loss by volume (the same as PBC). It is interesting to note that even though the PWC was heavier, both feedstocks produced the same volumes of char at the end. Both feedstocks had been dried at their respective processing locations; a moisture content between 12%-16% has been shown to improve efficiency and performance in biochar gasification production (Kaupp and Goss, 1983).

Particle size distribution. Particle size distribution of the feedstocks and resulting chars are listed in Table 2. All three batches for both PBC and RBC were within the consistency ranges for all sieve sizes, indicating a consistency of the batches for both biochars (Appendix A).

The feedstock PWC had a greater percentage of large-sized particles that were >2.0 -mm compared to PBC. The charring process decreased the larger particles of >6.3 -mm and >2.0 -mm to 0.29% and 50.3%, respectively, therefore increasing the amount of PBC particles

that were ≤ 0.71 -mm to 45%. The largest difference between PWC and PBC was seen with the 0.71-mm screen; there was an increase of approximately 30% of PBC particles (PWC 15%; PBC 45%). This is not surprising, as the gasification process tends to generate chars with smaller particles (Brewer et al., 2009; Yagicoglu et al., 2015). The greatest portion of particles of PBC were in the 2.0-mm and 0.71-mm screens, indicating that the particles were 2.0-mm and smaller. Biochars with particle sizes of 2.0-mm and less are very common; Northup (2013) used hardwood biochar that was 2.38-mm and less and Cao et al. (2014) used green waste biochar with particles 6.3-mm and less. However, the green waste biochar had a majority of the particles in the <0.6 -mm range.

Rice hulls had no particles >6.3 -mm, however the feedstock RH had 95% of particles >0.71 -mm. The charring process caused the greater percentages of particles to shift for RBC, to have 53% in the range of <0.71 -mm to >0.25 -mm. Altland and Locke (2013a) used gasified rice hulls as well, however the greater percentage of particles was 0.106-mm and less (25.8%). The differences between the rice hull biochar used in this study compared to Altland and Locke (2013a) could be due to the process of gasification and the amount of material used for the process, which was not stated in their study. The effect of charring can be seen in comparison of particle size of feedstock materials compared to finished biochar product, as well as differences in surface observations for the pine materials (Figs. 2A & 3) and very little differences for the rice materials (Figs. 2B & 4). Consistency between the batches for both biochars suggests consistency of the charring process at these particular particle sizes (beginning feedstock).

Porosity and bulk density. The charring process reduced the BD of the PWC feedstock to approximately half, from 0.25 to 0.13 g/cc for the PBC (Figure 5A). Lower BD can be a benefit for shipping and costs for use as a substrate (Wallach, 2008). Total porosity was increased almost 10% with PBC to 89.4% compared to PWC 79.5%. This was apparently due to an almost doubling of CC in PBC compared to PWC (52% to 28%). However, this seemed to be at the expense of AS (37% in PBC, 51% in PWC). This shift in CC and AS for the PBC would be expected as the PBC has more fine particles which would hold more water and drain less.

There was no difference in BD or TP for the RH feedstock and corresponding RBC (Figure 5B). There was a pronounced increase in CC for RBC compared to RH (55.8 and 22.4%, respectively), however AS decreased proportionally in RBC compared to RH (33 to 68%, respectively); again, to be expected with more fine particles in RBC. The effect of charring had a greater impact on RH to RBC with CC and AS; after charring the CC increased 33% and the AS decreased 35%. After charring the PWC, CC increased 24% and AS decreased 13%.

The physical properties of PBC and RBC fall within the normal ranges for components of container media as outlined by Mendez et al. (2015): AS 10%-30%, CC 60%-100% and TP 50%-80%. The AS of these biochars are similar to biochar reported from Yargicoglu et al. (2015) produced from aged oak and hickory wood by pyrolysis with an AS of 30%. Other biochars, including pine wood and coconut charcoal were reported to have AS much greater than PBC and RBC, at 47% and 60% respectively (Yargicoglu et al., 2015).

However, these other biochars are comparable in CC to PBC and RBC, with water-holding capacities ranging from 50% to 64% (Yargicoglu et al., 2015).

Hydration efficiency. The ability to capture and retain water is important for substrate components. PWC took three to four hydration events to reach near CC (Fig. 6A). The charring process did not adversely affect this process, with the PBC having nearly identical wetting curves as the PWC. In fact, the CC for PBC was slightly higher than the PWC by 7%. These values were similar to materials, such as PWC, that were separately produced and tested by Fields, et al., 2014.

Rice hulls had a very low ability to capture and retain water. RH captured between 13 and 15% water by volume on the first and all subsequent hydration events, although the CC was measured at 30% (Fig. 6B). The charring process greatly increased the CC to over 60%, however, the hydration events never captured more than 30% by volume. Both biochars, PBC and RBC, increase the water-holding capacities (CC) as seen in Figure 2, but appear to not have a positive effect on initial wettability to reach those CCs. It should be noted that these tests were done “as is” where all materials had a low moisture content. Increasing the moisture content may have improved the materials’ ability to capture water. However, Fields et al. (2014) saw no increase when the PWC had an initial moisture content of 50% by weight. Abel et al. (2013) also found that different biochars produced from whole maize plants had no effect on the wettability of soils and reported increases in water repellency.

Chemical properties. For several of the chemical tests conducted, there were some batches that did not fit the considered consistency ranges, however these consistency ranges were created with one standard deviation and all batches would fit in ranges using two

standard deviations (within 95.5% of normal distribution). Therefore, the tests with outlying batches are small differences. The PBC biochars had several elements from total elemental composition (Appendix B and C), total carbon (Appendix D), water extraction (Appendix E and F), and acid extraction (Appendix G), however most of these batches were outside the ranges by 1.0 to 0.01 mg·L⁻¹. The PBC batch #1 was outside the CEC consistency range by 0.04 mmol·100cc⁻¹ and batch #3 was outside the AEC consistency range by 0.13 mmol·100cc⁻¹. The RBC batches were within the consistency ranges for total carbon (Appendix D), total elemental composition (Appendix B and C) and CEC/AEC (Appendix H). Some of the RBC batches were outside the consistency ranges for water extraction (Appendix E and F) and acid extraction tests (Appendix G); most of these batches were outside the ranges by 1.4 to 0.001 mg·L⁻¹. Some of these batches that are outside the consistency ranges could be due to testing error, however it is important to measure these characteristics for specific biochars and their production and note that any small changes in production parameters could alter the end biochar product and create inconsistencies.

Total carbon, nitrogen and hydrogen. Total percentages of C and N increased after charring the PWC. The C content (%) for PBC is similar to wood chips produced through a smaller TLUD gasifier (84% C), however the H and N content are greater at 2.49% and 0.46%, respectively (Rivas, 2015). Though the wood chips used by Rivas (2015) were similar in particle size (average 2-mm), the temperatures at combustion were not; Rivas reported a temperature of 650°C whereas in this study the temperature was 750°C for wood chips. The C, H, and N contents for PBC and RBC are similar to biochars produced by pyrolysis of switchgrass and corn stover with ranges of 37%-62% C, 1.3%-2.5% H and

0.6%-1.3% N (Brewer et al., 2009). Yargicoglu et al. (2015) reported C, H and N contents for biochar produced from coconut charcoal, pine wood, aged oak and hickory wood and 90% pine with 10% fir wood; all but the coconut charcoal had lower C and N contents compared to PBC. These biochars were also similar to RBC with C, H, and N contents. The C:N ratios were high for PBC, ranging from 260:1 to 381:1. These ratios were similar to the C:N ratio of coconut charcoal (426:1) and pine wood pellet biochars with 290:1 C:N ratios, but were higher than other wood-type biochars reported by Yargicoglu et al. (2015), including pine wood biochar produced by slow pyrolysis with C:N of 143:1 and the pine wood chip biochar produced by Rivas (2015) with C:N of 183:1.

For RBC, there was not many differences between C% and N%, and H% was less than the feedstock RH (Table 3). Altland and Locke (2013a; 2013b) also used gasified rice hulls in their experiments, however they reported percent of C and N at 17.68 and 0.18, respectively, both very different from the gasified rice hulls in this experiment that averaged percent of C and N at 40.00 and 0.41, respectively. However, the process of gasification cannot be compared as the parameters were not reported. The C:N ratios of RBC were less than PBC, ranging from 95:1 to 102:1, and similar in C:N to biochar produced from aged oak and hickory wood with a C:N ratio of 152:1 (Yargicoglu et al., 2015).

Elemental analysis. For all the elements except Si and S, elements in PBC were greater than or equal to the total elemental concentrations in PWC feedstock (Table 3). The effect of charring shows large increases in macro-elements for PBC compared to PWC (Table 3). Potassium increased more than 4x (645 to 2871 mg·L⁻¹) and calcium increased over 3x (1462 to 5213 mg·L⁻¹) for PBC, while Mg and P increased around 2 fold (Table 4).

Pine wood biochar and RBC are similar to total elemental concentrations of biochar made from agricultural or forestry residues, except for P in which PBC was 3x greater and RBC was 10x greater (Dumroese et al., 2011). Biochars produced from olive mill and forest wastes had total elemental composition measured, however due to differences in feedstocks, was very different in composition compared to PBC and RBC (Fornes et al., 2015).

For all elements except S and Fe, RBC was greater than or equal to the total elemental concentrations of RH feedstock. There was also a large increase in macro-elements for RBC compared to RH. Both Ca and Mg increased 2.8x for RBC, whereas P and K increased over 3x (538 to 1765 mg·L⁻¹, 1442 to 4905 mg·L⁻¹, respectively). There were large amounts of Si in the RH and RBC materials, this is due to the naturally high contents of lignin and opaline silica found in rice hulls. Values ranged from 70,000 to 80,000 mg·L⁻¹ (Table 3). Silica is increasingly used as a supplement in soils, soilless substrates and hydroponic nutrient solutions. Several investigations have demonstrated Si was responsible for improved plant growth of various plant species (Liang et al., 2015), and Si was also found to play a role in plant disease resistance (Lewin and Reimann, 1969). The inclusion of Si in nutrient solutions resulted in a higher portion of Class I (i.e. high quality) flowers for gerbera (*Gerbera jamesonii* Adlam) plants when grown in hydroponic systems (Savvas et al., 2002). The differences in elemental composition between the PBC and RBC could be due to the position/job of the materials themselves. Rice hulls surround and protect the rice grain, which is considered a sink with the rice plant allocating nutrients towards this area. Whereas the wood materials would be composed of lignin, cellulose and hemicellulose.

Water soluble nutrients. The effect of charring with the available nutrients is very different from the total elemental test. Charring caused water-soluble K to increase; PBC was almost 4x greater and RBC was about 2x greater compared to their feedstocks. However, both PBC and RBC had less water-soluble B, Ca, Cu, Fe, Mg, Mn, N, P, S, and Zn than their respective feedstocks (Table 5). Altland and Locke (2013b) also reported that three different types of biochar, gasified rice hull biochar, sawdust biochar, and bark/wood biochar, were a source of K in soilless substrates. Lou et al. (2016) used a method of adding biochar to water and heating it at 100C for 3 h, then shaking the mixture in order to remove water extracts from wheat straw biochar and maize stem biochar for nutrient analysis. They found higher concentrations of all elements they reported, however this could be due to the different methods between their study and this one. The water-soluble nutrients of Mg, P, and Mn found for PBC and RBC are similar to reported measurements of biochar produced from olive mill waste and forest waste, however differences were found with other nutrients (Fornes et al., 2015). This was also found when comparing PBC and RBC to biochar made from hardwood pellets and pelletized wheat straw, several nutrients were similar in concentration such as K, Mg, Mn, and Cu, but others were very different (Vaughn et al., 2013). This is expected, as these biochars were produced from very different feedstocks and would have different elemental composition before and after charring.

The concentrations of water-soluble nutrients measured were very different from the nutrients measured by Mehlich-III acid extraction (Table 5). The Mehlich-III acid extraction is often used to measure available nutrients of soils and biochar (Major et al., 2010), however it greatly exaggerates the concentration that would be immediately available for plants in

solution. Nelson et al. (2011) found extractable P was 4x greater for corn cob biochar when using the Mehlich-III acid extraction compared to water-extractable P. When comparing macro-elements Ca, Mg, P and K across elemental composition, Mehlich-III (acid) extraction and water extraction, there is a clear trend of lower concentrations of these elements as the tests start with acid digestion – acid extraction – water extraction (Table 4). While elemental composition can show high levels of nutrients, in reality the water extraction shows that there are very low amounts of these nutrients available for the plant. The elemental composition shows PBC is composed of high levels of Ca and K, however water extraction (plant available) is 1000 fold less for Ca and 8 fold less for K. This is a similar trend for RBC, however the decrease from elemental K to water extractable K is 14 fold. Neither of these two chars would contribute much to the nutritive value in the soil solution when blended into a substrate for container production. Indeed, the process of charring may actually sequester these nutrients, much like carbon, and prevent their timely release into the substrate.

pH, EC and buffering capacity. Charring had an overall effect on the pH and EC of the materials. The PBC biochar pH values (8.7) were double those of the PWC (4.2; Table 6). Electrical conductivity also doubled compared to the feedstock PWC, but the values were not excessively high ($1.2 \text{ mS}\cdot\text{cm}^{-1}$). Among the batches, the pH and EC values for PBC were very similar suggesting batch consistency; pH only differed by 0.2 while the maximum difference of EC was $0.37 \text{ mS}\cdot\text{cm}^{-1}$. The charred rice hulls showed similar results; pH doubling from 4.37 to 9.47, and a similar increase in EC from 0.73 to $1.15 \text{ mS}\cdot\text{cm}^{-1}$. For both the pH and EC of PBC, all three batches were within the consistency ranges (Appendix I). All three batches of RBC were within the consistency ranges of EC, however one batch was

outside the consistency range for pH. With these observations, the biochar production process was considered consistent from batch to batch, however it is important to note that variability can occur if the production parameters were altered.

These pH and EC ranges are similar to other reports of biochar; biochar produced from forest waste ranged from 8.6-9.6 pH and 1.4 to 0.7 ($\text{mS}\cdot\text{cm}^{-1}$; Fornes et al., 2015), biochar produced from pine wood with pH of 8.47 and EC 0.04 ($\text{mS}\cdot\text{cm}^{-1}$; Yargicoglu et al., 2015), biochar produced from hardwood pellets (pH of 8.0) or pelletized wheat straw (pH of 9.5; Vaughn et al., 2013), biochar produced from wheat straw (9.6 pH) or maize stalks (9.5 pH; Lou et al., 2016), biochar produced from switchgrass had pH of 8.0 (Novak et al., 2009), and biochar produced from eucalyptus wood at 400°C had pH of 6.9 or 550°C had pH of 8.8 (Singh et al., 2010). Mukherjee et al. (2011) reported that pH is dependent on original feedstock species, as pH varies widely with biochar. With different biochars, pH can range from alkaline (8.6) to slightly acidic (3.7) all due to the feedstock used as well as temperature of production (Mukherjee et al., 2011). The feedstock for both materials, PWC and RH, had higher pH buffering capacities than their biochar counterpart (Table 6).

The pH buffering capacity of biochar was more varied than pH or EC (Table 6). Both PWC and RH feedstocks showed similar values of 18.17 and 19.36 $\text{mmol}\cdot\text{kg}^{-1}$, respectively. However, after charring, PBC buffering capacity dropped to 6.17 while the RBC dropped only to 12.28 $\text{mmol}\cdot\text{kg}^{-1}$. Both materials actually lost buffering capacity after charring, but the RBC was a smaller loss. There was slightly more variation in buffering capacity from batch to batch in both the original feedstocks and the resulting chars. This might be due to the dynamic nature of the test procedure (Barnes, 2013).

During the hydration efficiency testing we observed many pockets of air bubbles in the samples. The original feedstocks eventually hydrated the air pockets, but the chars never fully hydrated the sample over every hydration event, as well as having air bubbles surrounding some biochar particles. These bubbles were not cataloged or measured in any way, but their placement was later noted as possibly barriers to allow water to more fully hydrate the char samples. There could have been an issue with hydrophobicity as was suggested through the hydration curves (Figure 3), as well as the possibility that biochar has air bubbles in internal pores that effect the acid/base from interacting with all the sites on the biochar particles.

Samples of the same chars from each run were submerged in water and placed under a vacuum for 15 min. The initial reaction was a profusion of air bubbles leaving the chars. After this procedure, the degassed chars were analyzed for pH buffering capacity in the same manner as the un-gassed samples (Table 7). The pH for the PWC feedstock material rose from 4.2 to 8.8 with charring, but with additional degassing, the pH dropped almost 2 full units to 6.9. The buffering capacity went from 18.17 mmol·kg⁻¹ for the PWC feedstock to 6.17 for the PBC char, but rose after degassing to 14.84 mmol·kg⁻¹. This shift back up with degassing may be due to more surface area of the de-gassed char being in direct contact with the solutions that altered pH. The pH for RH feedstock went from 4.4 to 9.5 after charring. However, the degassing did not alter pH (9.3) for the rice hull char. The pH buffering capacity had a smaller decrease from 19.37 mmol·kg⁻¹ in the feedstock hulls to 12.28 mmol·kg⁻¹ in the char, with degassing resulting in 15.11 mmol·kg⁻¹. Degassing did alter the

pH buffering capacities of the biochars and essentially brought it back up to values similar to the materials before charring.

These values were similar to the pH buffering capacity noted for biochar made from crop residues, including canola, corn, soybean and peanut (Yuan et al., 2011). Yuan et al. (2011) noted that biochar had pH buffering capacity similar to those for weak alkaline substances. These feedstock and degassed biochar materials were also similar to Oxisols treated with canola straw biochar and peanut straw biochar (20-38 mmol·kg⁻¹), as well as Ultisols treated with the same biochars (15-36 mmol·kg⁻¹; Xu et al., 2012).

Cation/anion exchange capacities. The CEC of the feedstock PWC (29.02 mmol·kg⁻¹) was slightly higher than the average for PBC (27.69 mmol·kg⁻¹), and the feedstock RH (18.34 mmol·kg⁻¹) was slightly less than the average for RBC at 21.13 mmol·kg⁻¹ RBC (Table 8). There were no differences in CEC between RH feedstock and two of the three batches of RBC biochars (18.34, 19.80 and 21.13 mmol·kg⁻¹, respectively), however batch #2 of RBC was greater at 22.47 mmol·kg⁻¹. The effect of charring for both types of feedstocks appears to not greatly increase the CEC for these materials, both biochar types only had one batch that had a greater CEC (#1 for PBC, #2 for RBC) than the feedstock CEC. The CEC of all materials are similar or higher than several other greenhouse components (e.g. pine bark), which have CECs between 6-15 mmol·100cc⁻¹ (Nelson, 2011). Nieto et al., 2015 reported CEC for pruning waste biochar produced at 300°C and 500°C to be similar to PBC and RBC at 26 and 15.5 mmol 100g⁻¹, respectively. However, PBC and RBC had much lower CEC than biochars produced from oak, pine and grass species (10.2-69.2 cmol·kg⁻¹; Mukherjee et al., 2011) and biochar produced from tomato crop green-waste (~500 mmol·kg⁻¹; Dunlop et

al., 2015). However, the CEC for PBC and RBC by itself is well below the recommended CEC for container media: $200 \text{ mmol} \cdot 100\text{g}^{-1}$ (Mendez et al., 2015).

Charring had a large and opposite effect on AEC. The AEC of PWC was much higher than PBC (19.7 and $2.21 \text{ mmol} \cdot \text{kg}^{-1}$, respectively; Table 8). However, charring greatly increased the AEC of RBC, going from $3.92 \text{ mmol} \cdot \text{kg}^{-1}$ for RH to $25.88 \text{ mmol} \cdot \text{kg}^{-1}$. The AEC of PBC and RBC was similar to biochar made from alfalfa (0.938 - $2.15 \text{ cmol} \cdot \text{kg}^{-1}$), cellulose (0.6 - $4.11 \text{ cmol} \cdot \text{kg}^{-1}$) and corn stover (1.05 - $7.19 \text{ cmol} \cdot \text{kg}^{-1}$) at a pH of 8 (Lawrinenko, 2014). Lawrinenko (2014) showed AEC increases as pH decreases for those alfalfa, cellulose and corn stover biochars. However, to use these biochars in greenhouse media, the recommended pH is between 5.2 and 6.4 and therefore the biochars could not be used if their pH was at 4, which is the pH that biochar had the greatest AEC.

Conclusion

The production system used, TLUD gasifier, produced consistent physical and chemical results over the batches for both biochars. The batches among both PBC and RBC were consistent in particle size distribution, pH, water/acid extracted nutrients, and total elemental composition. The CEC and AEC measurements of RBC were similar between each run, indicating a consistency of process. These physical and chemical tests have reported the characterization of the two types of biochar and any beneficial impacts they might have on rhizosphere environment and plant growth. The particle size distribution is an important characterization of biochars, as it is a property that can be easily altered or changed, either in the feedstock form or after charring, to create various particle sizes and surface areas that can impact substrates with varying results. Therefore, knowing the particle size of the biochar

being used is very important in comparison to other biochars. However, the two feedstocks produced fundamentally different biochars that would interact with the physical and chemical environment of substrates in different ways.

There may have been an issue with the hydrophobicity of the biochars. Both biochar materials had a greater difference between their final volumetric water content and CC, therefore both PBC and RBC did not reach their full water-holding efficiencies after ten irrigation events. This suggests that these biochars used directly from the gasifier may be too dry to fully aid with the initial wettability of substrates. Even hydrating the biochars by the protocol for pH buffering (stir in water for 30mins) was not enough to fully hydrate the biochar particles, shown by the difference between biochar and degassed biochar.

Another issue with inconsistency of biochar improving plant growth could be due to using the acid (Mehlich-III) extraction or total elemental as a method to report nutrients. As shown with these studies, both acid extractions grossly overestimate the contribution of biochar to the plant available nutrients with some nutrients such as phosphorus being over 10x greater and potassium being over 6x greater than water extractable P and K. Therefore, reports that only include the acid extraction for plant nutrients are not fully corresponding available nutrients in the rhizosphere and increased/decreased plant growth when amending with biochar. In fact, since biochar lasts for up to a hundred years in the soil, the char may actually sequester not only carbon but plant nutrients as well. At least the uncharred plant residues will degrade and make the elements available to plant growth, while the chars may retard this process.

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Table 1. Parameters set or measured of biochar production from either pine wood chips (PBC) or rice hulls (RBC) made in a top-lit updraft (TLUD) gasifier with air flow set at $12 \text{ m}^3 \cdot \text{min}^{-1}$ for uniformity.

Material	Batch	Date	Max. temperature ($^{\circ}\text{C}$) ^z	Residency (hr) ^y	Start mass (kg) ^x	Finish mass (kg) ^w	Biomass lost (%) ^v
PBC	#1	3/14/14	737.8	4.92	767.48	528.89	31
	#2	4/17/14	716.0	5.38	784.71	536.15	32
	#3	12/15/14	779.9	3.75	685.83	519.82	24
	Average		744.6	4.68	746.02	528.29	29
RBC	#1	12/16/14	711.3	3.05	645.92	538.87	17
	#2	12/17/14	719.7	2.88	639.57	541.59	15
	#3	12/18/14	682.6	3.00	648.64	545.22	16
	Average		704.5	2.98	644.71	541.89	16

^zMaximum temperature is the measurement from the internal probe measuring the highest temperature of the flame front for each batch run.

^yResidency is the total amount of time the material was enduring the charring process, beginning from the ignition until flame front hit the bottom of the reactor and air flow was shut off.

^xStarting mass is the initial weight of the material without the weight of the gasifier, before ignition.

^wFinished mass is the final weight of the material after charring process is complete (air flow shut off) and without the weight of the gasifier.

^vBiomass lost was calculated by [(Starting mass minus Finished mass) divided by Starting mass] multiplied by 100.

Table 2. Particle size distribution of feedstock and biochar materials within batch (% weight).^z

Material	Batch	Screen sizes (mm)						
		6.3	2.0	0.71	0.5	0.25	0.106	<0.106
		% weight						
PWC ^y	#1	1.82 a	81.11 a	16.41 b	0.25 b	0.08 c	0.11 bc	0.22 c
	#2	1.60 a	83.56 a	14.30 b	0.16 b	0.13 c	0.08 c	0.18 c
	#3	2.05 a	82.41 a	14.77 b	0.22 b	0.11 c	0.19 bc	0.25 c
	Average	1.82	82.36	15.16	0.21	0.11	0.13	0.22
PBC ^w	#1	0.06 b	52.87 b	42.84 a	1.34 a	0.65 b	0.77 a	1.47 b
	#2	0.54 b	48.38 b	45.80 a	1.93 a	0.88 ab	0.48 ab	1.99 a
	#3	0.26 b	49.66 b	45.37 a	1.89 a	1.13 a	0.26 bc	1.43 b
	Average	0.29	50.3	44.67	1.72	0.89	0.50	1.63
RH ^v	#1	0.00 a	25.54 a	69.67 a	2.56 b	1.41 b	0.56 b	0.26 b
	#2	0.00 a	24.67 a	70.09 a	2.59 b	1.70 b	0.69 b	0.26 b
	#3	0.00 a	24.79 a	70.37 a	2.57 b	1.51 b	0.58 b	0.19 b
	Average	0.00	25.00	70.04	2.57	1.54	0.61	0.24
RBC ^u	#1	0.00 a	1.66 b	38.84 b	30.02 a	23.67 a	4.74 a	1.07 a
	#2	0.00 a	1.54 b	39.11 b	30.46 a	23.03 a	4.83 a	1.03 a
	#3	0.00 a	2.55 b	39.17 b	30.02 a	22.67 a	4.66 a	0.94 a
	Average	0.00	1.92	39.04	30.17	23.12	4.74	1.01

^zParticle size distribution data were collected from three samples from each batch run and represented as a mean percent by weight of the samples. Analysis performed using Ro-tap Shaker (Model B, W.S. Tyler, Mentor, Ohio) fitted with six sieves; 6.3 mm, 2mm, 0.71 mm, 0.5 mm, 0.25 mm, and 0.106 mm.

^yPWC = pine wood chips as feedstock (not charred).

^xMeans separated within column, only between feedstock and corresponding biochar (i.e. PWC compared to PBC), by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different.

^wPBC = biochar produced from pine wood chips.

^vRH = rice hulls as feedstock (not charred).

^uRBC = biochar produced from rice hulls.

Table 3. Total elemental composition of feedstock and biochar materials within batch. Elements include boron (B), calcium (Ca), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), molybdenum (Mo), phosphorus (P), potassium (K), silica (Si), sulfur (S) and zinc (Zn). Total carbon (C), hydrogen (H) and nitrogen (N) as well as ratios for the materials.

Material	Batch	Total elemental composition (mg·L ⁻¹) ^z											
		B	Ca	Cu	Fe	Mg	Mn	Mo	P	K	Si	S	Zn
PWC ^y	-	3.3 c ^x	1461.7 c	4.1 b	5924.0 a	719.4 b	112.0 b	0.30 a	180.53 c	653.8 b	7154 a	401.3 a	19.2 b
PBC ^w	#1	7.7 ab	5115.5 b	15.2 a	3091.0 a	1245.5 a	317.0 a	0.30 a	461.33 ab	2854.2 a	1268.1 b	92.3 b	25.7 ab
	#2	6.6 b	4839.2 b	7.6 ab	2047.0 a	1169.4 a	296.7 a	0.32 a	424.7 b	2699.8 a	546.1 b	67.6 b	27.1 ab
	#3	9.1 a	5684.0 a	10.3 ab	475.0 a	1333.3 a	340.3 a	0.30 a	513.52 a	3058.5 a	622.9 b	111.8 b	31.9 a
RH ^v	-	2.3 b	736.2 c	1.40 b	774.0 a	353.6 b	185.3 b	0.41 b	538.5 c	1442.4 b	80469 a	307.7 a	9.8 b
RBC ^u	#1	5.0 a	2129.1 a	7.03 a	268.7 b	968.7 a	501.0 a	0.53 ab	1769.6 ab	4950.3 a	75628 a	129.2 bc	26.0 a
	#2	4.4 a	2089.5 ab	5.80 a	315.3 b	1020.3 a	518.7 a	0.42 ab	1790.6 a	4962.7 a	86665 a	96.7 c	26.5 a
	#3	4.7 a	2037.5 b	5.37 a	200.7 b	987.6 a	502.3 a	0.57 a	1733.7 b	4801.6 a	76186 a	166.0 b	25.2 a
		C:H:N ratios ^t											
		Total C (%)	Total H (%)	Total N (%)	C:H	C:N	C:H:N						
PWC	-	45.14 c ^s	3.21 a	0.1 c	14:1	451:1	451:32:1						
PBC	#1	79.96 b	1.69 b	0.21 b	47:1	381:1	381:8:1						
	#2	82.12 ab	1.70 b	0.22 b	48:1	373:1	373:8:1						
	#3	85.86 a	1.39 c	0.33 a	62:1	260:1	260:4:1						
RH	-	39.11 b	3.31 a	0.35 b	14:1	112:1	112:10:1						
RBC	#1	40.79 ab	0.83 c	0.43 a	96:1	95:1	95:2:1						
	#2	39.87 ab	0.91 c	0.39 ab	90:1	102:1	102:2:1						
	#3	41.08 a	1.05 b	0.41 a	82:1	100:1	100:3:1						

^zTotal elemental composition measured by dry ash procedure and wet digestion method by inductively coupled plasma optical emission spectroscopy (ICP-OES).

^yPWC = pine wood chips as feedstock (not charred).

^xMeans separated within column for total elemental composition, only between feedstock and corresponding biochar (i.e. PWC compared to PBC), by Least Significant Differences (LSD) at $P \leq 0.05$. Means followed by the same letter are not significantly different (n=3).

^wPBC = biochar produced from pine wood chips.

^vRH = rice hulls as feedstock (not charred).

^uRBC = biochar produced from rice hulls.

^tTotal C, H, and N was determined by total combustion method and CHNS/O Analyzer (2400 Series II PerkinElmer, Waltham, MA).

^sMeans separated within column for C:H:N ratios, only between feedstock and corresponding biochar (i.e. PWC compared to PBC), by LSD at $P \leq 0.05$. Means followed by the same letter are not significantly different (n=3).

Table 4. Comparison of selected nutrient values using different extraction methods for pine wood chips (PWC), charred wood chips (PBC), rice hulls (RH) and charred rice hulls (RBC).

Extraction method	Material							
	PWC		PBC		RH		RBC	
	Mean	<i>s.d.</i>	Mean	<i>s.d.</i>	Mean	<i>s.d.</i>	Mean	<i>s.d.</i>
	Calcium (mg·L ⁻¹)							
Total elemental ^z	1462 ^y	391.7	5213 ^x	410.4	736	16.8	2085	54.9
Mehlich-III acid ^w	478	30.6	590	110.9	228	9.7	288	54.4
Water extract ^v	34	1.1	5	1.1	7	1.0	2	0.2
	Magnesium (mg·L ⁻¹)							
Total Elemental	719	418.2	1249	85.2	354	10.9	992	44.1
Mehlich-III acid	144	7.5	144	34.2	312	18.6	172	18.2
Water extract	16	0.5	16	2.9	19	2.3	4	0.6
	Phosphorous (mg·L ⁻¹)							
Total Elemental	180	79.5	467	45.9	538	36.0	1765	32.4
Mehlich-III acid	58	4.2	55	7.4	394	24.0	381	47.4
Water extract	16	0.6	2	0.1	58	4.5	33	4.2
	Potassium (mg·L ⁻¹)							
Total Elemental	654	215.5	2871	239.7	1442	29.1	4905	190.0
Mehlich-III acid	439	23.6	1300	174.5	2016	123.3	2150	202.2
Water extract	95	3.5	360	54.0	200	11.8	350	57.6

^zTotal elemental composition measured dry ash procedure and wet digestion by inductively coupled plasma optical emission spectroscopy (ICP-OES).

^yMeans for feedstocks materials (PWC and RH) determined from n=3, means within batch.

^xMeans for biochar materials (PBC and RBC) determined across all three batches of each biochar type (n=9).

^wMehlich-III determined using the Mehlich-III extraction [reagents include ammonium nitrate, ammonium fluoride, acetic acid, nitric acid and ethylenediaminetetraacetic acid (EDTA); Mehlich, 1984].

^vWater extract determined by saturated media extract and vacuum filtration with an auto-flow spectrophotometric analyzer.

Table 5. Available nutrients of feedstock and biochar material within batch, measured by two different methods; water extraction and acid (Mehlich-III) extraction. Nutrients include boron (B), calcium (Ca), chloride (Cl), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), nitrogen (N), phosphorus (P), potassium (K), sodium (Na), sulfur (S) and zinc (Zn).

		Water extraction (mg·L ⁻¹) ^z												
Material	Run	B	Ca	Cl	Cu	Fe	Mg	Mn	N	P	K	Na	S	Zn
PWC ^y	-	0.08 a ^x	34.2 a	1.0 c	0.02 a	1.73 a	16.0 a	2.62 a	2.4 a	15.8 a	95.3 c	6.4 b	8.4 a	0.2 a
PBC ^w	#1	0.01 bc	5.0 c	3.8 a	0.01 b	0.02 b	10.7 b	0.06 b	0.8 b	1.5 b	344.3 b	8.5 a	1.8 bc	0.0 b
	#2	0.02 b	5.0 c	1.8 b	0.01 b	0.04 b	9.5 b	0.09 b	0.8 b	1.5 b	325.3 b	8.3 a	1.8 c	0.0 b
	#3	0.01 c	6.9 b	1.8 b	0.01 b	0.02 b	15.0 a	0.06 b	0.8 b	1.5 b	435.3 a	6.8 b	2.3 b	0.0 b
RH ^v	-	0.09 a	7.6 a	4.1 d	0.02 a	0.3 a	19.0 a	6.2 a	1.9 a	58.4 a	199.7 d	15.6 b	6.3 a	0.03 a
RBC ^u	#1	0.01 b	1.2 b	6.3 b	0.01 b	0.03 b	4.2 b	0.5 b	0.5 b	33.7 b	356.0 b	22.9 a	5.9 a	0.0 b
	#2	0.001 c	1.4 b	7.2 a	0.01 b	0.03 b	4.6 b	0.5 b	0.4 b	37.3 b	420.7 a	20.7 a	4.6 ab	0.0 b
	#3	0.001 c	1.2 b	5.1 c	0.01 b	0.02 b	3.6 b	0.5 b	0.6 b	28.2 c	294.7 c	16.4 b	3.2 b	0.01 b
		Acid extraction (mg·L ⁻¹) ^t												
		Ca	Cu	Mg	Mn	P	K	Na	S	Zn				
PWC	-	478.3 b	0.40 b	144.0 b	36.4 b	51.7 bc	439.0 c	11.3 b	26.3 a	3.9 a				
PBC	#1	617.0 a	0.77 a	223.0 a	43.9 a	61.3 a	1513.0 a	24.0 a	8.7 bc	3.7 ab				
	#2	459.7 b	0.70 a	153.0 b	30.3 b	46.7 c	1277.3 b	13.0 b	7.7 c	3.1 bc				
	#3	693.0 a	0.80 a	198.7 a	48.0 a	58.0 ab	1587.3 a	13.0 b	11.0 b	2.8 c				
RH	-	228.3 b	0.50 a	311.7 a	125.1 a	393.7 a	2016.0 b	43.7 a	43.0 a	6.8 a				
RBC	#1	319.0 a	0.30 a	191.3 b	108.4 b	423.3 a	2343.7 a	44.3 a	26.7 b	6.2 b				
	#2	327.0 a	0.33 a	174.3 bc	104.6 b	390.3 a	2303.0 a	42.0 a	18.0 b	5.5 c				
	#3	219.3 b	0.37 a	153.7 c	84.2 c	330.3 b	1969.7 b	37.0 b	16.3 b	4.9 c				

^zDetermined by saturated media extract and vacuum filtration with an auto-flow spectrophotometric analyzer.

^yPWC = pine wood chips as feedstock (not charred).

^xMeans separated within column by method, only between feedstock and corresponding biochar (i.e. PWC compared to PBC), by Least Significant Differences (LSD) at $P \leq 0.05$. Means followed by the same letter are not significantly different (n=3).

^wPBC = biochar produced from pine wood chips.

^vRH = rice hulls as feedstock (not charred).

^uRBC = biochar produced from rice hulls.

^tDetermined using the Mehlich-III extraction [reagents include ammonium nitrate, ammonium fluoride, acetic acid, nitric acid and ethylenediaminetetraacetic acid (EDTA); Mehlich, 1984].

Table 6. pH, electrical conductivity and pH buffering capacity of feedstock and biochar materials within batch.^z

Material	Run	pH	Electrical conductivity (mS/cm)	pH buffering capacity ^y mmol kg ⁻¹
PWC ^x	#1	4.2 c ^w	0.57 c	12.91 a
	#2	4.2 c	0.54 c	20.94 a
	#3	4.2 c	0.57 c	20.66 a
	Average	4.2	0.56	18.17
PBC ^v	#1	8.8 ab	1.13 b	7.23 b
	#2	8.7 b	1.05 b	6.79 b
	#3	8.9 a	1.42 a	4.49 b
	Average	8.7	1.20	6.17
RH ^u	#1	4.4 bc	0.75 d	22.50 a
	#2	4.4 b	0.68 d	20.22 a
	#3	4.3 c	0.76 d	15.38 a
	Average	4.37	0.73	19.37
RBC ^t	#1	9.5 a	0.95 c	11.45 b
	#2	9.5 a	1.33 a	10.84 b
	#3	9.4 a	1.16 b	14.55 a
	Average	9.47	1.15	12.28

^zpH and electrical conductivity were conducted using the 1:1 dilution method with Hanna meter (HI 9811, Hanna Instruments, Ann Arbor, MI).

^ypH buffering capacity measured by titration and averaged over the points of the titration curve between pH of 5.0 and 7.0.

^xPWC = pine wood chips as feedstock (not charred).

^wMeans separated within column, only between feedstock and corresponding biochar (i.e. PWC compared to PBC), by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different.

^vPBC = biochar produced from pine wood chips.

^uRH = rice hulls as feedstock (not charred).

^tRBC = biochar produced from rice hulls.

Table 7. pH and pH buffering capacity of biochar and degassed biochar materials within batch.^z

Material	Run	pH	pH Buffering Capacity ^y mmol kg ⁻¹
PBC ^x	#1	8.8 a ^w	7.23 b
	#2	8.7 a	6.79 b
	#3	8.9 a	4.49 b
Average		8.8	6.17
PBC-DE ^v	#1	7.4 b	14.12 a
	#2	6.7 b	16.51 a
	#3	6.6 b	13.88 a
Average		6.9	14.84
RBC ^u	#1	9.5 a	11.45 a
	#2	9.5 a	10.84 b
	#3	9.4 a	14.55 b
Average		9.47	12.28
RBC-DE ^t	#1	9.4 a	5.54 b
	#2	9.3 a	17.79 a
	#3	9.1 a	22.00 a
Average		9.27	15.11

^zDegassed materials were saturated in water and placed under pressurized vacuum to remove all air bubbles within biochar particles.

^ypH buffering capacity measured by titration and averaged over the points of the titration curve between pH of 5.0 and 7.0.

^xPBC = biochar produced from pine wood chips.

^wMeans separated within column, only between biochar and corresponding degassed biochar (i.e. PBC compared to PBC-DE), by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different.

^vPBC-DE = degassed biochar produced from pine wood chips.

^uRBC = biochar produced from rice hulls

^tRBC-DE = degassed biochar produced from rice hulls.

Table 8. Cation exchange capacity (CEC) and anion exchange capacity (AEC) of feedstock and biochar materials within batch.^z

Material	Run	Cation exchange capacity ^y	Anion exchange capacity ^x
		mmol·100cc ⁻¹	
PWC ^w	-	29.02 ab ^v	19.70 a
PBC ^u	#1	32.18 a	1.81 b
	#2	23.98 c	1.62 b
	#3	26.91 bc	3.19 b
Average		27.69	2.21
RH ^t	-	18.34 b	3.92 c
RBC ^s	#1	19.80 ab	30.29 a
	#2	22.47 a	28.93 a
	#3	21.13 ab	18.41 b
Average		21.13	25.88

^zMeasured at pH of 4.2 for PWC, 8.8 for PBC, 4.4 for RH, and 9.5 for RBC.

^yCation exchange capacity measured using procedures described by Kloss et al. (2012) and Dumroese et al. (2011).

^xAnion exchange capacity measured using the procedure described by Lawrinenko (2014).

^wPWC = pine wood chips as feedstock (not charred).

^vMeans separated within column, only between feedstock and corresponding biochar (i.e. PWC compared to PBC), by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different.

^uPBC = biochar produced from pine wood chips.

^tRH = rice hulls as feedstock (not charred).

^sRBC = biochar produced from rice hulls.



Figure 1. (A) Top-lit updraft (TLUD) gasifier used to produce pine wood chip and rice hull biochars. (B) Gasifier with lid and gas stock attached to show entire system. (C) The air fan used to blow $12\text{-m}^3\cdot\text{min}^{-1}$ and (D) the connecting tube connected to the bottom of the gasifier to force air up.



Figure 2. (A) On the left, pine wood chips feedstock (loblolly pine; *Pinus taeda*) and final pine wood biochar product (right) made using the top-lit updraft (TLUD) gasifier. (B) On the left, rice hull feedstock and final rice hull biochar product (right).

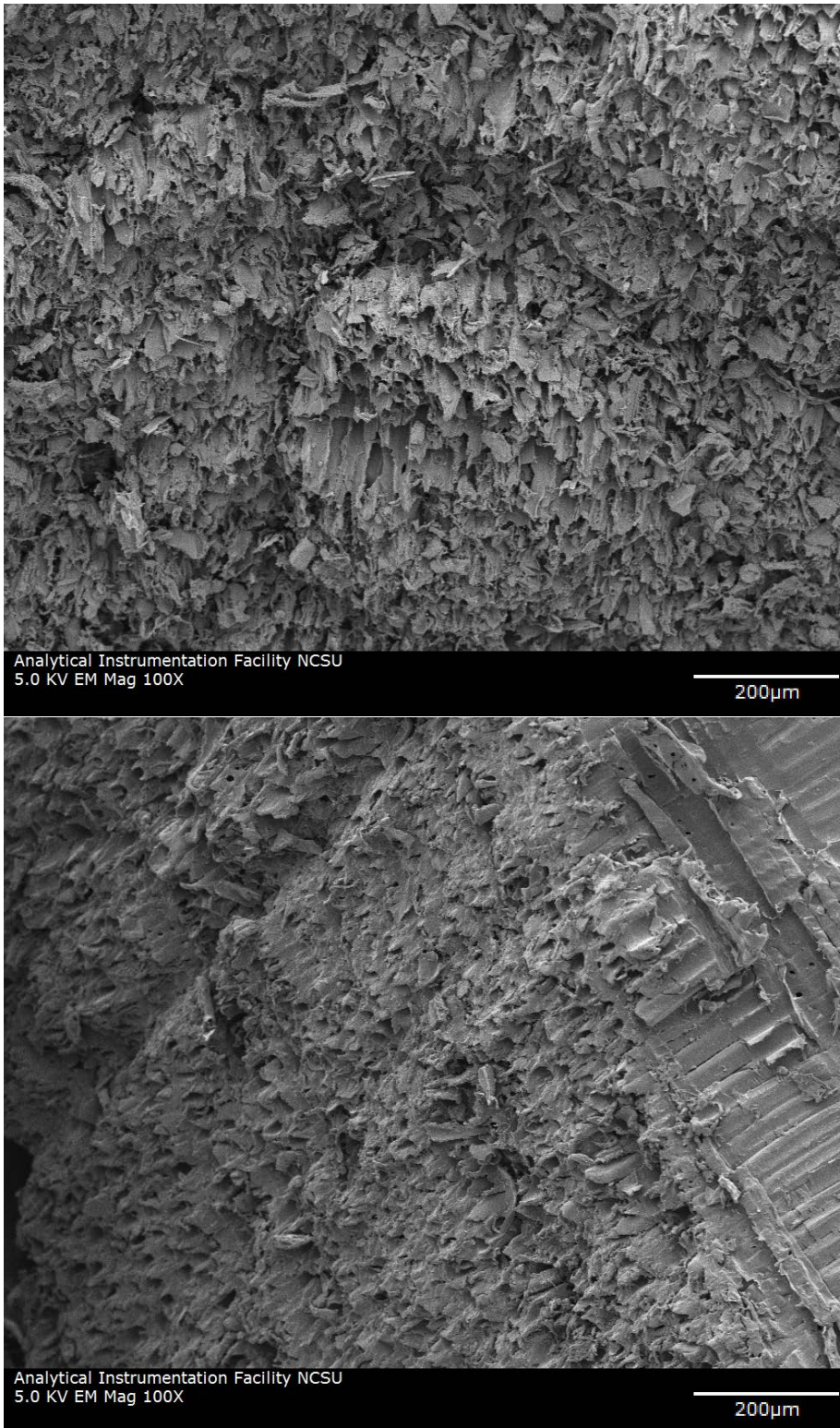
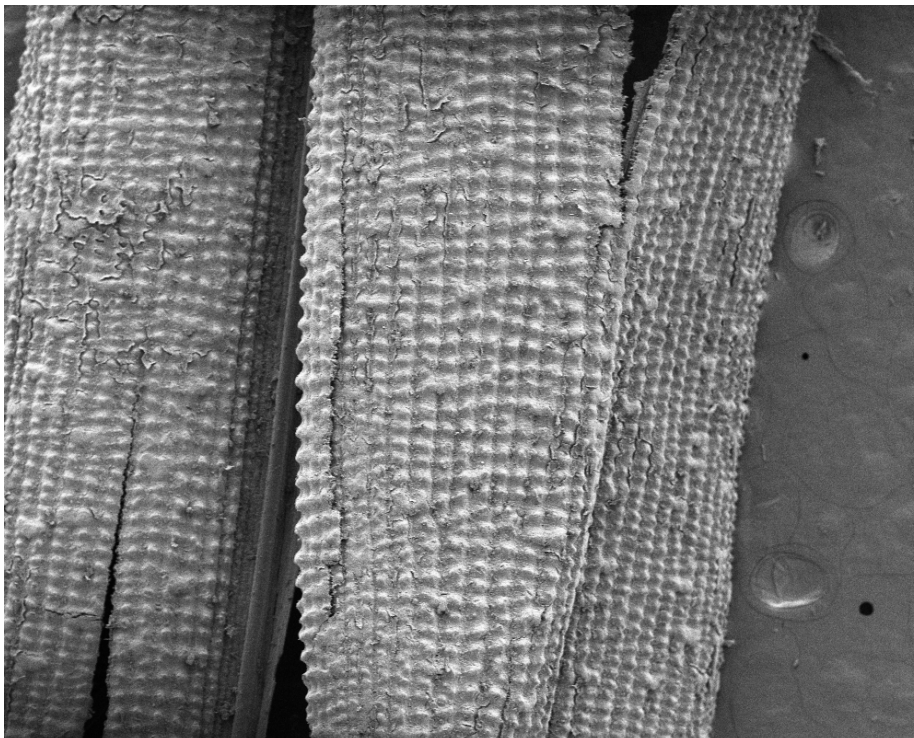


Figure 3. Surface view of (A) pine wood chips (PWC) and (B) pine wood biochar (PBC) at 100x magnification.



Analytical Instrumentation Facility NCSU
5.0 KV EM Mag 30X

500µm



Analytical Instrumentation Facility NCSU
5.0 KV EM Mag 30X

500µm

Figure 4. Surface view of (A) rice hulls (RH) and (B) rice hull biochar (RBC) at 30x magnification.

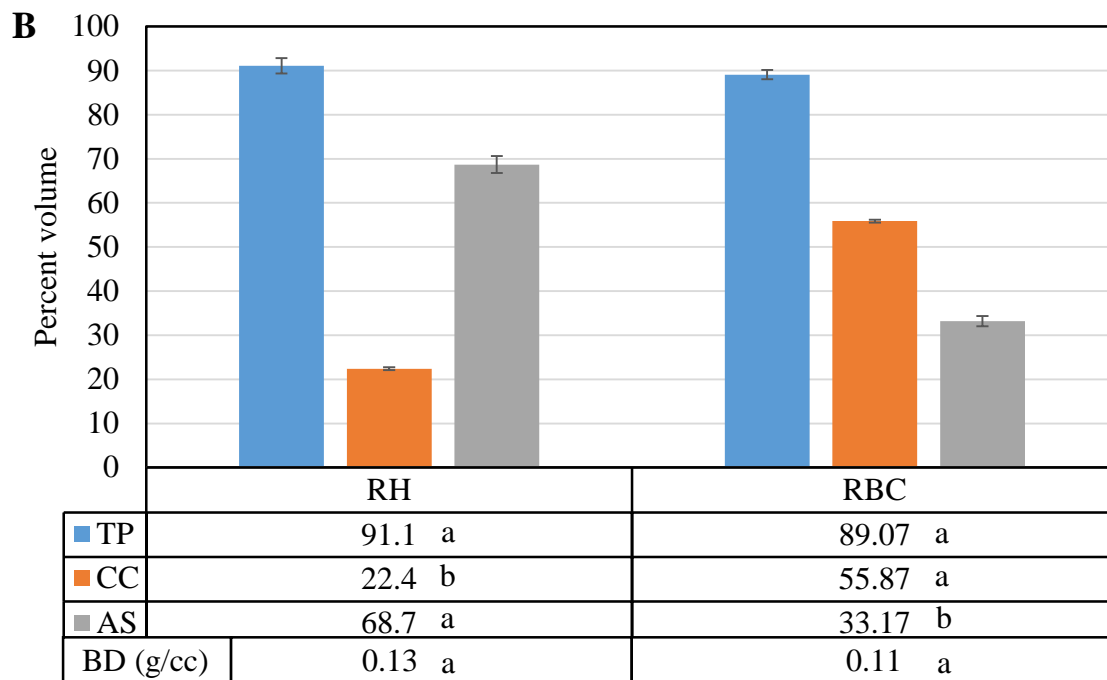
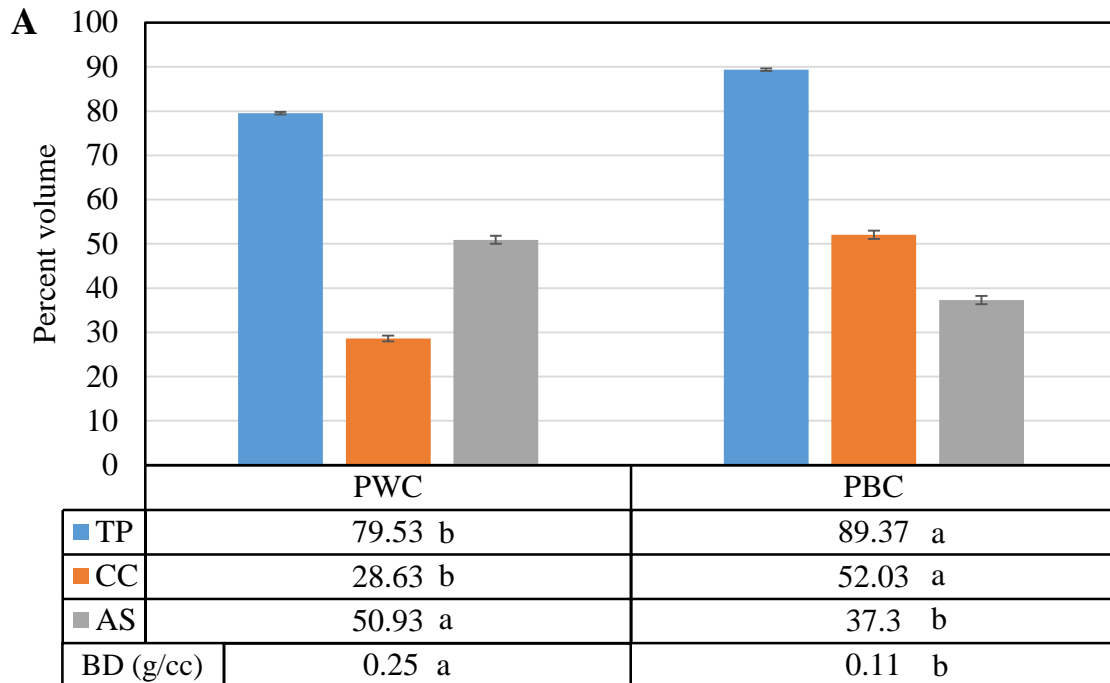


Figure 5. Physical properties including total porosity (TP), container capacity (CC), air space (AS) and bulk density (BD) of (A) pine wood chip (PWC) feedstock and wood biochar (PBC) and (B) rice hull (RH) feedstock and rice biochar (RBC) were collected from three samples from each substrate blend and represented as means. Analysis performed using the North Carolina State University Porometer Method (Fonteno, 1996). Means separated by each physical property by Least Significant Difference (LSD) at $P \leq 0.05$; means followed by the same letter are not significantly different. Standard error bars are shown to indicate sample variation.

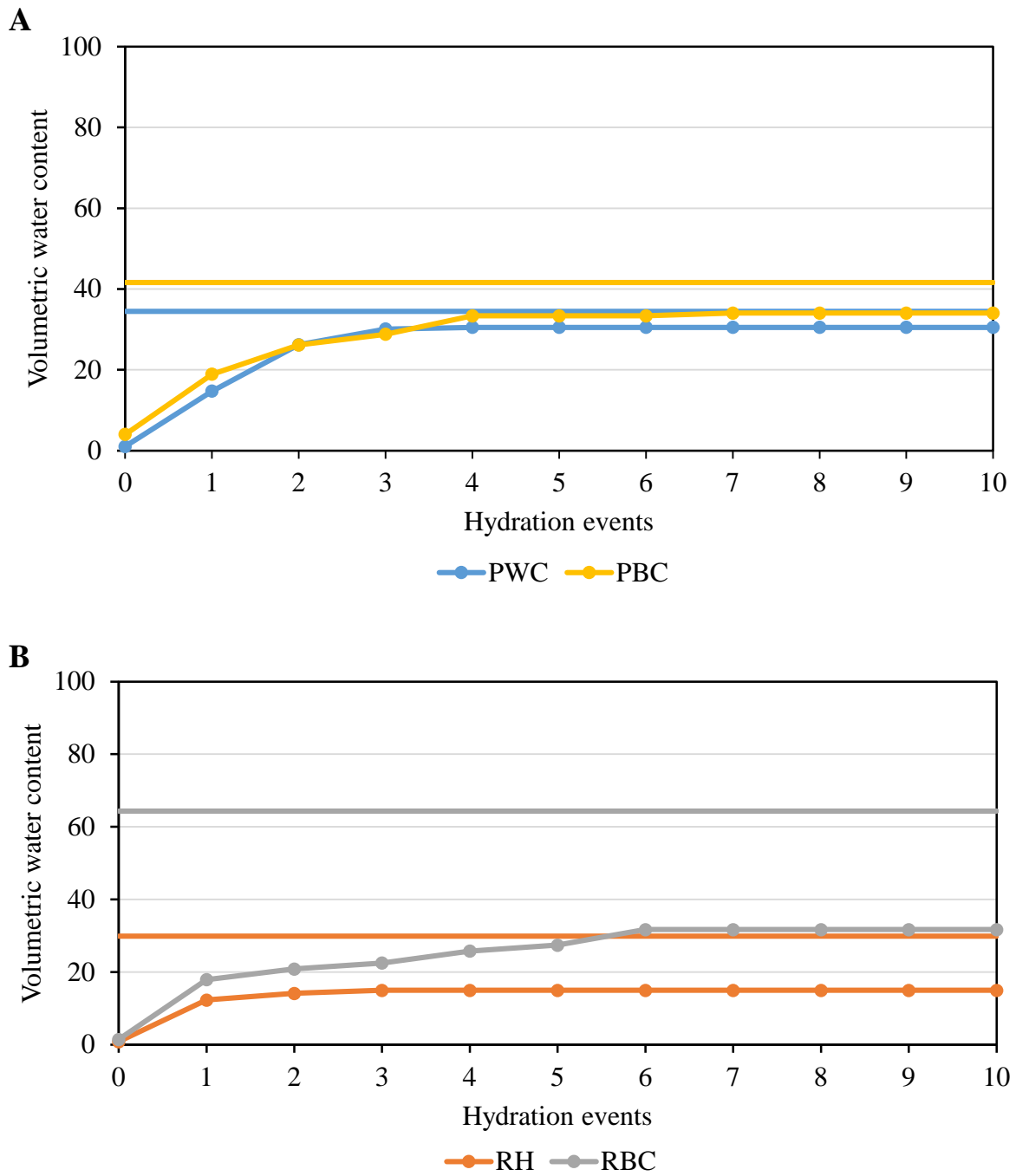


Figure 6. Hydration efficiency curves for (A) pine wood chip feedstock (PWC) compared to biochar produced from pine wood chips (PBC) and (B) rice hull feedstock (RH) compared to biochar produced from rice hulls (RBC) with container capacity represented as solid lines for each material.

CHAPTER 3

Physical and Chemical Effects of Two Consistently Produced Biochars Amended to Peat-Based Greenhouse Substrates

(In the format appropriate for submission to HortScience)

Physical and Chemical Effects of Two Consistently Produced Biochars Amended to Peat-Based Greenhouse Substrates¹

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Physical and Chemical Effects of Two Consistently Produced Biochars Amended to Peat-Based Greenhouse Substrates

Additional index words: gasification, liming, pH buffering, pine wood chips, rice hulls

Abstract. The potential benefits of biochar, including nutrient retention and exchange, has brought attention to the possibility of biochar as a component in horticultural substrates. In these studies, two biochars were produced from either pine wood chips (loblolly pine; *Pinus taeda* L.) or rice hulls using a top-lit updraft gasifier. The objectives of these studies were 1) to compare physical and chemical properties of two types of biochar with those of perlite when amended in peat-based substrates; 2) determine the effects of these biochars on pH of peat substrates over time; and 3) determine the interaction of biochar with calcitic lime additions on pH when amended to peat substrates. These biochars can match perlite in total porosity and air space, due to particle size, while having increased container capacity that hold more water for container-grown plants. Levels of phosphorus (P) and potassium (K) rose 1.5 to 10 fold with additions of pine wood biochar and rice hull biochar, respectively. Both biochars had high pH values (8.8-9.5 pH) but when amended to peat as an aggregate, had little direct effect on substrate pH. A laboratory experiment was conducted to determine the effect of pulverized biochars on the pH of peat. Even at a smaller particle size (<2.0-mm), these biochars still had little effect on peat pH with rates as high as 14.25 kg·m³. However,

when peat is amended with these biochars as an aggregate (at 10%-30% v/v) and calcitic lime is added from rates of 1.19-7.13 kg·m³, biochar can enhance the effect of lime on pH in fallow containers.

Introduction

The influence of biochars on soilless substrates can be traced back to Santiago and Santiago (1989) recommending a type of ‘charcoal’ as a substrate for potting mix because it acts as a sponge absorbing and retaining water, gases and solutions. However, even since 1989, biochars and their effect on greenhouse and nursery container substrates has been studied less than with biochar amended soils (Altland and Locke, 2012; Steiner and Harttung, 2014). In 2012, Cox et al. reported a summary of 33 papers on the use of biochar with horticultural crops in field soils with studies using corn, water spinach, cassava, banana and cashews (Cox et al., 2012). However, there is still little research reporting the effects of biochar on horticultural crops or container substrates for the horticultural industry; although there are some researchers that suggest the potential for horticultural use of biochar exists in the soilless substrates used for container production of greenhouse crops (Gu et al., 2013; Northup, 2013; Peterson and Jackson, 2014; Vaughn et al., 2015b).

There is potential for different biochars to positively impact plant growth, both increasing shoot and roots. Rice-husk char amended to Cambodian soils has been found to increase final biomass, root biomass, plant height and number of leaves of lettuce (*Lactuca sativa*) and Chinese cabbage (*Brassica chinensis*) plants (Carter et al., 2013). Pepper (*Capsicum annuum*) plants showed an increase in plant growth and productivity with nutrient-poor, wood-derived biochar when amended to coconut fiber and tomato (*Solanum lycopersicum*) plants showed an increase in plant height and leaf size with the same biochar and substrate (Graber et al., 2010). Red oak biochar has been found to increase shoot biomass with hybrid poplar cuttings when amended to peat-based substrates (Headlee et al., 2014).

Biochar can also influence physicochemical properties when amended to container substrates. Cao et al. (2014) and Dumroese et al. (2011) reported biochar amended substrates had improved hydraulic conductivity, water retention and increase permanent wilting point, using urban green waste biochar in scoria green roof substrates and agricultural/forestry residue biochar amended to peat, respectively. Biochar produced from 70% agricultural waste (rice hulls, pecan shells, walnut shells and coconut shells) and 30% rubber car tires has been amended to green roof media (gravel, sand silt and clay) has reportedly decreased nitrogen (N), phosphorus (P) and organic carbon in runoff (Beck et al., 2011), the same has been shown with hardwood and softwood bark biochar amended to peat and peat-perlite substrates (Nemati et al., 2015). Reports of red oak biochar have shown significantly higher cation exchange capacity (CEC) and exchangeable nutrients when amended to peat and peat-vermiculite substrates (Headlee et al., 2014) and biochar produced from urban greenwaste has shown inherent nutrient content (Kaudal et al., 2015). Different biochars' pH can range from 5.4 to 10.3 (Nemati et al., 2015; Fornes et al., 2015; Spokas et al., 2012) and may have the potential to neutralize acidity caused by both peat and root activity (Bedussi et al., 2015). Gasified rice hulls amended to a commercial soilless mix of peat and perlite have shown potential for increased available phosphate and potassium (K), as well as potential for decreasing nitrate leaching from substrates (Altland and Locke, 2013a; Altland and Locke, 2013b). Decreasing nitrate or phosphate leaching from substrates may be due to the anion exchange capacity (AEC) of biochar, although the mechanism is unknown (Lawrinenko, 2014).

The potential increase in CEC and AEC of substrates amended with biochar would be beneficial. Increased CEC would aid in keeping cations, such as K, calcium (Ca) and

magnesium (Mg) that are important to plant growth in the substrate and surrounding substrate solution, increasing availability to the plants and exchanging vital nutrients. This would decrease nutrients lost through leaching, as would increasing AEC in the substrate lead to the potential of leaching fewer anions. If stable anion exchange sites exist on biochar surfaces, then substrates amended with biochars of high AEC should exhibit reduced leaching losses of nitrate and phosphate (Lawrinenko, 2014). Another possible benefit of amending biochar to peat-based substrates is the capacity for neutralizing the peat acidity and stabilizing the pH over time. This would create a better root environment for greenhouse/nursery plants in containers to grow in while also potentially decreasing the necessity or amount of other additions (e.g. lime, acidic/basic fertilizers) to the crop during production. Soilless substrate pH and pH buffering capacity are some of the important chemical properties for successful nutritional management in crop production (Huang et al., 2010). Differences in buffering capacity can occur depending on the source of peat moss used and other components in the substrate, and these differences could contribute to problems of rapid substrate pH changes during the course of crop production (Huang et al., 2010).

Biochar has been shown to be a potential alternative to perlite in greenhouse substrates. Biochar made from different feedstocks can still have similar particle size distributions to perlite, therefore adjusting substrate physical properties similarly (Nemati et al., 2015; Northup, 2013). Chemically, biochar is different than perlite but also adds beneficial improvements, such as increased CEC/AEC, increased pH, and increased nutrient retention, to peat-based substrates (Altland and Locke, 2013a; Diez, et al., 2013; Nemati et al., 2015; Steiner and Harttung, 2014). However, one major disadvantage of biochars is the

wide range of retail prices for the current available commercial products, from \$8.80-\$1482.80 per cubic meter, with the mean price for pure biochar being \$291.5/m³ (Jirka and Tomlinson, 2014). Compared to other substrate components such as peat (\$11-22/m³), perlite (\$30-40/m³) and pine bark (\$16.5/m³), biochar could be the most expensive component and the prices are also dependent on location and type/production of biochar.

Another issue found with biochar is the variation in the literature regarding its physical and chemical properties, due to highly inconsistent reporting of materials and methods for producing biochar. Production method, production parameters and feedstock type are not always sufficiently reported (Downie et al., 2009). The production of biochar needs to be fully explained and understood in order to have a more consistent product that is reproducible and economical. The physical and chemical properties of biochar need to be consistently measured to correctly characterize the biochar made from a particular feedstock and charring process with known parameters. It is also important to measure how the biochar will affect the substrate and root zone which ultimately impact plant growth positively or negatively. Determining total elemental composition and plant available nutrients will show which nutrients would exchange in the substrate solution as well as the potential to decrease the amount of fertilizer applied to the crops. Other variable properties (e.g. AEC, CEC, elemental composition) of biochar also need to be determined as they are inconstant due to the ranges of production and postproduction factors such as storage or activation (Spokas et al., 2012). Even with these differences in production method and feedstock, the end product is termed 'biochar'. Any organic material can be charred, by pyrolysis, gasification or torrefaction. The resulting biochar will have trace minerals, pH and specific surface

chemistry/physical attributes that reflect the original feedstock and process of production (Bates, 2010).

While various studies have reported improvements to plants and root environment, there are just as many reports showing decreased or no improvement to plant growth (Cao et al., 2014; Conversa et al., 2015; Gu et al., 2013; Steiner and Harttung, 2014; Vaughn et al., 2015a), creating a summary of reports showing inconsistent benefits of biochar (Spokas et al., 2012). The limited data set or reports using biochar in soilless substrates contributes to the difficulty of drawing any conclusions on biochar effectiveness in horticulture (Cox et al., 2012). Future research will need to explore the impact of the vast range of biochar properties on their potential use in greenhouse and nursery container production (Altland and Locke, 2012).

The objectives of these studies were 1) to measure the physical/chemical properties of two types of biochar and perlite when amended to peat-based substrates in order to determine validity of biochar as a perlite replacement; 2) to determine the effects of these two biochars on pH of peat substrates over time; and 3) to measure the interaction of biochar with calcitic lime additions on pH when amended to peat substrates in order to determine the potential of biochar as a lime replacement.

Materials and Methods

Biochar production. The biochars were produced at NC State University with a top-lit updraft (TLUD) gasifier, a 2.5-m high and 1-m diameter black iron drum (Fig. 1A). The feedstock received for the charring process were pine wood chips and rice hulls. Loblolly pine wood chips (*Pinus taeda* L.) sized to 6.35-mm chips were purchased (West Farms Wood Products LLC, Newton Grove, NC). Rice hulls were purchased (Riceland Food Inc.,

Stuttgart, AR) and both feedstocks were stored on pallets under shelter. A conveyor was used to fill the reactor to ensure level placement of the total 1.5-m³ of feedstock material loaded in the reactor. The feedstock was then ignited with lighter fluid sprayed in three concentric circles on top of the feedstock and the lid of the reactor was sealed shut to control the oxygen in reactor/combustion of the feedstock. Combustion was sustained by regulating the amount of air entering from the bottom and passing up through the material (12-m³·min⁻¹). A vent in the lid of the reactor allowed combustible gas from the process to leave the system, and this gas was flared (ignited) to reduce the amount of smoke and odor produced (Fig. 1B & 1C). Once the flame front reached the bottom of the gasifier, the air flow was shut off and compressed nitrogen gas was then forced through the biochar in the reactor from the bottom for 24 h to prevent any flare up as the biochar cooled. This process was then repeated for the second feedstock (rice hulls). Once cooled the pine wood biochar (PBC) or rice biochar (RBC), was removed from the reactor and stored in 1.5-m³ industrial bags under shelter.

Internal temperature in the middle of the reactor was recorded with a data logger (Measurement Computing, model: USB-5201, Norton, MA). For the pine wood chips, the maximum internal temperature was 745°C and 705°C for the rice hulls. The entire gasification process took 4.68 h for pine wood chips and 2.97 h for rice hulls. The total mass of pine wood chips loaded into the reactor was 746 kg and only 645 kg for rice hulls. The final mass of the PBC (after charring) was 528 kg, indicating 29% of the biomass was consumed. The final mass of RBC was 542 kg, showing a 16% loss of biomass.

Biochar as a substrate component. Peat moss (Berger Tourbe de Sphaigne Blonde Golden; BP-P; Quebec, Canada) was amended with either perlite (PL), PBC or RBC at rates of 90:10, 85:15, 80:20, 75:25, and 70:30 (v/v) peat : amendment. Fifteen substrates were

mixed for testing physical and chemical properties on 11 Aug. 2015 and tested for initial pH with a pH meter (HI 9811, Hanna Instruments, Ann Arbor, MI) using the 1:1 dilution method; one part material (100 mL) was mixed vigorously with one part deionized (DI) water and allowed to equilibrate for 15 min (Lang, 1996).

Physical properties including air space (AS), container capacity (CC), total porosity (TP) and bulk density (BD) were determined for each substrate blend, using the North Carolina State University Porometer method (Fonteno, 1996). To determine particle size distribution (PSD) of the biochar and perlite, three samples of each aggregate from each batch run were dried at 105° C for 48 h and placed in a Ro-tap Shaker (Model B, W.S. Tyler, Mentor, OH) fitted with six sieves; 6.3 mm, 2mm, 0.71 mm, 0.5 mm, 0.25 mm, and 0.106 mm for five min. The sample from each sieve was weighed, and particle size was expressed as a percentage of the total weight of the sample. Properties and PSD were determined using three representative samples of each blend.

Samples of each substrate blend were submitted to the North Carolina Department of Agriculture and Consumer Services (NCDA), Agronomic Division (Raleigh, NC) for plant available nutrient analysis. Plant available nutrients were determined at NCDA by saturated media extract and vacuum filtration, the solution was analyzed for nitrate (NO₃-N) by nitrate-hydrazine reduction (Kempers, 1988); NH₄-N was determined by a modified Berthelot reaction (Krom, 1980); and urea concentration was determined with the diacetyl monoxime thiosemicarbazide colorimetric method (Sullivan and Havlin, 1991) with an auto-flow spectrophotometric analyzer (San++ Segmented Flow Auto-Analyzer, Skalar Instruments; Breda, The Netherlands). Total concentrations of P, K, calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), and sodium (Na)

are determined from the solution of saturated media extract with Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES; Optima 8000 PerkinElmer, Waltham, MA).

Three representative samples of each substrate blend were tested for pH buffering capacity, as well as three samples of 100% biochar from both PBC and RBC. All samples (1.5 g equivalent dry weight) were diluted to 50 g with degassed DI water and were titrated with 0.1 N hydrochloric acid (HCl) or 0.1 N sodium hydroxide (NaOH) using an automatic titrator (Titralab[®] 856, Radiometer Analytical SAS, Villeurbanne Cedex, France). The substrate blend suspensions were taken through two titration cycles between pH 3 and 11; the first cycle aimed to hydrate the material and expose cation exchange sites and involved increasing the pH from its ambient level near pH 9 up to pH 11 and then decreasing the pH to 3. The second cycle was used to generate the pH buffering curve. The parameters of the titration process follow the outline proposed by Barnes (2013).

The CEC of the substrate blends were measured based on procedures described by Kloss et al. (2012) and Dumroese et al. (2011). Initially, 2 g of substrate and 40 mL of degassed DI water were mixed and allowed to saturate overnight. Samples were then vacuum filtered (Whatman[®] Qualitative #1) and the substrate was placed in a flask with 20 mL of 0.2 M barium chloride (BaCl₂) and mixed for 2 h on an oscillating shaker (G10 Gyrotory[®] Shaker; New Brunswick Scientific Co., Edison, NJ). The final solution was filtered with 0.45- μ m nylon filters (25mm syringe filters; Fisher Scientific, Waltham, MA). The concentrations of Na, K, Mg, Ca, Al, Fe and Mn were measured via ICP-OES (Perkin Elmer 8000, Waltham, MA). The AEC of the substrate samples was determined according to the procedure described by Lawrinenko (2014). One gram samples of substrates were mixed

with 40 mL of degassed DI water and 2 mL of 1 M potassium bromide (KBr) and placed on an oscillating shaker for 48 h. Substrate residue was then vacuumed filtered and rinsed with DI water two or three times until conductivity of the solution was $\leq 5\mu\text{S}$ (HI 9813-6, Hanna Instruments, Ann Arbor, MI). Next, 2 mL of 2.5 M calcium chloride (CaCl_2) was added to the substrate slurry which was then mixed for 48 h. The concentrated solution was diluted with 200 mL of DI water; 10 mL of this solution was then filtered with 0.45- μm nylon filters. Bromide was detected from the final solution using an Ion Chromatograph (Dionex 500, Thermo Fischer Sci, Sunnyvale CA). AEC and CEC calculations were performed following the methods described by Coleman et al., (1959).

Pulverized biochar study. Both PBC and RBC biochars were pulverized to $<2\text{mm}$ using a hand mill (4-F hand mill; Quaker City Mill, Philadelphia, PA). The pulverized biochars were visually comparable to a powder-form of biochar, as seen in Fig. 2. The control was 100% peat and no biochar additions. The pulverized pine wood biochar (PBC_p) and rice hull biochar (RBC_p), were blended similarly as a dolomitic lime (#100 mesh) substitute and added to the peat substrate. On 14 July 2015, PBC_p or RBC_p was added to 0.014- m^3 of peat at 1.78, 3.56, 5.34, or 7.13 $\text{kg}\cdot\text{m}^3$, and each substrate was placed into a clear, resealable plastic bags (#35XE41 1.8mil LDPE; Grainger, Raleigh, NC). The substrates were allowed to equilibrate for 24 h, and pH was then measured daily for the next six days. Three representative samples of 100 mL from each bag were used for the 1:1 dilution method off pH measurement; one part material (100 mL) was mixed vigorously with one part deionized (DI) water and allowed to equilibrate for 15 min (Lang, 1996). On 22 July 2015, the experiment was repeated with higher rates of PBC_p or RBC_p addition at 0, 10.69,

12.47, or 14.25 kg·m³. These substrates were measured every 24 h for three days to observe if the higher rates of pulverized biochar would raise the pH of the peat moss.

pH enhancement study. Seven substrates were used in testing the responsiveness of pH with biochar/peat substrates; 100% peat, 90% peat amended with 10% PBC or RBC, 80% peat amended with 20% PBC or RBC, and 70% peat amended with 30% PBC or RBC. Substrates were mixed on 7 Jan 2016 and allowed to equilibrate for 24 h when initial pH was determined (Day 0). Each substrate was separated into 15 clear, resealable plastic bags with 177 cm³ in each bag. Three bags from each substrate were then grouped together to receive calcitic lime (#200; Mississippi Lime Company, Vicksburg, MS) at one of the five following rates: 0, 1.19, 2.38, 4.75, and 7.13 kg·m³. The pH of every substrate was measured 24 h after the addition of lime (Day 1), then 48 h (Day 3), and 96 h later (Day 7) with weekly measurements until Day 28, with final measurement at Day 42. Three samples were measured from every substrate bag resulting in nine samples from each treatment, and pH was measured using the 1:1 dilution method.

The 70% peat amended with 30% PBC or RBC substrates were used to grow marigolds (*Tagetes erecta* ‘Inca Orange’) and tomatoes (*Solanum lycopersicum* L. ‘Better Boy’). Marigold and tomato seeds were sown into a 288-plug tray (1.5 L × 1.5 W × 3.5 H-cm) containing (by volume) 65% peat : 20% PL : 15% vermiculite (Fafard Super Fine Germination Mix; Sungro Horticulture, Agawam, MA) on a greenhouse bench in Raleigh, NC, on 2 Nov 2015. On 18 Nov 2015 substrates were mixed and amended with 2.38 kg·m³ of calcitic lime (#200), then greenhouse containers (12.7-cm dia; Dillen Products, Middlefield, OH) were filled to the top with each substrate and lightly tapped three times to settle the substrate. One plug of each species was planted into the center of the container with six

replications of each substrate/species. Planted containers were placed on a greenhouse bench, randomized, and fertigation was supplied every day for 3 min with 200 mg·L⁻¹ nitrogen derived from 20N-4.4P-16.6K (Peatlite Special, Peters Professional; The Scotts Co., Marysville, OH) injected at 1:100 ratio by a Dosatron injector (D14MZ2; Dosatron International, Inc., Clearwater, FL) using low-volume spray stakes (PC Spray Stake; Netafilm, Ltd., Tel Aviv, Israel). Pour-throughs were conducted weekly, from 7 days after planting (DAP) to 28 DAP, to measure the pH of every planted container according to the pour-through extraction procedure (Wright, 1986).

Statistical analyses. Data from analyses and studies were subjected to the general linear model (GLM) procedures and means were separated by Least Significant Differences (LSD) at $P \leq 0.05$ (SAS Institute Version 9.3, Cary, NC). Additionally, the pulverized biochar study and the pH enhancement study were subjected to general linear model procedures when comparing across all substrates, Dunnett's test to compare to a single control ($P \leq 0.05$), and linear regression for pH over time.

Results and Discussion

Biochar as a component: Physical properties. Although there were slight differences in TP among the blends, both biochars showed similar values for TP compared to PL (Table 1). Perlite values ranged from 88.5% to 90.9%, while both biochars ranged from 90.6% to 93.8% volume. All bulk densities were also similar and ranged from 0.10 to 0.12 g/cc in all treatments. Container capacities also varied little, with perlite additions having CC's of 59.9% to 63.6% volume and both biochars measuring 62.9% to 67.7%. Air space was similar among the blends, with all treatments ranging from 24.1% to 30.9% volume. All CC and AS

measurements were within 5% or 6% of each other, indicating both biochars produced similar properties at all combinations compared to PL.

Container capacity for all substrates are similar to CC reported by Nemati et al. (2015) for peat amended with hardwood or softwood bark biochar (54%-65% CC), however TP and AS was much higher than Nemati et al. (2015) substrates (74%-79% TP; 12%-19% AS). The only differences seen for TP was among the substrates at the rate of 85 peat : 15 amendment and 75 peat : 25 amendment, with the PBC having a larger TP than PL at both rates (Table 1). This could be due to PBC having more particles <2.0-mm and >0.71-mm. Container capacity was different among the substrates at the rate of 90 peat : 10 amendment and 80 peat : 20 amendment, with the amendment PBC having a larger CC than PL at 10% rate and the RBC amendment having a larger CC than PL at 20% rate. This could be due to the higher percentage of particles 0.5-mm and smaller for RBC.

Perlite and PBC had the largest percentage (55% and 53%, respectively) of particles >2.0-mm, however both biochars, PBC and RBC, had more particles <2.0-mm and >0.71-mm (43% and 39%, respectively; Fig. 3). Rice hull biochar had a greater percentage of particles <0.71-mm (30%) and >0.25-mm (24%), and most of the RBC can be found from 0.71-mm to 0.25-mm with a total of 93%. The greatest portion of PL and PBC ranges from 2.0-mm to 0.71-mm with totals of 82% and 96%, respectively. For the smallest fine-sized particles (<0.106-mm), PL had a greater percentage with 3% compared to 1.5% for PBC and 1% for RBC.

Total porosity, CC and AS are within the recommended ranges for those properties; 80%-90% TP, 60%-70% CC and 10%-20% AS (Nelson, 2011). Comparing among all substrates for CC, 10% PBC had the largest percentage and was different from all rates of

PL, 15% and 30% RBC, 25% and 30% PBC (Table 1). At the lowest amendment amount of 10%, PBC created more pore spaces for water than all the PL substrates. Air space was not different among the substrates at any ratio of peat : amendment; comparing among all substrates for AS only 20% PL was different from 25% PL and 10% RBC. It is important to note not only the differences in PSD, as well as the general differences in observation of the particle shapes for PL, PBC and RBC (Fig. 4). The PL and PBC are the closest in shape but PL is more rounded whereas PBC is more cubic. The RBC is noticeably flat and elongated in shape. Biochar, or any material for that matter, can be processed in ways to produce materials of different sizes and shapes that will alter the air and water properties accordingly.

Biochar as a component: Chemical properties. Pine wood biochar reduced N content at all blend ratios over perlite (Table 2). Although the numbers are small, the concentrations were reduced by half (e.g., 12 to 6 mg·L⁻¹). At 20% and above, the RBC also showed reduced N, although not as great a reduction. These reductions in N could be due to the biochars consuming the N in the substrate/peat, as N was removed from the feedstock as a result of the thermochemical degradation of biomass (Rivas, 2015). Phosphorous was mostly unaffected by adding PBC, however, the rice hull char provided a 4 to 9 fold increase in P as the amount of RBC increased in ratio. The greatest increase of plant available nutrients came in additional K. Potassium increased from 3 to 13 mg/L in PBC and 3 to 39 mg/L with additions of RBC. The amount of K available is similar to substrates amended with biochar made from hardwood logs and softwood bark (Nemati et al., 2015). Comparing among all substrates, all percentages of RBC have higher amounts of P and K than the other substrates. Altland and Locke (2013a) found that using gasified rice hull biochar in substrates increased the amount of P and K and suggest that rice hull biochar can provide sufficient P and K for container-

grown plants over a six week production cycle. At all rates, the PL amendment had higher amounts of plant available Ca, Cl, Mg, and Na compared to the other substrates. The PBC amendment had higher amounts of plant available B at all rates compared to the other substrates.

The initial pH of all substrate blends was low (3.8 to 4.53; Table 3), and none of the pH values from initial to 31 days later were above 4.83, which is lower than the recommended range for soilless substrates (5.4-6.6) but within the range for acid-loving crops (4.5-5.8; Nelson, 2011). For the initial pH, all blends with PL were lower than the other substrates. After 31 days, pH had linearly risen for every substrate blend, with all PBC blends higher in pH than the other substrates. The pH buffering capacities of 100% biochar were low, indicating that they would not have a significant effect on pH of peat that is already acidic in nature, even when peat is amended with 10% PL (v/v; Fig. 5). If the titration curves of the biochars were above the titration curve of 10% PL, this would indicate that less biochar can be used to raise the pH of peat. Calculating from the titration curves, 4.3 kg of PBC and 1.2 kg of RBC would need to be added to bring the 10% PL substrate to a pH of 6; which would be equal to 1 peat : 4 PBC or 1 peat : 1 RBC by volume.

The pH buffering capacity was lower for PBC at all rates except for the 10% rate (Table 3). Perlite seemed to have equal or better buffering capacity than either biochar. The pH buffering capacities of the 70 peat : 30 amendments and 75 peat : 25 amendments were all similar to reported values for a soilless mix of peat, perlite and dolomitic lime at a pH of 4.96 (9.8 meq·L⁻¹; Huang et al., 2010); however the pH buffering capacities of the substrates with 10%, 15% and 20% amendments were low and would fall below the recommended rates for soilless substrates. In the range of pH important for plant growth in substrates (5.4-6.6), the

biochar substrates at all rates do not provide enough pH buffering capacity; there seems to be greater pH buffering occurring at pH >7.0 where titration curve for the substrates show more OH⁻ is required to change the pH (Fig. 6).

The CEC of peat : perlite mixes was 10 to 12 mmol·100cc⁻¹ (Table 4). Pine wood biochar CEC ranged from 14.5 to 17.9, only a slight increase, while the rice hull char was more variable and slightly lower. Depending on the organic substrate components, such as peat, pine bark or composts, CEC may be pH dependent and can increase with increasing pH (Silber, 2008). Biochar has the potential to retain important nutrients for plants, with increased CEC this decreases nutrient leaching (Nemati et al., 2015). All substrates are similar or higher than several other greenhouse components (e.g. pine bark), which have CECs between 6-15 mmol·100cc⁻¹ (Nelson, 2011; Table 4). This range for substrate CEC is considered desirable for greenhouse root substrates and higher levels are not common but also desirable (Nelson, 2011).

Anion exchange capacities tended to increase with increasing ratios of amendments for PL, PBC and RBC. The AEC generally dropped with RBC additions. These AEC sites can be significant in the retention of sulfate, phosphate and nitrate, and allow for greater N availability to plants and less leaching in production situations (Altland et al., 2008). However, AEC has been shown to increase with decreasing pH (Altland et al., 2008; Lawrinenko, 2014) and greenhouse substrates for non-acid-loving crops are recommended at 5.4 to 6.6 which is only slightly acidic but not acidic enough to full capitalize on AEC of most components. Different biochars, including gasified rice hulls, sawdust biochar and bark/wood biochar, have been reported to initially leach less nitrate when amended to soilless substrates, and biochars are speculated to have an effect on moderating nitrate and phosphate

levels within a substrate (Altland and Locke, 2013b). With the addition of biochar to peat, especially increasing the ratio to 20% or 30% increases the AEC to a level that has potential to retain important anions for plant growth.

Pulverized biochar study. Different biochars have shown potential as a replacement for lime in greenhouse substrates, especially with biochars at different particle sizes. For PBC_p, pH was increased initially from 3.78 (control) to only 4.06 (at 7.13 kg·m³; Fig. 7A). Even after 7 days, the maximum pH attained was 4.12. The rice hull biochar had a similar, albeit lower effect, ranging from 3.78 (control) to 3.92 (at 5.34 and 7.13 kg·m³) initially with a maximum pH of 4.03 during the 7 day incubation period (Fig. 7B). Biochar made from hardwood increased pH to or above the recommended range of 5.4 to 6.6, with smaller particles increasing the pH compared to the larger sizes (Northup, 2013). This was not seen with pulverizing PBC and RBC, although pulverizing the biochars increased the pH for PBC from 8.7 to 9.4, however there was no change in pH for RBC (pH of 9.5). The pH of biochar can change depending on the temperature and air flow through the production system, and Rivas (2015) reported having basic functional groups and no acidic functional groups on the surface of pine wood and rice biochar produced in a similar TLUD gasifier.

All substrates with the additions of PBC_p and RBC_p never reached the recommended pH range for substrates, while also showing a trend of decreasing pH after Day 6 (Fig. 7). At a rate of 1.78 kg·m³, both PBC_p and RBC_p were similar to the control (peat with no additions) at all days with the exception of RBC_p at Day 6. Initially, at Day 1, only 5.34 and 7.13 kg·m³ rates for PBC_p and RBC_p were different from control and this continued for the rest of the measurement days for PBC_p only. Increasing the additions of PBC_p and RBC_p to 10.69, 12.47 and 14.25 kg·m³ were then used, and all substrates with PBC_p and RBC_p had

higher pHs than the control, but only reached a pH of 4.4 and 4.05 respectively (Fig. 8). Total elemental analysis of these chars showed Ca and Mg levels of 1,462 and 719 mg·L⁻¹ for PBC and 5,167 and 1,200 mg·L⁻¹ for RBC, respectively (Judd, 2016). However, the water soluble levels of Ca and Mg were 34 and 16 mg·L⁻¹ for PBC and 5 and 16 mg·L⁻¹ for RBC, respectively. When blended at 10 to 30% with peat those water soluble values are between 1 and 2 mg·L⁻¹ for both chars (Table 2). There seems to be little help for increasing pH directly with the addition of these two biochars, due to the pH buffering capacity and nutrient content.

pH enhancement study. Some biochar types have been used on their own as a lime substitute (Steiner and Harttung, 2014). However, the pH of the substrates used in these experiments were not in the recommended range for greenhouse substrates. Lime was added at 1.19 and 2.38 kg·m³ to both of the 30% biochar substrates and 100% peat. The pH achieved with the lower lime rate was almost a full pH unit higher (3.87 to 4.7) in the 30% PBC compared to the 100% peat (Fig. 9). The pH of peat with the lower lime rate averaged about 4.5 for 42 days, while the PBC substrate was 5.5 to 6.0 with the same lime treatment, reaching the recommended pH range for greenhouse substrates. The higher lime rate of 2.38 kg·m³ was again almost a full pH unit higher with 30% PBC going from 6.0 to 6.7 in 28 days (Fig. 9). This reaction occurred with the same biochar materials that had essentially no effect on pH by themselves (without lime; Table 5). At higher lime rates of 4.75 and 7.13 kg·m³, 30% PBC had pH values up to 8.11 for Days 1 – 42 (Fig. 10). The pH of 30% PBC amended at 2.38 kg·m³ had similar pH values over time compared to 100% peat amended at 4.75 kg·m³. Clearly, the effect of the lime was enhanced by the presence of the PBC.

RBC at 30% also had higher pH compared to the 100% peat substrates at 1.19, 2.38 and 4.75 kg·m³ (Figs. 11 & 12). However, the stimulatory effect of the char was not seen at the 7.13 kg·m³ rate. There were similar trends between 30% PBC and RBC pH values. Both lime rates of 1.19 and 2.38 kg·m³ had higher pHs with 30% RBC compared to the 100% peat (Fig. 11). The pH of 30% RBC with 2.38 kg·m³ had similar or higher pH values than 100% peat with 4.75 kg·m³ of lime. The 30% RBC with 1.19 kg·m³ lime reached the recommended range at Day 7 and continued until Day 42, whereas 30% with 2.38 kg·m³ lime reached the recommended range at Day 1 and continued. The difference between 30% RBC and 100% peat at 7.13 kg·m³ lime showed to have the least difference in pH, with Day 1 through Day 42 having differences of 0.16 to 0.26 in pH (Fig. 12).

The 10 and 20% of both biochar substrates in the study had pH values that were all higher than the 100% peat every day for 28 days (Table 6). This effect on pH is seen as an enhancement of the lime reaction, since the biochar itself had almost no influence on substrate pH in the above experiments.

The amount of lime added to greenhouse substrates can depend on the components in the substrate, however some peat-based substrates require 5.34-8.90 kg·m³ of lime (Owen et al., 2014). Both of these chars affected the lime reaction without influencing the pH directly. This is the first report of this process.

The 30% PBC substrate, amended with 2.38 kg·m³ and planted with marigolds or tomatoes, started with a pH similar to the 30% PBC (incubated and fallow) amended with the same rate of lime (Fig. 13A), however over time the pH of the substrate containing plants decreased in the production setting. The pH of the PBC substrate growing marigolds decreased about 0.7 pH units, similar to the decrease for the tomato plants in the same

substrate. The same trend was seen with 30% RBC substrate, the fallow substrate amended with $2.38 \text{ kg}\cdot\text{m}^3$ had a similar beginning pH as the 30% RBC substrate growing marigolds and tomatoes. The fallow substrate increased in pH over time but the marigold-planted substrate decreased in pH by 0.6 and the tomato-planted substrate decreased by 0.4 (Fig. 13B). The fallow substrates indicated an enhancement of lime through the biochars, however when in a production setting the conditions (fertilizers, plants, environment) decreased this enhancement and pushed the pH back down.

Conclusion

Biochars at 10 to 30% volume additions did not appreciably change the physical properties compared to equivalent perlite additions; Northup (2013) working with hardwood biochar also recommended 30% biochar with 70% peat for physical properties. Adding the chars actually lowered the waterextractable N levels in all mixes compared to perlite. This may be due to the chars immobilizing more N. However, these values were low (15 to $5 \text{ mg}\cdot\text{L}^{-1}$). PBC did not affect P levels, but RBC increased them 4 to 10 fold from 3 to $40 \text{ mg}\cdot\text{L}^{-1}$, and Altland and Locke (2013b) reported gasified rice hulls can provide a portion of the P and K for plants and lessen the need for P/K fertilizers. The pHs of PBC and RBC were 8.7 and 9.5, respectively (Judd, 2016). However, there was little available Ca or Mg (Table 2). There was also no improved pH buffering capacity with the chars, or even a reduction of buffering when blended with peat. Therefore, it was unusual, but not surprising, that neither of these chars altered pH of the mixes, either as a pulverized “lime-like” addition or as a component from 10 to 30% volume. The surprise was the apparent pH enhancement effect when combining char and lime together. The char seemed to increase the lime reaction, as if more lime was being added, often increasing the pH well over a full pH unit.

From this data and others, it is clear that biochars made from different feedstocks can produce different benefits and results. The addition of biochar to mineral field soils is considered a long-term effect, i.e., a practice that will have effects for many years. However, most horticultural substrates are designed to be used on crops as short as 4 to 15 weeks in greenhouse production and one to three years in woody nursery production. Couple that with the extra demands placed on growing plants with better temperature, photoperiod and fertility controls, not to mention the high cost of the char itself, it is not surprising that, to date, the benefits for improvement with biochar are variable and therefore harder to realize in horticultural substrates.

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Table 1. Physical properties of substrate blends made with peat amended with either perlite (PL), pine wood biochar (PBC) or rice hull biochar (RBC).

		Physical properties ^z			
Ratio	Substrate	Total porosity ^y	Container capacity ^x	Air space ^w	Bulk density ^v
(v/v)		(% vol)			(g·cm ⁻³)
90:10	Peat:PL	90.60 a ^u	63.60 b	26.93 a	0.10 b
	Peat:PBC	93.80 a	67.70 a	26.10 a	0.11 a
	Peat:RBC	90.57 a	66.50 ab	24.10 a	0.10 b
85:15	Peat:PL	89.47 b	61.73 a	27.73 a	0.10 a
	Peat:PBC	91.70 a	65.40 a	26.30 a	0.10 a
	Peat:RBC	90.60 ab	62.90 a	27.73 a	0.10 a
80:20	Peat:PL	90.90 a	59.97 b	30.90 a	0.10 a
	Peat:PBC	91.33 a	63.23 ab	28.10 a	0.10 a
	Peat:RBC	91.70 a	64.50 a	27.23 a	0.10 a
75:25	Peat:PL	88.53 b	63.20 a	25.33 a	0.11 a
	Peat:PBC	93.73 a	64.37 a	29.37 a	0.10 a
	Peat:RBC	92.53 ab	65.60 a	26.97 a	0.11 a
70:30	Peat:PL	90.27 a	64.20 a	26.10 a	0.12 a
	Peat:PBC	91.43 a	63.33 a	28.13 a	0.11 ab
	Peat:RBC	92.50 a	63.97 a	28.47 a	0.11 b

^zPhysical property data were collected from three samples from each substrate blend and represented as means. Analysis performed using the North Carolina State University Porometer Method (Fonteno, 1996).

^yTotal porosity = container capacity + air space.

^xContainer capacity is (wet weight – oven dry weight) ÷ volume of the sample.

^wAir space is the volume of water drained from the sample ÷ volume of the sample.

^vBulk density is the oven dry weight of the sample ÷ volume of the sample.

^uMeans separated within column, only between substrates of the same ratio, by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different (n=3).

Table 2. Plant available nutrients of substrate blends made with peat amended with either perlite (PL), pine wood biochar (PBC) or rice hull biochar (RBC). Nutrients include boron (B), calcium (Ca), chloride (Cl), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), nitrogen (N), phosphorus (P), potassium (K), sodium (Na), sulfur (S) and zinc (Zn).

Ratio (v/v)	Substrate	Plant available nutrients (mg·L ⁻¹) ^z												
		B	Ca	Cl	Cu	Fe	Mg	Mn	N	P	K	Na	S	Zn
90:10	Peat:PL	0.02 b ^y	1.40 a	10.97 a	0.00 a	0.06 b	1.59 a	0.03 b	14.90 a	3.35 b	3.33 c	7.52 a	8.52 a	0.01 a
	Peat:PBC	0.04 a	0.99 b	4.84 b	0.00 a	0.06 b	0.80 b	0.02 b	10.28 b	4.03 b	8.57 b	5.04 b	6.14 b	0.01 a
	Peat:RBC	0.02 b	0.97 b	4.07 b	0.00 a	0.08 a	1.07 b	0.04 a	13.35 a	12.40 a	15.70 a	4.35 c	7.91 a	0.01 a
85:15	Peat:PL	0.02 c	1.38 a	10.51 a	0.00 a	0.07 a	1.42 a	0.02 b	14.33 a	3.06 c	3.16 c	8.14 a	8.14 a	0.01 a
	Peat:PBC	0.04 a	0.77 b	4.51 b	0.00 a	0.06 a	0.58 c	0.02 b	8.52 b	4.01 b	10.00 b	3.70 c	5.03 b	0.01 a
	Peat:RBC	0.03 b	0.78 b	3.14 c	0.00 a	0.06 a	0.91 b	0.04 a	12.74 a	15.27 a	20.20 a	4.72 b	7.44 a	0.01 a
80:20	Peat:PL	0.02 c	1.44 a	11.03 a	0.00 a	0.06 a	1.50 a	0.03 b	14.89 a	3.20 c	3.31 c	8.88 a	8.67 a	0.01 a
	Peat:PBC	0.05 a	0.68 b	4.00 b	0.00 a	0.06 a	0.42 c	0.02 c	6.82 c	3.99 b	11.27 b	3.02 c	4.29 b	0.01 ab
	Peat:RBC	0.03 b	0.83 b	1.77 c	0.00 a	0.07 a	0.90 b	0.05 a	10.96 b	20.43 a	28.43 a	5.10 b	7.78 a	0.00 b
75:25	Peat:PL	0.02 c	1.09 a	10.53 a	0.00 a	0.05 a	1.15 a	0.02 b	12.03 a	2.97 c	2.90 c	8.90 a	7.38 a	0.01 a
	Peat:PBC	0.05 a	0.61 b	4.21 b	0.00 a	0.06 a	0.34 c	0.01 b	5.95 c	4.29 b	12.30 b	2.79 c	3.57 b	0.01 a
	Peat:RBC	0.03 b	0.74 b	3.38 b	0.00 a	0.06 a	0.74 b	0.04 a	9.26 b	22.53 a	31.90 a	4.68 b	6.78 a	0.01 a
70:30	Peat:PL	0.02 b	1.17 a	9.60 a	0.00 b	0.07 a	1.06 a	0.02 b	11.16 a	3.29 b	2.91 c	10.27 a	7.26 a	0.02 a
	Peat:PBC	0.04 a	0.65 b	3.81 b	0.00 b	0.07 a	0.39 c	0.01 c	5.94 c	2.82 b	13.50 b	2.96 c	4.51 c	0.01 a
	Peat:RBC	0.02 b	0.72 b	3.26 b	0.01 a	0.07 a	0.63 b	0.05 a	8.05 b	27.10 a	39.10 a	4.48 b	6.33 b	0.01 a

^zPlant available nutrients measured with water extraction by the North Carolina Department of Agriculture and Consumer Services (NCDA), Agronomic Division (Raleigh, NC).

^yMeans separated within column by, only between substrates of the same ratio, by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different (n=3).

Table 3. pH and pH buffering capacity of substrate blends made with peat amended with either perlite (PL), pine wood biochar (PBC) or rice hull biochar (RBC).

Ratio (v/v)	Substrate	Chemical properties ^z				pH buffering capacity ^v mmol·kg ⁻¹
		Initial pH ^y	10 pH ^x	31 pH ^w		
90:10	Peat:PL	3.80 c ^u	3.77 b	3.97 c	L ^{***t}	2.76 a
	Peat:PBC	4.07 a	4.17 a	4.40 a	L ^{**}	1.26 a
	Peat:RBC	3.93 b	4.07 a	4.23 b	L ^{**}	1.52 a
85:15	Peat:PL	3.83 c	3.93 c	4.17 b	L ^{***}	6.94 a
	Peat:PBC	4.20 a	4.43 a	4.57 a	L ^{**}	4.52 b
	Peat:RBC	4.10 b	4.20 b	4.17 b	L ^{**}	3.81 b
80:20	Peat:PL	3.87 c	3.90 c	4.13 c	L ^{**}	8.48 a
	Peat:PBC	4.37 a	4.57 a	4.73 a	L ^{**}	4.50 b
	Peat:RBC	4.23 b	4.33 b	4.40 b	L ^{**}	6.52 a
75:25	Peat:PL	3.90 c	4.07 c	4.27 c	L ^{***}	8.28 a
	Peat:PBC	4.53 a	4.63 a	4.93 a	L ^{***}	4.67 b
	Peat:RBC	4.33 b	4.40 b	4.50 b	L [*]	9.04 a
70:30	Peat:PL	4.03 b	4.03 c	4.27 b	L ^{**}	9.26 a
	Peat:PBC	4.37 a	4.70 a	4.83 a	L ^{**}	5.99 b
	Peat:RBC	4.47 a	4.53 b	4.63 b	L ^{**}	8.06 a

^zpH measurements were conducted using the 1:1 dilution method; one part material (100 mL) was mixed vigorously with one part deionized (DI) water and allowed to equilibrate for 15 min (Lang, 1996).

^yInitial pH of the substrates was measured 24 h after substrates were mixed.

^xpH of substrates was measured ten days after initial pH measurement.

^wpH of substrates was measured 31 days after the ten day pH measurement.

^vpH buffering capacity was measured using an automatic titrator (Titralab[®] 856, Radiometer Analytical SAS, Villeurbanne Cedex, France) with samples diluted to 50 g with degassed DI water.

^uMeans separated within column, only between substrates of the same ratio, by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different ($n=3$).

^tLinear regression of each substrate pH over time, *, **, *** represent significant effects when $P \leq 0.05, 0.01$, and 0.001 , respectively.

Table 4. Cation and anion exchange capacities of substrate blends made with peat amended with either perlite (PL), pine wood biochar (PBC) or rice hull biochar (RBC).

Ratio (v/v)	Substrate	Chemical properties ^z	
		Cation exchange capacity ^y	Anion exchange capacity ^x
		mmol·100cc ⁻¹	
90:10	Peat:PL	11.98 a ^w	10.08 b
	Peat:PBC	14.53 a	16.09 a
	Peat:RBC	14.54 a	9.60 b
80:20	Peat:PL	10.94 b	22.38 a
	Peat:PBC	15.52 a	24.94 a
	Peat:RBC	6.61 c	12.30 a
70:30	Peat:PL	10.54 b	22.02 a
	Peat:PBC	13.83 b	20.68 ab
	Peat:RBC	17.89 a	13.71 b

^zChemical property data were collected from three samples from each substrate blend and represented as means.

^yCation exchange capacity measured using procedures described by Kloss et al. (2012) and Dumroese et al. (2011).

^xAnion exchange capacity measured using the procedure described by Lawrinenko (2014).

^wMeans separated within column, only between substrates of the same ratio, by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different (n=3).

Table 5. The effect on pH when amending peat moss with two types of biochar, pine wood (PBC) or rice hulls (RBC) at different rates with 0 kg·m³ of lime.

Rate	Substrate	pH ^z								
		Day ^y								
		0	1	3	7	14	21	28	42	
0 kg·m ³	100% PT ^x	3.77 e ^w	3.80 d	3.80 e	3.91 g	3.96 f	3.87 f	4.06 e	3.92 d	L*** ^v
	10% PBC ^u	4.23 cd	4.29 c	4.29 c	4.38 d	4.48 d	4.38 d	4.47 d	4.12 c	Q***
	20% PBC ^t	4.47 b	4.56 b	4.54 b	4.68 b	4.84 b	4.79 b	4.84 b	4.41 b	Q***
	30% PBC ^s	4.63 a	4.80 a	4.79 a	4.81 a	5.02 a	4.98 a	4.92 a	4.88 a	Q***
	10% RBC ^r	4.17 d	4.26 c	4.20 d	4.21 f	4.32 e	4.27 e	4.47 d	3.90 d	Q***
	20% RBC ^q	4.27 c	4.30 c	4.28 c	4.32 e	4.43 d	4.39 d	4.62 c	4.13 c	L***
	30% RBC ^p	4.50 b	4.59 b	4.58 b	4.56 c	4.69 c	4.61 c	4.78 b	4.33 b	Q***

^zpH measurements were conducted using the 1:1 dilution method; one part material (100 mL) was mixed vigorously with one part deionized (DI) water and allowed to equilibrate for 15 min (Lang, 1996).

^yDay 0 is initial pH of the substrates 24 h after being were mixed, the starting day for all the following measurements.

^x100% PT = 100% peat substrate.

^wMeans separated within column by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different (n=9).

^vLinear (L) or quadratic (Q) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$.

^u10% PBC = 90% peat with 10% pine wood biochar (v/v) substrate.

^t20% PBC = 80% peat with 20% pine wood biochar (v/v) substrate.

^s30% PBC = 70% peat with 30% pine wood biochar (v/v) substrate.

^r10% RBC = 90% peat with 10% rice hull biochar (v/v) substrate.

^q20% RBC = 80% peat with 20% rice hull biochar (v/v) substrate.

^p30% RBC = 70% peat with 30% rice hull biochar (v/v) substrate.

Table 6. The effect on pH when amending peat moss with two types of biochar, pine wood (PBC) or rice hulls (RBC) at different rates of lime.

Rate	Substrate	pH ^z								
		Day ^y								
		0	1	3	7	14	21	28	42	
1.19 kg·m ³	100% PT ^x	3.87 d ^w	4.47 d	4.50 d	4.52 e	4.62 e	4.56 d	4.73 d	4.42 c	Q**** ^v
	10% PBC ^u	4.23 bc	4.92 b	5.03 b	4.90 c	5.11 c	5.04 c	5.24 c	4.68 b	Q****
	20% PBC ^t	4.43 a	5.18 a	5.28 a	5.29 a	5.57 a	5.60 a	5.72 a	5.27 a	Q****
	10% RBC ^s	4.17 c	4.80 c	4.81 c	4.80 d	4.87 d	5.00 c	5.17 c	4.44 c	Q****
	20% RBC ^r	4.30 b	5.14 a	5.17 a	5.19 b	5.32 b	5.37 b	5.60 b	5.23 a	L****
2.38 kg·m ³	100% PT	3.97 d	5.20 d	5.06 bc	4.99 d	5.16 d	5.19 d	5.71 d	5.49 c	Q****
	10% PBC	4.33 b	5.62 c	5.59 abc	5.59 c	5.88 b	5.90 bc	6.10 c	5.77 b	L****
	20% PBC	4.50 a	5.59 c	5.63 ab	5.68 b	5.92 b	6.01 b	6.40 a	5.90 a	L****
	10% RBC	4.23 c	5.74 b	4.92 c	5.51 c	5.74 c	5.84 c	6.22 b	5.28 d	L****
	20% RBC	4.33 b	5.89 a	5.80 a	5.77 a	6.12 a	6.16 a	6.47 a	5.89 a	L****
4.75 kg·m ³	100% PT	3.93 d	6.08 c	6.03 d	5.99 d	6.26 c	6.49 d	6.90 e	6.02 c	L****
	10% PBC	4.30 b	6.26 b	6.33 c	6.32 c	6.69 b	6.78 c	7.00 d	6.64 b	L****
	20% PBC	4.53 a	6.32 ab	6.54 a	6.52 a	7.07 a	7.39 a	7.53 a	7.20 a	L****
	10% RBC	4.13 c	6.33 a	6.44 b	6.40 b	6.79 b	7.03 b	7.17 c	6.70 b	L****
	20% RBC	4.30 b	6.39 a	6.39 bc	6.42 b	6.76 b	7.06 b	7.30 b	6.69 b	L****
7.13 kg·m ³	100% PT	3.97 c	6.37 d	6.53 d	6.37 c	6.98 d	7.33 d	7.38 d	6.91 e	L****
	10% PBC	4.27 b	6.56 c	6.72 b	6.59 b	7.34 b	7.76 b	7.96 b	7.91 a	Q****
	20% PBC	4.50 a	6.76 a	6.81 a	6.70 a	7.51 a	7.93 a	8.06 a	7.71 b	L****
	10% RBC	4.37 b	6.60 c	6.67 c	6.57 b	7.06 cd	7.32 d	7.50 c	7.02 d	L****
	20% RBC	4.30 b	6.67 b	6.71 bc	6.61 b	7.13 c	7.44 c	7.50 c	7.18 c	L****

^zpH measurements were conducted using the 1:1 dilution method; one part material (100 mL) was mixed vigorously with one part deionized (DI) water and allowed to equilibrate for 15 min (Lang, 1996).

^yDay 0 is initial pH of the substrates 24 h after being mixed, the starting day for all the following measurements.

^x100% PT = 100% peat substrate.

^wMeans separated within column by Least Significant Difference (LSD), $P \leq 0.05$. Means followed by the same letter are not significantly different ($n=9$).

^vLinear (L) or quadratic (Q) regression of each substrate pH over time, **** represent significant effects when $P \leq 0.001$.

^u10% PBC = 90% peat with 10% pine wood biochar (v/v) substrate.

^t20% PBC = 80% peat with 20% pine wood biochar (v/v) substrate.

^s10% RBC = 90% peat with 10% rice hull biochar (v/v) substrate.

^r20% RBC = 80% peat with 20% rice hull biochar (v/v) substrate.

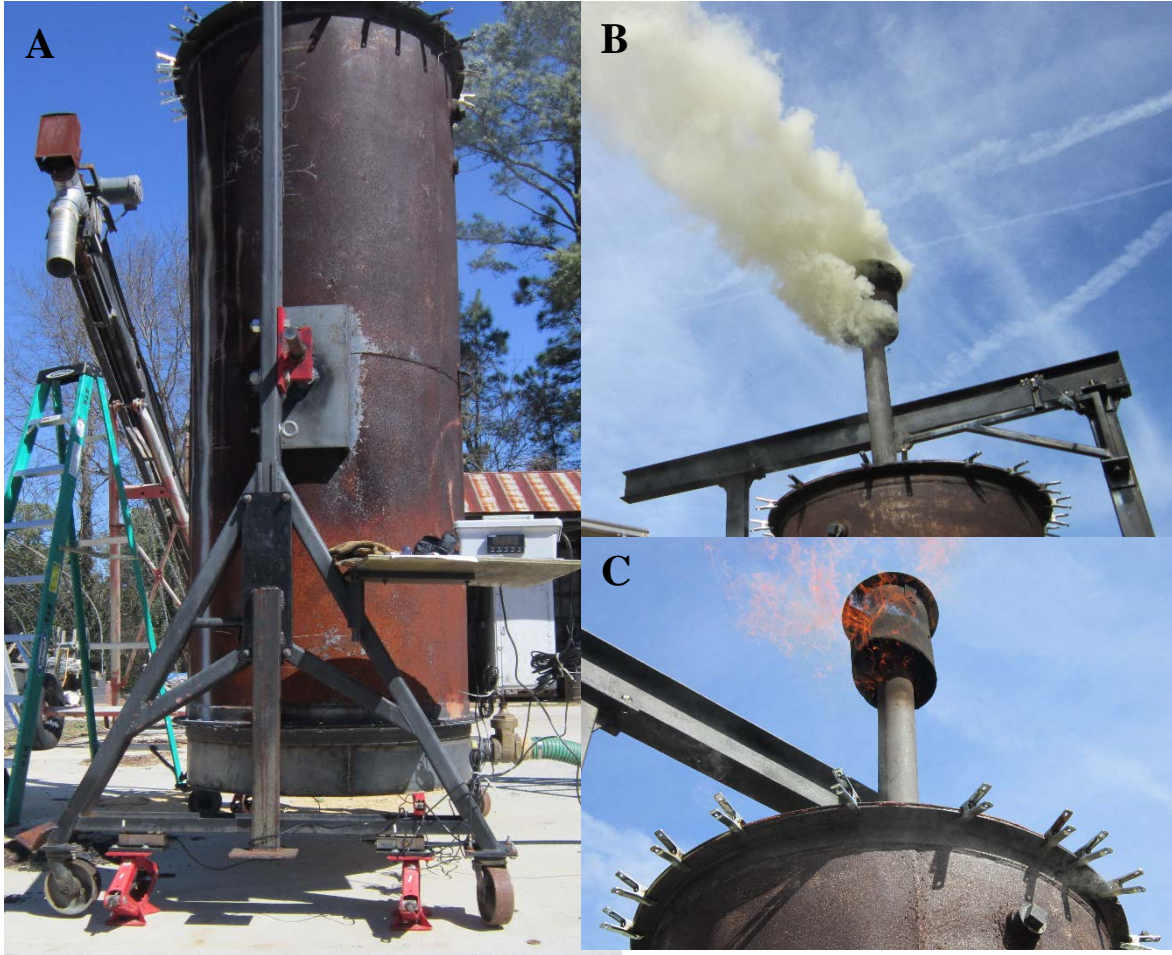


Figure 1. (A) The top-lit updraft (TLUD) gasifier with frame. After filling the reactor with feedstock and igniting, the resulting gas is visible (B) and can be lit to reduce amount of smoke in the air (C).



Figure 2. (A) On the left, biochar produced from pine wood (loblolly; *Pinus taeda*), that was then ground to <2-mm size (right; PBC_p). (B) On the left, biochar produced from rice hulls, that was then ground to <2-mm size (right; RBC_p).

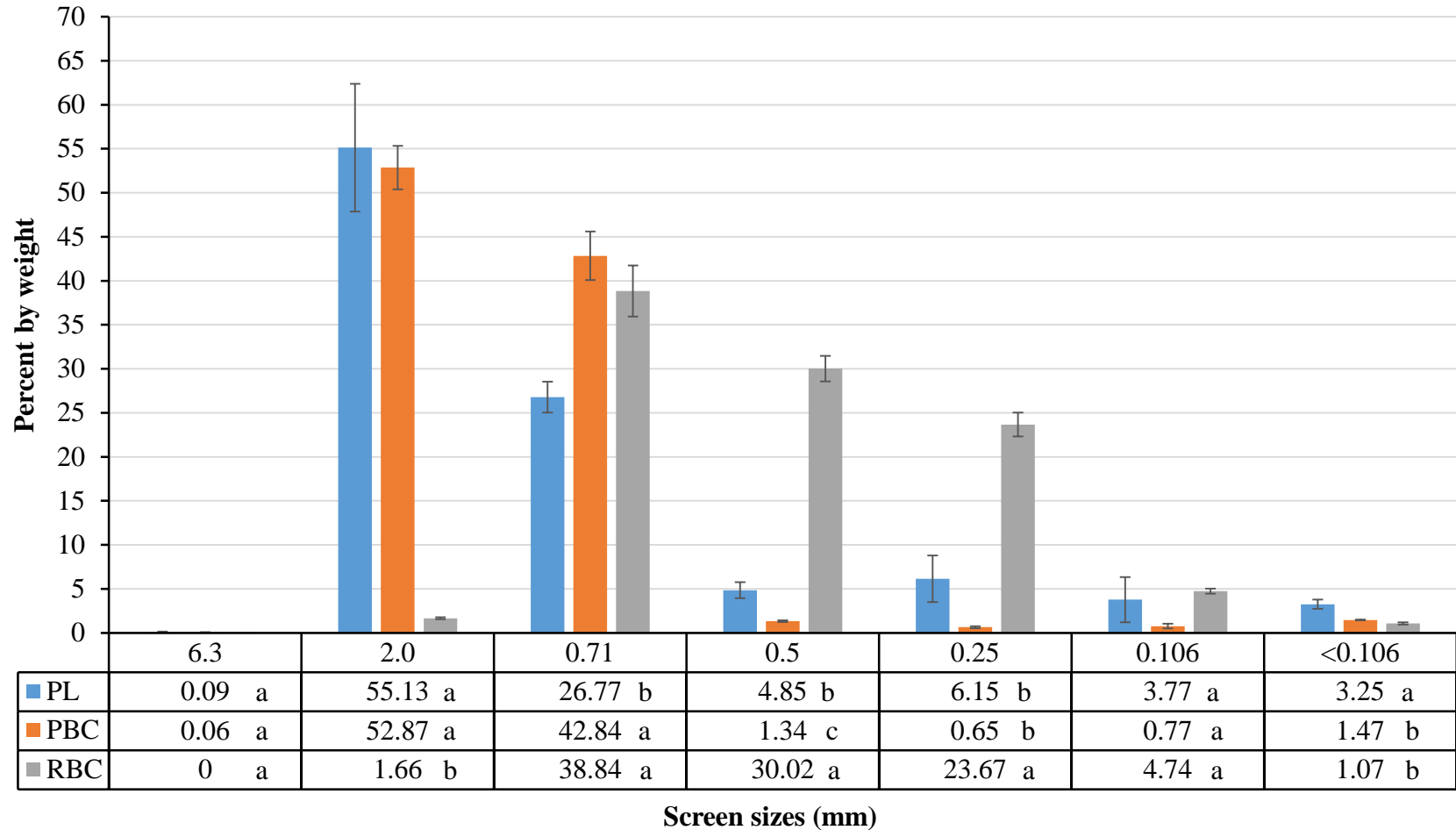


Figure 3. Particle size distribution, using six sieves with a pan, of perlite (PL), pine wood biochar (PBC) and rice hull biochar (RBC). Error bars represent standard error and means separated by each sieve size by Least Significant Difference (LSD) at $P \leq 0.05$; means followed by the same letter are not significantly different (n=3).



Figure 4. Comparison of particle size and shape of rice hull biochar (RBC), perlite (PL) and pine wood biochar (PBC).

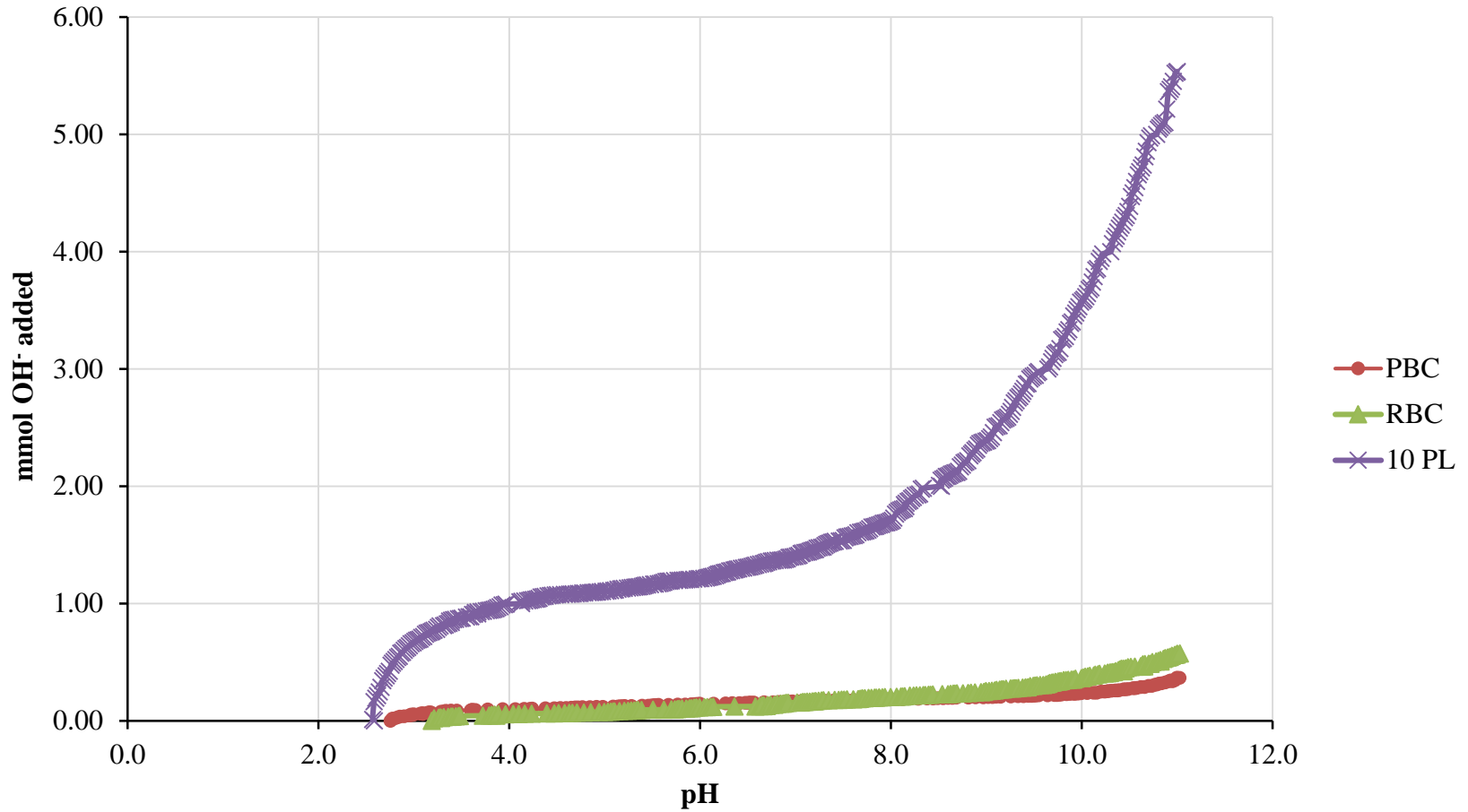


Figure 5. Titration curves (of one sample) for peat amended with 10% perlite (PL), 100% pine wood biochar (PBC) and 100% rice hull biochar (RBC). Determining amount of biochar needed at a particular pH: mmol H^+ neutralized kg^{-1} 10% PL divided by mmol H^+ kg^{-1} biochar = $\text{kg biochar kg}^{-1}$ peat.

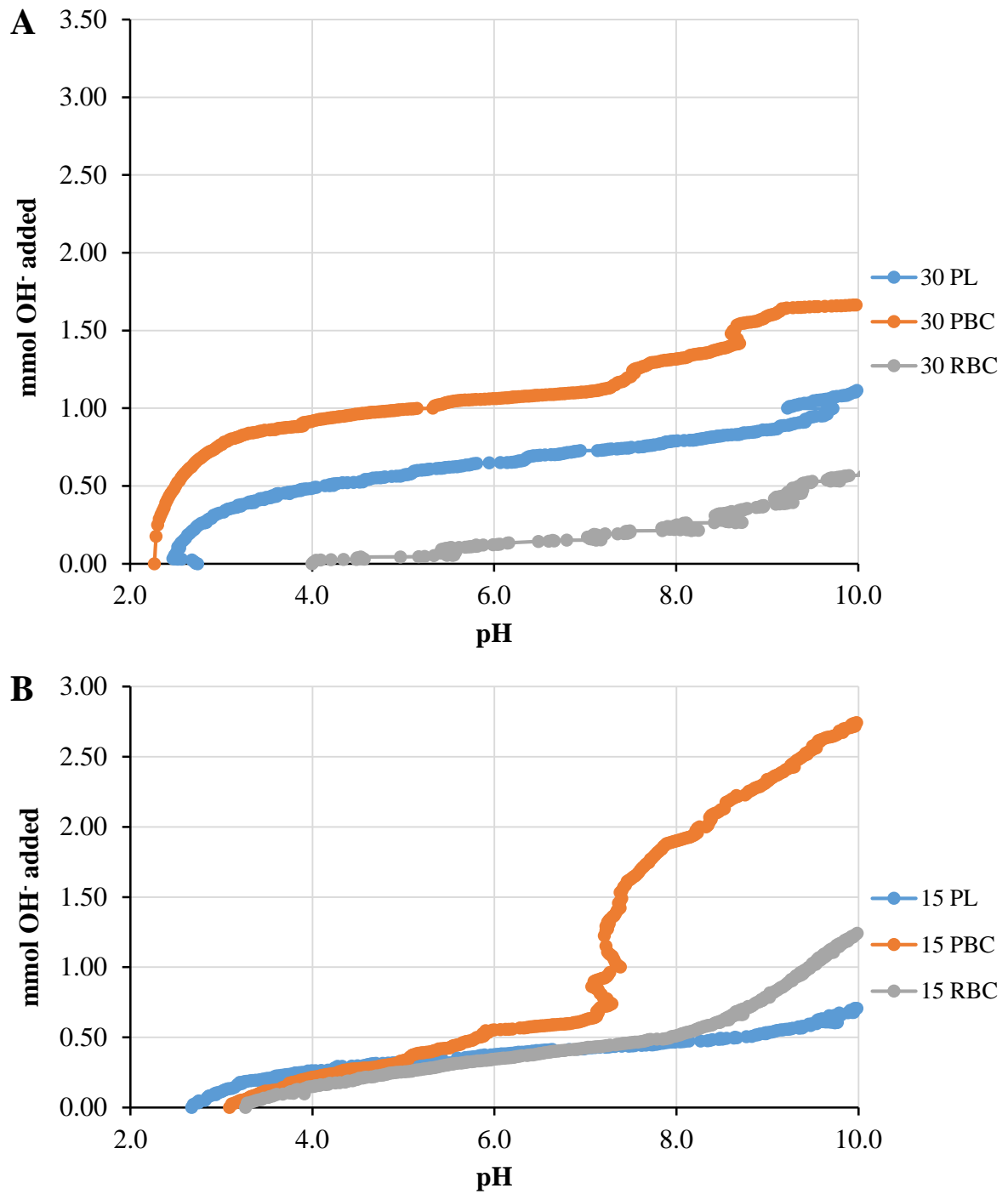


Figure 6. Titration curves (for one sample) for peat amended with either perlite (PL), pine wood biochar (PBC) or rice hull biochar (RBC) at 30% (A) or 15% (B).

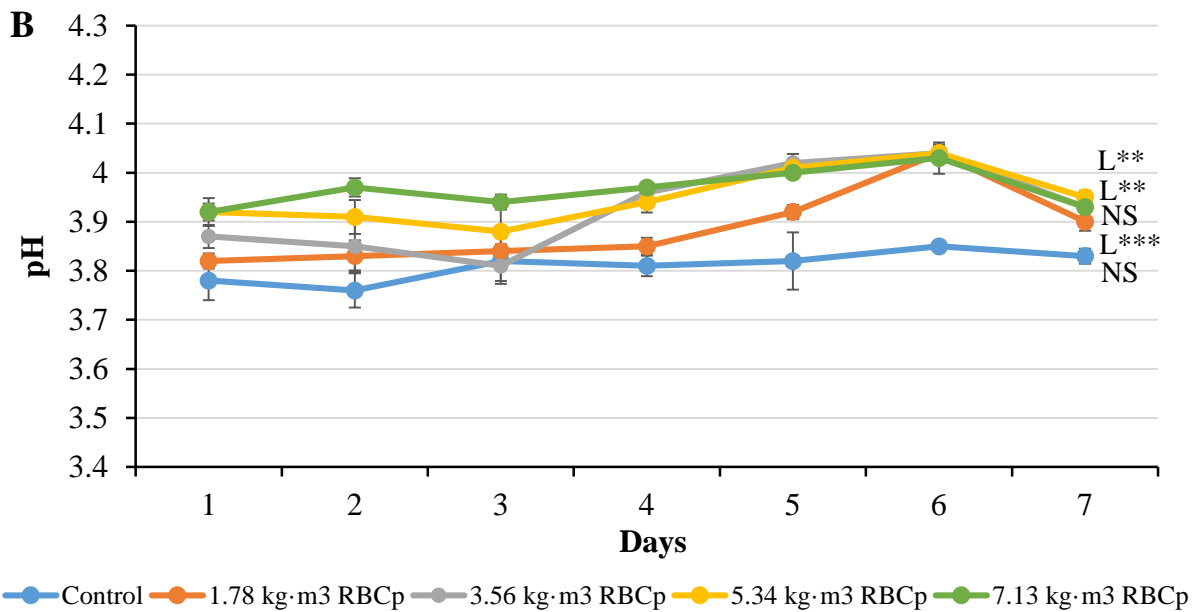
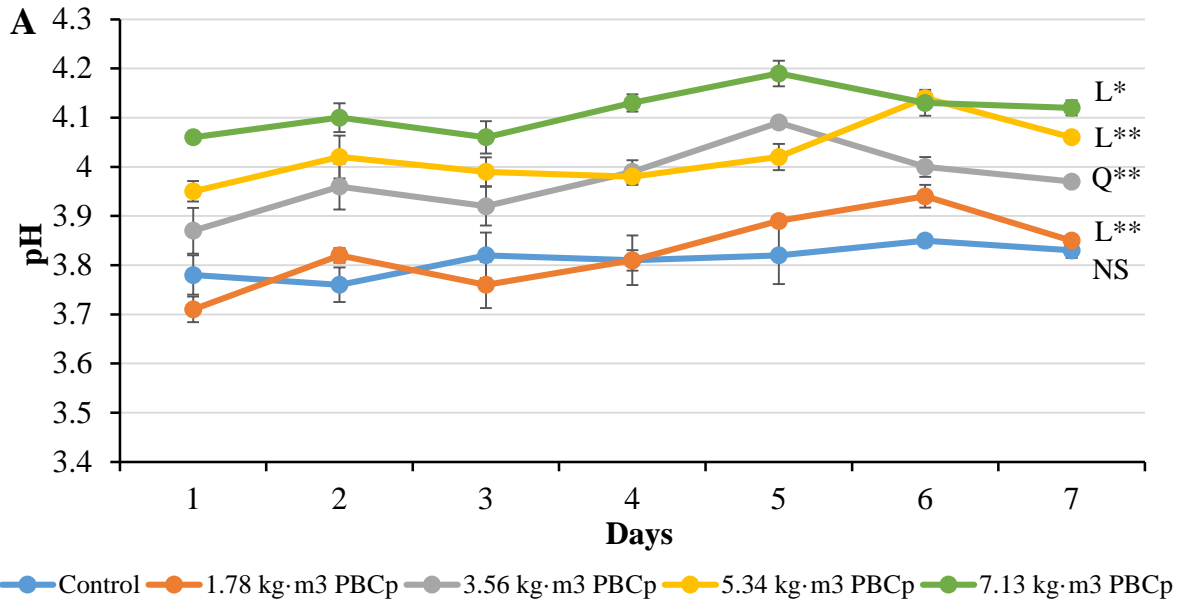


Figure 7. Comparison of pulverized biochars, (A) pine wood (PBC_p) or (B) rice hull (RBC_p), when added to peat moss at four different rates (1.78, 3.56, 5.34 or 7.13 kg·m³) and compared to 100% peat (control) with no biochar addition to show effects on pH over time. Error bars represent standard error; non-significant (NS), linear (L), or quadratic (Q) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$.

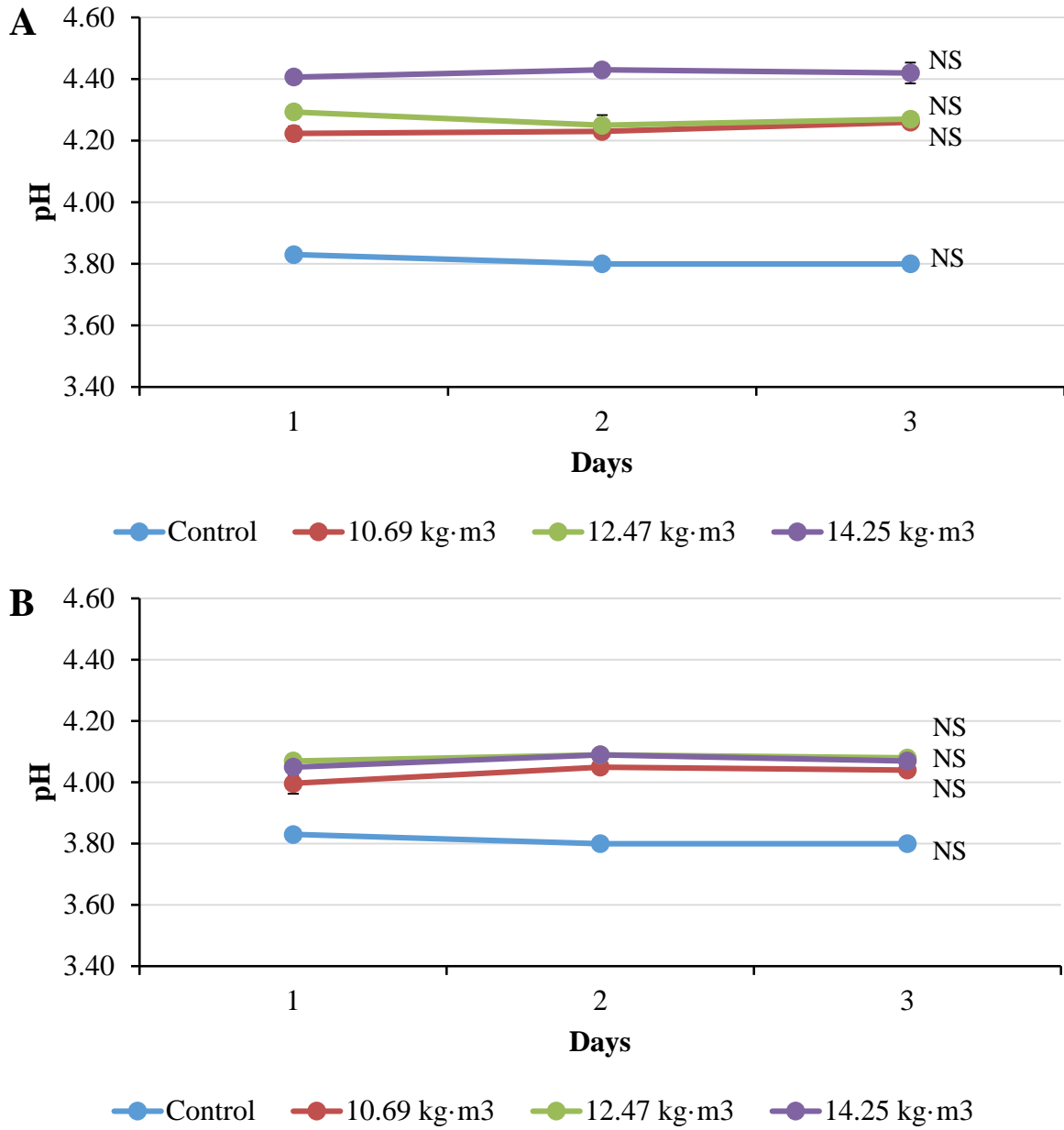


Figure 8. Comparison of pulverized biochars, (A) pine wood (PBC_p) or (B) rice hull (RBC_p), when added to peat moss at three different rates (10.69, 12.47, and 14.25 kg·m³) and compared to 100% peat (control) with no biochar addition to show effects on pH over time. Error bars represent standard error; there was non-significant (NS) effects of each substrate pH over time.

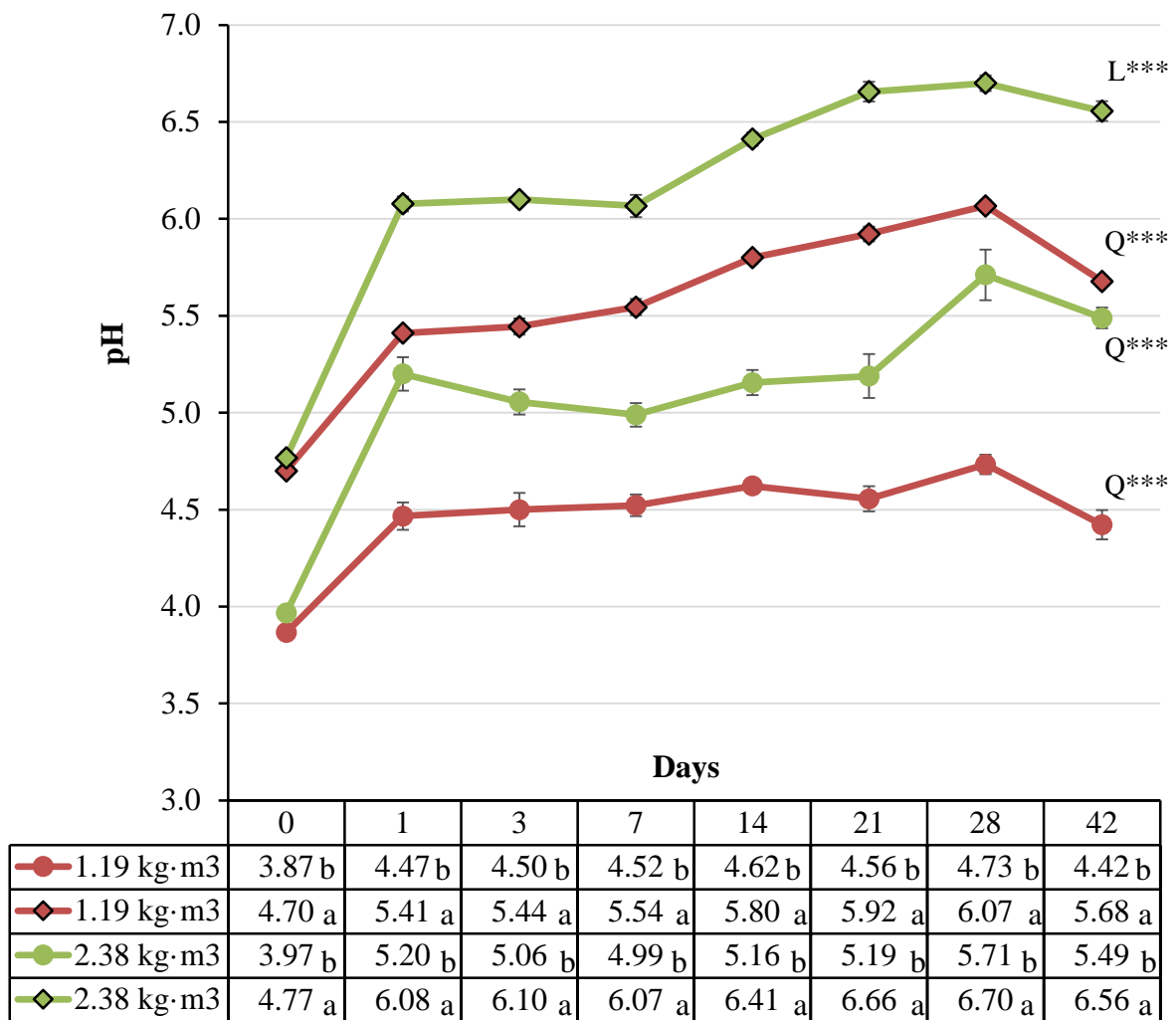
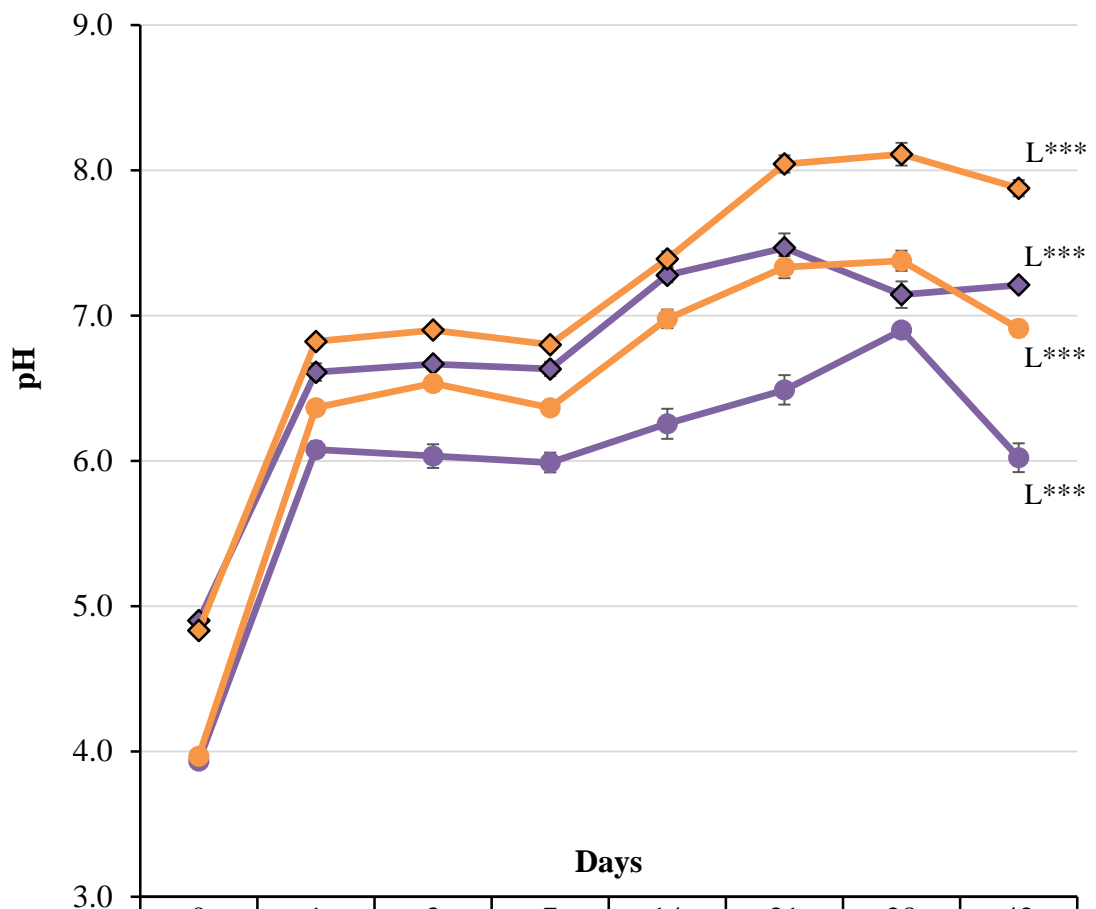
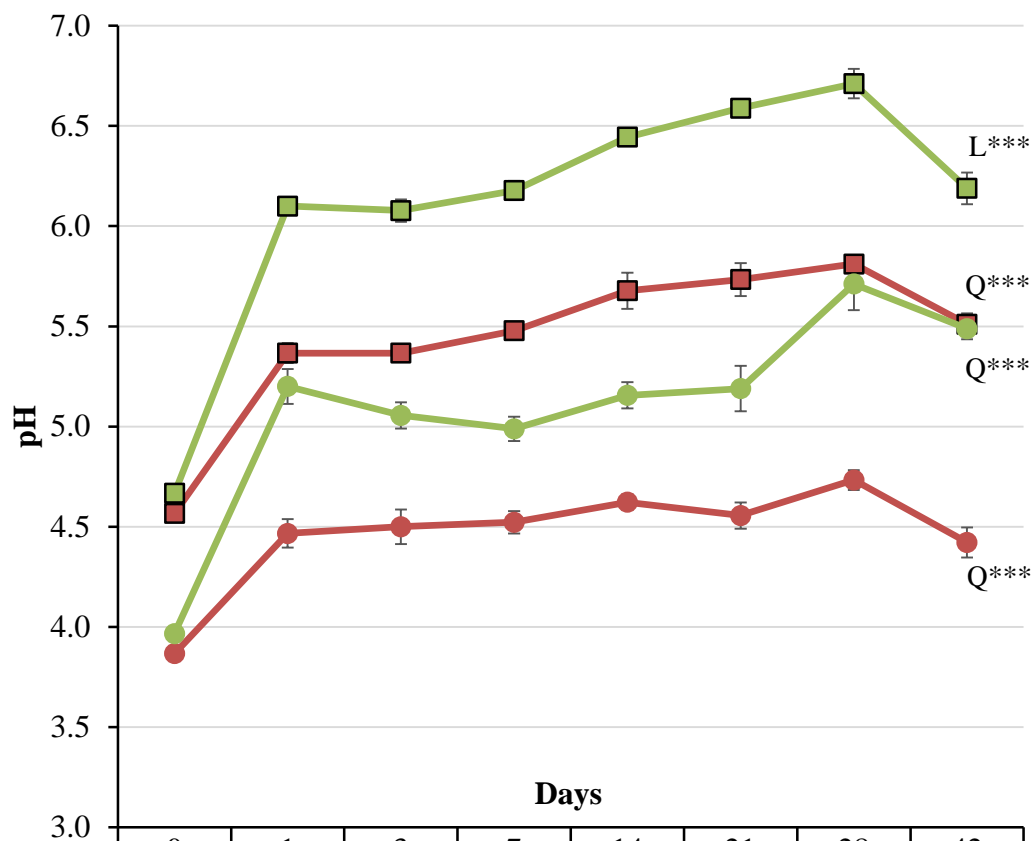


Figure 9. Comparison of 100% peat (●) to 30% pine wood biochar (PBC) with 70% peat (◆) with lime (200 mesh) at two different rates (1.19 or 2.38 kg·m³) to show effects on pH over time. Error bars represent standard error; linear (L) or quadratic (Q) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$. Table shows means separated within column, only between substrates of the same lime rate (represented by color), by Dunnett control test, $P \leq 0.05$. Means followed by the same letter are not significantly different.



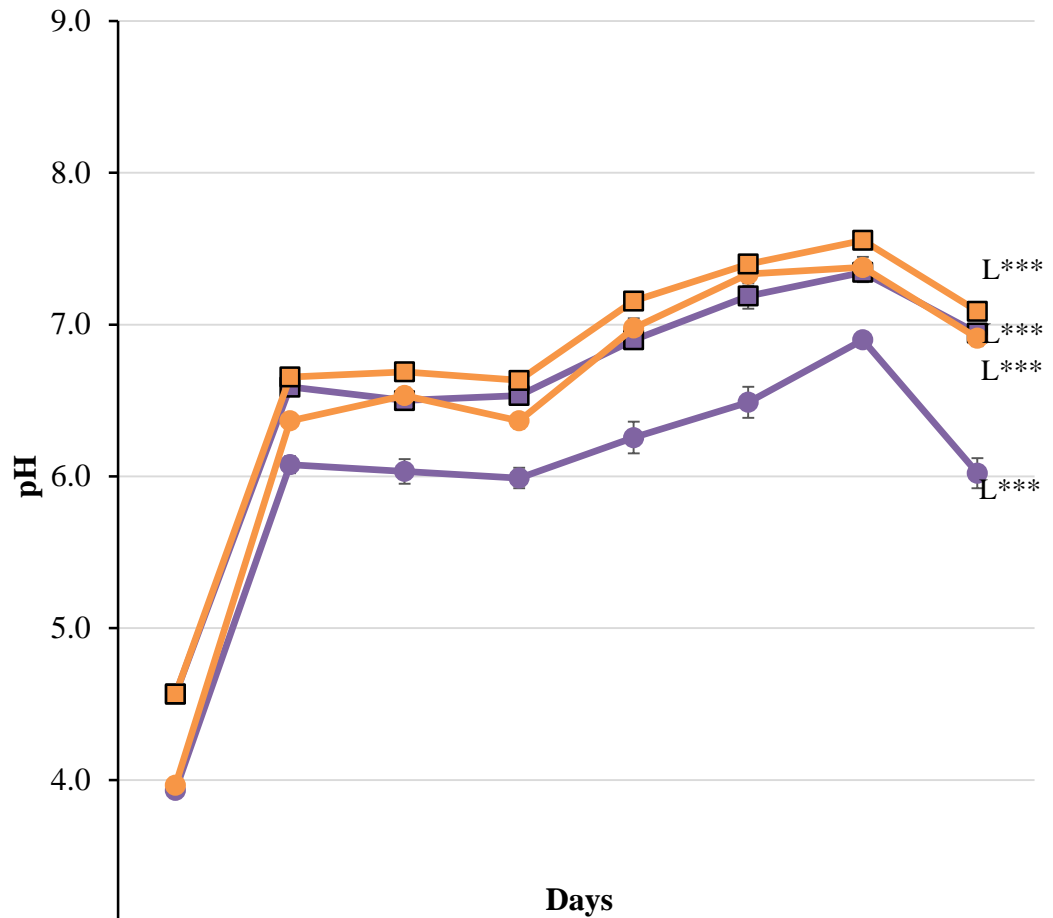
	0	1	3	7	14	21	28	42
● 4.75 kg·m ³	3.93 b	6.08 b	6.03 b	5.99 b	6.26 b	6.49 b	6.90 b	6.02 b
◆ 4.75 kg·m ³	4.90 a	6.61 a	6.67 a	6.63 a	7.28 a	7.47 a	7.14 a	7.21 a
● 7.13 kg·m ³	3.97 b	6.37 b	6.53 b	6.37 b	6.98 b	7.33 b	7.38 b	6.91 b
◆ 7.13 kg·m ³	4.83 a	6.82 a	6.90 a	6.80 a	7.39 a	8.04 a	8.11 a	7.88 a

Figure 10. Comparison of 100% peat (●) to 30% pine wood biochar (PBC) with 70% peat (◆) with lime (200 mesh) at two different rates (4.75 or 7.13 kg·m³) to show effects on pH over time. Error bars represent standard error; linear (L) or quadratic (Q) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$. Table shows means separated within column, only between substrates of the same lime rate (represented by color), by Dunnett control test, $P \leq 0.05$ (n=9). Means followed by the same letter are not significantly different.



	0	1	3	7	14	21	28	42
● 1.19 kg·m ³	3.87b	4.47b	4.50 b	4.52 b	4.62 b	4.56 b	4.73 b	4.42b
■ 1.19 kg·m ³	4.57a	5.37 a	5.37 a	5.48 a	5.68 a	5.73 a	5.81 a	5.51 a
● 2.38 kg·m ³	3.97b	5.20b	5.06 b	4.99 b	5.16 b	5.19 b	5.71 b	5.49b
■ 2.38 kg·m ³	4.67a	6.10 a	6.08 a	6.18 a	6.44 a	6.59 a	6.71 a	6.19 a

Figure 11. Comparison of 100% peat (●) to 30% rice hull biochar (RBC) with 70% peat (□) with lime (200 mesh) at two different rates (1.19 or 2.38 kg·m³) to show the effects on pH over time. Error bars represent standard error; linear (L) or quadratic (Q) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$. Table shows means separated within column, only between substrates of the same lime rate (represented by color), by Dunnett's control test, $P \leq 0.05$. Means followed by the same letter are not significantly different.



	0	1	3	7	14	21	28	42
● 4.75 kg·m ³	3.93 b	6.08 b	6.03 b	5.99 b	6.26 b	6.49 b	6.90 b	6.02 b
■ 4.75 kg·m ³	4.57 a	6.59 a	6.50 a	6.53 a	6.90 a	7.19 a	7.34 a	6.94 a
● 7.13 kg·m ³	3.97 b	6.37 b	6.53 b	6.37 b	6.98 b	7.33 a	7.38 b	6.91 b
■ 7.13 kg·m ³	4.57 a	6.66 a	6.69 a	6.63 a	7.16 a	7.40 a	7.56 a	7.09 a

Figure 12. Comparison of 100% peat (●) to 30% rice hull biochar (RBC) with 70% peat (□) with lime (200 mesh) at two different rates (4.75 or 7.13 kg·m³) to show the effects on pH over time. Error bars represent standard error; linear (L) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$. Table shows means separated within column, only between substrates of the same lime rate (represented by color), by Dunnett's control test, $P \leq 0.05$ (n=9). Means followed by the same letter are not significantly different.

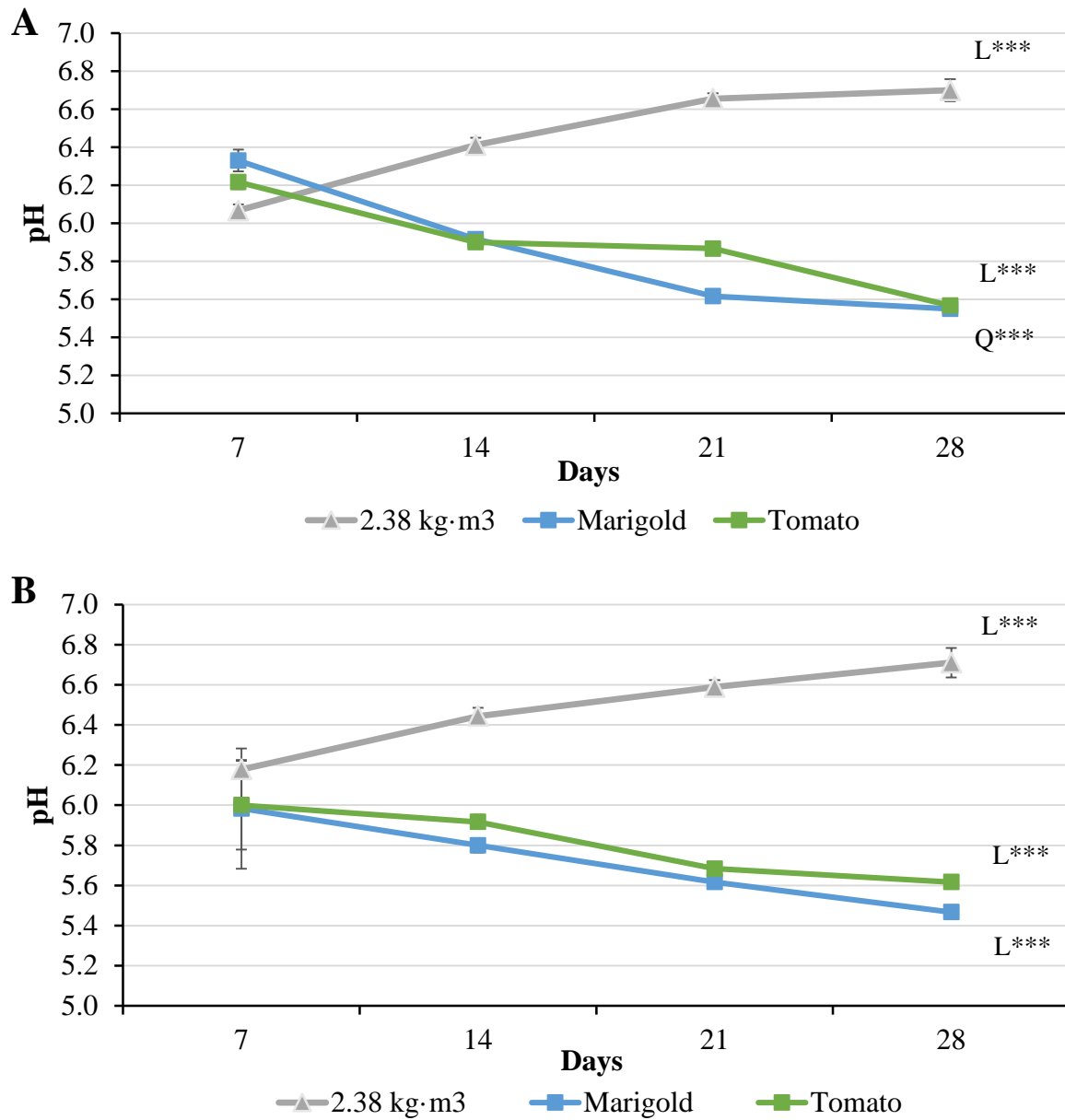


Figure 13. (A) Comparison of 30% pine wood biochar (PBC) with 70% peat limed at 2.38 kg·m³ with no plants (\blacktriangle) and with marigold (\square) or tomato (\square). (B) Comparison of 30% rice hull biochar (RBC) with 70% peat limed at 2.38 kg·m³ with no plants (\blacktriangle) and with marigold (\square) or tomato (\square). Error bars represent standard error; linear (L) regression of each substrate pH over time, *** represent significant effects when $P \leq 0.001$.

APPENDICES

Appendix A: Particle size distribution of biochar materials across batches (% weight) with standard deviation (s.d.), and means from each individual batch.^z

Pine wood biochar (PBC)														
Sample ^y	Screen sizes (mm)													
	6.3	<i>s.d.</i>	2.0	<i>s.d.</i>	0.71	<i>s.d.</i>	0.5	<i>s.d.</i>	0.25	<i>s.d.</i>	0.106	<i>s.d.</i>	<0.106	<i>s.d.</i>
Top	0.10	0.01	46.93	5.61	47.38	4.14	2.26	0.65	1.06	0.30	0.51	0.16	1.76	0.52
Middle	0.21	0.19	53.68	3.52	41.68	3.81	1.52	0.25	0.72	0.17	0.66	0.58	1.53	0.11
Bottom	0.55	0.56	50.30	1.13	44.95	1.95	1.38	0.22	0.89	0.65	0.34	0.22	1.60	0.32
Consistency range	0.29 ±0.25 ^x		50.30 ±3.42		44.67 ±3.30		1.61 ±0.37		0.89 ±0.37		0.50 ±0.30		1.63 ±0.32	
Batch means ^w														
PBC #1	0.06		52.87		42.84		1.34		0.65		0.77		1.47	
PBC #2	0.54		48.38		45.80		1.93		0.88		0.48		1.99	
PBC #3	0.26		49.66		45.37		1.89		1.13		0.26		1.43	
Rice hull biochar (RBC)														
Sample	6.3	<i>s.d.</i>	2.0	<i>s.d.</i>	0.71	<i>s.d.</i>	0.5	<i>s.d.</i>	0.25	<i>s.d.</i>	0.106	<i>s.d.</i>	<0.106	<i>s.d.</i>
Top	0.00	0.00	2.16	1.74	42.90	3.50	28.57	1.92	21.15	2.35	4.28	0.81	0.94	0.16
Middle	0.00	0.00	1.80	0.19	37.95	1.30	30.67	0.73	23.66	0.89	4.87	0.33	1.06	0.24
Bottom	0.00	0.00	1.79	0.29	36.28	2.60	31.25	1.40	24.55	1.44	5.08	0.25	1.04	0.13
Consistency range	0.00 ±0.00		1.92 ±0.74		39.04 ±2.47		30.16 ±1.35		23.12 ±1.56		4.74 ±0.46		1.01 ±0.18	
Batch means														
RBC #1	0.00		1.66		38.84		30.02		23.67		4.74		1.07	
RBC #2	0.00		1.54		39.11		30.46		23.03		4.83		1.03	
RBC #3	0.00		2.55		39.17		30.02		22.67		4.66		0.94	

^zParticle size distribution data were collected from three samples from each batch run and represented as a mean percent by weight of the samples. Analysis performed using Ro-tap Shaker (Model B, W.S. Tyler, Mentor, Ohio) fitted with six sieves; 6.3 mm, 2mm, 0.71 mm, 0.5 mm, 0.25 mm, and 0.106 mm.

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within consistency range (n=9) for that batch to be considered consistent.

Appendix B: Total elemental composition of biochar materials across batches with standard deviation (s.d.), and means from each individual batch. Elements include boron (B), calcium (Ca), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), and molybdenum (Mo).^z

Pine wood biochar (PBC)														
Sample ^y	(mg·L ⁻¹)													
	B	s.d.	Ca	s.d.	Cu	s.d.	Fe	s.d.	Mg	s.d.	Mn	s.d.	Mo	s.d.
Top	8.20	1.21	5339.1	363.2	14.10	9.05	1883.0	2479.51	1297.87	105.04	330.33	31.09	0.30	0.00
Middle	7.63	1.14	5246.9	576.7	9.10	4.42	3034.0	4054.66	1248.00	56.90	321.67	17.04	0.32	0.03
Bottom	7.60	1.74	5052.7	381.0	9.90	4.19	695.67	250.42	1202.38	89.17	302.00	22.52	0.30	0.00
Consistency range	7.81 ±1.36 ^x		5212.9 ±440.3		11.03 ±5.89		1870.89 ±2261.53		1249.42 ±83.70		318.00 ±23.55		0.31 ±0.01	
Batch means ^w														
PBC #1	7.7		5115.5		15.2		3091.0		1245.5		317.0		0.30	
PBC #2	6.6		4839.2		7.6		2047.0		1169.4		296.7		0.32	
PBC #3	9.1		5684.0		10.3		475.0		1333.3		340.3		0.30	
Rice hull biochar (RBC)														
Sample	(mg·L ⁻¹)													
	B	s.d.	Ca	s.d.	Cu	s.d.	Fe	s.d.	Mg	s.d.	Mn	s.d.	Mo	s.d.
Top	4.66	0.22	2078.4	48.19	4.64	0.49	265.63	74.06	970.60	45.84	495.79	21.25	0.45	0.08
Middle	4.66	0.64	2060.8	57.29	7.10	0.39	269.41	108.03	995.03	60.22	506.66	26.18	0.51	0.12
Bottom	4.69	0.53	2116.9	63.16	6.45	2.00	249.88	56.91	1010.90	28.34	519.85	10.60	0.56	0.13
Consistency range	4.67 ±0.62		2085.4 ±56.21		6.06 ±0.96		261.64 ±79.67		992.18 ±44.80		507.43 ±19.34		0.51 ±0.38	
Batch means														
RBC #1	5.0		2129.1		7.03		268.7		968.7		501.0		0.53	
RBC #2	4.4		2089.5		5.80		315.3		1020.3		518.7		0.42	
RBC #3	4.7		2037.5		5.37		200.7		978.6		502.3		0.57	

^zTotal elemental composition measured by dry ash procedure and wet digestion method by inductively coupled plasma optical emission spectroscopy (ICP-OES).

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix C: Total elemental composition of biochar materials across batches with standard deviation (s.d.), and means from each individual batch. Elements include phosphorus (P), potassium (K), silica (Si), sulfur (S) and zinc (Zn).^z

Pine wood biochar (PBC)										
Sample ^y	(mg·L ⁻¹)									
	P	s.d.	K	s.d.	Si	s.d.	S	s.d.	Zn	s.d.
Top	489.37	58.27	3018.73	31.76	1103.3	452.8	92.07	20.44	28.77	3.63
Middle	468.38	36.41	2808.47	296.05	736.7	518.3	95.90	19.22	29.23	3.01
Bottom	441.80	44.53	2785.30	302.67	580.0	364.3	83.73	29.67	26.67	3.25
Consistency range	466.52 ±46.40 ^x		2870.83 ±210.16		806.7 ±445.1		90.57 ±23.11		28.22 ±3.30	
Batch means ^w										
PBC #1	461.33		2854.2		1268.1		92.3		25.7	
PBC #2	424.70		2699.8		546.1		67.6		27.1	
PBC #3	513.52		3058.5		622.9		111.8		31.9	
Rice hull biochar (RBC)										
Sample	(mg·L ⁻¹)									
	P	s.d.	K	s.d.	Si	s.d.	S	s.d.	Zn	s.d.
Top	1756.5	43.31	4734.57	57.96	73878.2	10190.2	124.50	41.33	25.11	0.18
Middle	1773.1	29.59	4918.50	232.00	78868.6	9036.6	138.85	52.17	25.76	1.42
Bottom	1764.3	35.30	5061.57	81.62	85732.5	4112.3	128.46	40.12	26.87	0.61
Consistency range	1764.63 ±36.07		4904.88 ±123.86		79493.1 ±7779.7		130.60 ±44.54		25.91 ±0.74	
Batch means										
RBC #1	1769.6		4950.3		75628.0		129.2		26.0	
RBC #2	1790.6		4962.7		86665.0		96.7		26.5	
RBC #3	1733.7		4801.6		76186.0		166.0		25.2	

^zTotal elemental composition measured by dry ash procedure and wet digestion method by inductively coupled plasma optical emission spectroscopy (ICP-OES).

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix D: Total carbon (C), hydrogen (H) and nitrogen (N) for biochar materials across batches with standard deviation (s.d.), and means from each individual batch.^z

Pine wood biochar (PBC)						
Sample ^y	Total C (%)	s.d.	Total H (%)	s.d.	Total N (%)	s.d.
Top	83.07	1.35	1.57	0.07	0.25	0.04
Middle	82.19	3.98	1.63	0.24	0.27	0.08
Bottom	82.68	3.66	1.58	0.32	0.25	0.08
Consistency range	82.65 ±3.00 ^x		1.59 ±0.21		0.26 ±0.07	
Batch means ^w						
PBC #1	79.96		1.69		0.21	
PBC #2	82.12		1.70		0.22	
PBC #3	85.86		1.39		0.33	
Rice hull biochar (RBC)						
Sample	Total C (%)	s.d.	Total H (%)	s.d.	Total N (%)	s.d.
Top	39.78	1.36	0.94	0.17	0.41	0.03
Middle	40.72	1.07	0.94	0.12	0.41	0.03
Bottom	41.23	0.34	0.91	0.11	0.41	0.02
Consistency range	40.58 ±0.92		0.93 ±0.13		0.41 ±0.03	
Batch means						
RBC #1	40.79		0.83		0.43	
RBC #2	39.87		0.91		0.39	
RBC #3	41.08		1.05		0.41	

^zTotal elemental composition measured by dry ash procedure and wet digestion method by inductively coupled plasma optical emission spectroscopy (ICP-OES).

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix E: Available nutrients of biochar materials across batches with standard deviation (s.d.), and means from each individual batch. Nutrients include boron (B), calcium (Ca), chloride (Cl), copper (Cu), iron (Fe), magnesium (Mg), and manganese (Mn).

Pine wood biochar (PBC)														
Water extraction (mg·L ⁻¹) ^z														
Sample ^y	B	s.d.	Ca	s.d.	Cl	s.d.	Cu	s.d.	Fe	s.d.	Mg	s.d.	Mn	s.d.
Top	0.01	0.00	5.94	1.24	2.21	0.74	0.007	0.006	0.02	0.00	12.07	0.65	0.06	0.01
Middle	0.01	0.01	5.70	1.01	2.47	1.15	0.010	0.000	0.03	0.02	11.79	3.76	0.07	0.03
Bottom	0.01	0.01	5.17	1.18	2.73	1.58	0.007	0.005	0.03	0.02	11.42	4.35	0.06	0.02
Consistency range	0.01 ±0.007 ^x		5.60 ±1.14		2.47 ±1.16		0.008 ±0.004		0.03 ±0.01		11.76 ±2.92		0.06 ±0.02	
Batch means ^w														
PBC #1	0.01		5.0		3.8		0.01		0.02		10.7		0.06	
PBC #2	0.02		5.0		1.8		0.01		0.04		9.5		0.09	
PBC #3	0.01		6.9		1.8		0.01		0.02		15.0		0.06	
Rice hull biochar (RBC)														
Water extraction (mg·L ⁻¹)														
Sample	B	s.d.	Ca	s.d.	Cl	s.d.	Cu	s.d.	Fe	s.d.	Mg	s.d.	Mn	s.d.
Top	0.004	0.005	1.22	0.03	6.15	1.10	0.007	0.005	0.03	0.006	3.92	0.65	0.46	0.10
Middle	0.004	0.005	1.22	0.20	6.10	1.13	0.007	0.005	0.02	0.006	4.43	0.46	0.46	0.08
Bottom	0.001	0.000	1.35	0.19	6.27	1.02	0.007	0.005	0.02	0.006	4.02	0.63	0.51	0.04
Consistency range	0.003 ±0.003		1.26 ±0.14		6.17 ±1.08		0.007 ±0.005		0.02 ±0.006		4.12 ±1.74		0.48 ±0.07	
Batch means														
RBC #1	0.01		1.2		6.3		0.01		0.03		4.2		0.5	
RBC #2	0.001		1.4		7.2		0.01		0.03		4.6		0.5	
RBC #3	0.001		1.2		5.1		0.01		0.02		3.6		0.5	

^zDetermined by saturated media extract and vacuum filtration with an auto-flow spectrophotometric analyzer.

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix F: Available nutrients of biochar materials across batches with standard deviation (s.d.), and means from each individual batch. Nutrients include nitrogen (N), phosphorus (P), potassium (K), sodium (Na), sulfur (S) and zinc (Zn).

Pine wood biochar (PBC)												
Water extraction (mg·L ⁻¹) ^z												
Sample ^y	N	s.d.	P	s.d.	K	s.d.	Na	s.d.	S	s.d.	Zn	s.d.
Top	0.78	0.10	1.45	0.14	373.33	46.37	7.81	1.52	2.04	0.54	0.001	0.00
Middle	0.68	0.06	1.58	0.16	363.67	64.59	7.85	0.67	1.98	0.12	0.001	0.00
Bottom	0.86	0.08	1.52	0.07	368.00	72.69	7.87	0.82	1.88	0.27	0.001	0.00
Consistency range	0.77 ±0.08 ^x		1.52 ±0.12		368.33 ±61.22		7.84 ±1.00		1.97 ±0.31		0.001 ±0.00	
Batch means ^w												
PBC #1	0.8		1.5		344.3		8.5		1.8		0.001	
PBC #2	0.8		1.5		325.3		8.3		1.8		0.001	
PBC #3	0.8		1.5		435.3		6.8		2.3		0.001	
Rice hull biochar (RBC)												
Water extraction (mg·L ⁻¹)												
Sample	N	s.d.	P	s.d.	K	s.d.	Na	s.d.	S	s.d.	Zn	s.d.
Top	0.53	0.11	32.77	6.25	351.33	58.59	20.97	3.72	4.66	1.44	0.004	0.005
Middle	0.58	0.21	33.27	2.75	346.67	72.23	19.73	4.92	4.78	2.83	0.004	0.005
Bottom	0.38	0.00	33.13	4.76	373.33	63.37	19.30	1.80	4.21	0.77	0.004	0.005
Consistency range	0.50 ±0.11		33.06 ±4.59		357.11 ±64.73		20.00 ±3.48		4.55 ±1.68		0.004 ±0.005	
Batch means												
RBC #1	0.5		33.7		356.0		22.9		5.9		0.001	
RBC #2	0.4		37.3		420.7		20.7		4.6		0.001	
RBC #3	0.6		28.2		294.7		16.4		3.2		0.010	

^zDetermined by saturated media extract and vacuum filtration with an auto-flow spectrophotometric analyzer.

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix G: Acid extraction of nutrients for biochar materials across batches with standard deviation (s.d.), and means from each individual batch. Nutrients include calcium (Ca), copper (Cu), magnesium (Mg), manganese (Mn), phosphorus (P), potassium (K), sodium (Na), sulfur (S) and zinc (Zn).^z

Pine wood biochar (PBC)																		
Sample ^y	Acid extraction (mg·L ⁻¹)																	
	Ca	s.d.	Cu	s.d.	Mg	s.d.	Mn	s.d.	P	s.d.	K	s.d.	Na	s.d.	S	s.d.	Zn	s.d.
Top	610.0	134.7	0.80	0.10	196.3	22.9	41.8	10.3	56.7	7.8	1467.7	160.9	16.0	3.5	9.7	3.1	3.3	0.2
Middle	580.0	77.1	0.73	0.06	191.0	35.4	40.3	6.5	55.7	6.7	1501.0	202.5	17.0	7.8	9.0	0.0	3.2	0.8
Bottom	579.7	155.8	0.73	0.12	187.3	53.3	40.1	12.3	53.7	10.4	1409.0	220.3	17.0	7.8	8.7	2.1	3.0	0.8
Consistency range	589.9 ±122.5 ^x		0.75 ±0.09		191.5 ±37.2		40.7 ±9.7		55.4 ±8.3		1459.2 ±194.6		16.7 ±6.4		9.1 ±1.7		3.2 ±0.6	
Batch means ^w																		
PBC #1	617.0		0.77		223.0		43.9		61.3		1513.0		24.0		8.7		3.7	
PBC #2	459.7		0.70		153.0		30.3		46.7		1277.3		13.0		7.7		3.1	
PBC #3	693.0		0.80		198.7		48.0		58.0		1587.3		13.0		11.0		2.8	
Rice hull biochar (RBC)																		
Sample	Acid extraction (mg·L ⁻¹)																	
	Ca	s.d.	Cu	s.d.	Mg	s.d.	Mn	s.d.	P	s.d.	K	s.d.	Na	s.d.	S	s.d.	Zn	s.d.
Top	283.7	52.1	0.30	0.00	166.7	13.6	95.3	11.4	375.0	45.2	2228.7	93.5	42.0	2.6	21.0	2.6	5.4	0.5
Middle	288.3	73.9	0.30	0.10	179.3	22.0	101.9	15.5	398.3	52.0	2206.3	308.9	41.3	4.7	22.7	13.3	5.7	0.9
Bottom	293.3	59.8	0.40	0.17	173.3	23.2	100.0	13.5	370.7	59.7	2181.3	240.1	40.0	4.0	17.3	4.2	5.6	0.6
Consistency range	288.4 ±61.9		0.33 ±0.09		173.1 ±19.6		99.1 ±13.5		381.3 ±52.3		2205.4 ±214.2		41.1 ±3.8		20.3 ±6.7		5.6 ±0.7	
Batch means																		
RBC #1	319.0		0.30		191.3		108.4		423.3		2343.7		44.3		26.7		6.2	
RBC #2	327.0		0.33		174.3		104.6		390.3		2303.0		42.0		18.0		5.5	
RBC #3	219.3		0.37		153.7		84.2		330.3		1969.7		37.0		16.3		4.9	

^zDetermined using the Mehlich-III extraction [reagents include ammonium nitrate, ammonium fluoride, acetic acid, nitric acid and ethylenediaminetetraacetic acid (EDTA); Mehlich, 1984].

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix H: Cation exchange capacity (CEC) and anion exchange capacity (AEC) for biochar materials across batches with standard deviation (s.d), and means from each individual batch.^z

Pine wood biochar (PBC)				
Sample ^y	CEC (mmol·100cc ⁻¹) ^x	<i>s.d.</i>	AEC (mmol·100cc ⁻¹) ^w	<i>s.d.</i>
Top	28.29	6.87	2.41	0.77
Middle	27.59	3.84	2.02	0.77
Bottom	27.19	2.64	2.18	1.03
Consistency range	27.69 ±4.45 ^v		2.20 ±0.86	
Batch means ^u				
PBC #1	32.18		1.81	
PBC #2	23.98		1.62	
PBC #3	26.91		3.19	
Rice hull biochar (RBC)				
Sample	CEC (mmol·100cc ⁻¹)	<i>s.d.</i>	AEC (mmol·100cc ⁻¹)	<i>s.d.</i>
Top	20.41	0.64	21.27	5.29
Middle	22.30	1.34	27.40	7.73
Bottom	20.69	3.53	28.96	9.88
Consistency range	21.13 ±1.84		25.88 ±7.63	
Batch means				
RBC #1	19.80		30.29	
RBC #2	22.47		28.93	
RBC #3	21.13		18.41	

^zMeasured at pH of 8.8 for PBC and 9.5 for RBC.

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xCation exchange capacity measured using procedures described by Kloss et al. (2012) and Dumroese et al. (2011).

^wAnion exchange capacity measured using the procedure described by Lawrinenko (2014).

^vAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^uBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.

Appendix I: pH and electrical conductivity (EC) of biochar materials across batches with standard deviation (s.d.), and means from each individual batch.^z

Pine wood biochar (PBC)				
Sample ^y	pH	<i>s.d.</i>	EC	<i>s.d.</i>
Top	8.83	0.22	1.21	0.14
Middle	8.75	0.19	1.19	0.21
Bottom	8.82	0.15	1.21	0.25
Consistency range	8.8 ±0.19 ^x		1.20 ±0.20	
Batch means ^w				
PBC #1	8.8		1.13	
PBC #2	8.7		1.05	
PBC #3	8.9		1.42	
Rice hull biochar (RBC)				
Sample	pH	<i>s.d.</i>	EC	<i>s.d.</i>
Top	9.50	0.25	1.13	0.19
Middle	9.47	0.18	1.12	0.21
Bottom	9.43	0.21	1.19	0.18
Consistency range	9.47 ±0.21		1.15 ±0.19	
Batch means				
RBC #1	9.4		1.16	
RBC #2	9.3		1.33	
RBC #3	9.1		0.96	

^zpH measured by 1:1 dilution.

^yLocation from which the samples were taken from each batch; each batch is a replication.

^xAverage standard deviation ± the average mean across batches used to determine the acceptable range for consistency.

^wBatch means determined from the data within one batch (n=3), mean must fall within the consistency range (n=9) for that batch to be considered consistent.