Chemical and physical properties of *Paulownia elongata* biochar modified with oxidants for horticultural applications.

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Abstract

Treatment of biochar with oxidants such as acids and hydrogen peroxide has been shown to alter porosity, increase adsorption of chemicals, and introduce functional groups on the biochar surfaces, all of which are desirable for their use in horticultural applications.

Biochar was produced from the pyrolysis of wood from seven-year-old *Paulownia elongata* (PE) trees using a top-lit updraft design stove. PE biochar was subsequently treated with 30% (v/v) sulfuric acid, 30% (w/v) oxalic acid, and 10 and 30% (w/w) H₂O₂.

After thorough rinsing with deionized water to remove residual acids and H₂O₂, biochars were examined for chemical and physical properties. All of the biochars had high carbon percentages, due to the high temperatures (>1000 °C) reached during pyrolysis. Surface areas, micropore surface areas, % micropore surface areas, pH values, cation exchange capacities and electrical conductivities decreased with all oxidant treatments. Analysis of surface functionality by ATR-FTIR showed limited changes resulting from the treatments. TPO/MS analysis showed that all treatments resulted in chars with higher oxidation temperatures suggesting that these are more stable toward oxidation. Unlike reports of biochar derived from different feedstocks and different pyrolysis methods, it appears that treatment of PE biochar with oxidants is unnecessary for its use in horticultural applications.

Keywords: Biochar; carbon sequestration; cation exchange capacity; *Paulownia* wood

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1. Introduction

Over the past decade biochar has been the focus of a great deal of research worldwide, primarily because of its ability to sequester carbon in the soil while improving soil chemical and physical properties (Lal, 2011; Sohi, 2012; Novak et al., 2016). In contrast to other organic material, biochar is highly resistant to decomposition and therefore would be expected to have great longevity in soils (Laird, 2008; Sohi, 2012). While most research on the use of biochars to improve soil fertility has been conducted on agricultural field soils, biochars have also been examined for utilization as components of greenhouse root media (Graber et al., 2010; Dumroese et al., 2011; Altland and Locke, 2012; Tian et al., 2012; Vaughn et al., 2013; Headlee et al., 2014; Kaudal et al., 2015; Vaughn et al., 2015b) and as replacements for peat moss in sand-based golf greens (Brockhoff et al., 2010; Carey et al., 2015; Vaughn et al., 2015a). For both of these applications, biochar chemical and physical properties which are optimum for agricultural soils may not be appropriate for use in root media and golf greens. Desirable properties of greenhouse media, such as stability of organic matter, carbon:nitrogen ratio, bulk density and porosity, have been found to be quite different than properties for most agricultural soils (Nelson, 2011). For example, cation exchange capacity (CEC), which is defined as the number of exchangeable cations per dry weight that a soil or root media are capable of holding at a given pH and is available for plants, can range from values as low as 2 cmol kg⁻¹ in sandy soils to as high as 100 cmol kg⁻¹ in clay soils (Brady and Weil, 2008). A complete root medium should have a CEC value of at least 6 cmol kg⁻¹ (Nelson, 2011). Vermiculite is a common component of root media due its good CEC (~20 cmol kg⁻¹), porosity, and low bulk density. Biochars are generally high in porosity, low in bulk density, and have been shown to have similar CECs to vermiculite (Brewer et al., 2011; Headlee et al., 2014; Novak et al., 2016). Specifications

for United States Golf Association golf green construction recommend a minimum of 90% sand to provide adequate drainage while preventing compaction. Organic matter, generally peat moss, is added to sand during green construction due to the poor water and nutrient retention properties of sand (USGA, 2004; McCoy, 2013). However, peat and most organic materials are prone to decomposition in only a few years. Biochar, which decomposes very slowly but has excellent water and nutrient retention properties, would appear to be an excellent root zone amendment (Bigelow et al., 2004; Vaughn et al., 2015a).

Chemical modification, or activation of biochar, has been studied with a variety of reagents. These include acids (e.g. oxalic, phosphoric and sulfuric), bases (potassium and sodium hydroxides, potassium carbonate), and hydrogen peroxide, all of which have been found to introduce functional groups (-COOH, -OH and -C=O) to biochar surfaces. This activation has been reported to increase the ability of various biochars to adsorb antibiotics, dyes, heavy metals and pesticides (Xue et al., 2012; Feng et al., 2013; Vithanage et al., 2015; Wang et al., 2015; Yakout et al., 2015; Nair and Vinu, 2016). A recent study found that treatment of biochar from bur cucumber (*Sicyos angulatus* L.) with sulfuric and oxalic acids increased surface areas and pore volumes (Vithanage et al., 2015), while H₂O₂ treatment of pine wood biochar significantly increased CEC values (Huff and Lee, 2016).

Paulownia is a genus of fast-growing trees native to China (Zhu et al., 1986). Two species in this genus are being examined as biomass crops in the southeastern U.S., including *P. elongata* (PE) and *P. fortunei* (Yadav et al., 2013). *Paulownia* plantations with 1680 trees ha⁻¹ in central Georgia, USA could yield approximately 100 tons of biomass after three years of growth (Basu et al., 2015). Another study found that 2000 trees ha⁻¹ yielded up to 300 tons of wood annually only 5-7 years after planting (Jiménez

et al., 2005). *Paulownia* trees also respond favorably to short-rotation coppicing, yielding greater biomass than coppiced willow (*Salix viminalis*) (Yadav et al., 2013). Previously, we found increased growth of creeping bentgrass (*Agrostis stolonifera* L.) in simulated golf greens to which biochar produced from PE wood was added compared to bentgrass growth in simulated greens with biochar derived from two other parent materials (Vaughn et al., 2015a).

The objective of the present study was to determine if PE biochar treated with several oxidizing agents (30% (v/v) sulfuric acid, 30% (w/v) oxalic acid, and 10 and 30% (v/v) H₂O₂) would enhance chemical and physical properties of the biochar for use as a component of greenhouse root media and for use in golf greens as a replacement for peat moss.

2. Materials and methods

2.1 Production of biochar

PE biochar was produced using a top-lit updraft (TLUD) stove as previously reported (Vaughn et al., 2015c) from wood chips obtained from main trunks and branches of seven-year-old PE trees grown at Fort Valley State University, Fort Valley, GA, USA. The TLUD stove was fitted with three thermistors located at 5 (thermistor 1), 10 (thermistor 2) and 15 (thermistor 3) cm below the top of the inner fuel cylinder to record the pyrolysis temperatures. Pyrolysis of the biochar was quenched by dumping the biochar into deionized water after pyrolysis was completed. Cooled biochar was further rinsed with additional deionized water (rinse water was not measured for pH and soluble salt content) until no visible ash was present on the biochar pieces (biochar appeared evenly black). Biochar was then dried at 65 °C for 24 h before chemical modifications were performed.

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123 *2.2 Chemical treatment and properties of biochars*

124 Biochar samples (10 g) were placed in 1 L Erlenmeyer flasks to which either 250
125 mL of 30% (w/v) oxalic acid, 30% (v/v) H₂SO₄, 10% H₂O₂ or 30% H₂O₂ were added and
126 shaken for 4 h at 25 °C using an orbital shaker. The biochar samples were subsequently
127 washed with 100 mL aliquots of distilled water ten times under vacuum filtration and
128 then dried in an oven at 65 °C for 24 h. Electrical conductivity (EC) and pHs of the
129 biochars were measured using the methods of [Ameloot et al., 2013](#), and CECs were
130 determined by the method of [Rajkovich et al., 2012](#).

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132 *2.3 Surface area properties*

133 Surface area measurements were performed on a Quantachrome ASiQ
134 (Quantachrome Instruments, Boynton Beach, FL, USA). Samples were degassed at 200
135 °C for 10 h prior to analysis. Surface areas were determined at -196 °C using N₂ as the
136 adsorbate in a relative pressure range of 0.025-0.30 using the Brunauer-Emmett-Teller
137 (BET) method for total surface area calculations. The pressure range used in the
138 calculation for each sample was chosen based on the best linear fit for points in the
139 0.025-0.30 range ([Brunauer et al., 1938](#)). Visual properties of the biochar surfaces were
140 analyzed using a Hitachi Model 3400N II scanning electron microscope (SEM; Hitachi
141 High Technologies America, Inc., Pleasanton, CA). Because the biochar samples were
142 solid and dry, fixation, dehydration and critical point drying steps were not required.
143 Sample fragments were mounted with two sided adhesive carbon disc to specimen stubs
144 and sputter coated (60 seconds using 30 mA current at 10kV) with gold using a Denton
145 Vacuum Desk V Cold Sputter/Etch Unit (Denton Vacuum, LLC, Moorestown, NJ) prior
146 to analysis.

2.4 Elemental composition, density determination of biochars

Elemental composition data of the biochars (using approximately 2 mg of biochar for each measurement) for percent carbon, hydrogen, nitrogen and oxygen were obtained using a Perkin Elmer 2400 CHNO series II Analyzer (Norwalk, CT), with cysteine as the standard. Ash content was determined using a Q2950 (TA Instruments, New Castle, DE) thermogravimetric analyzer by heating samples to 1000°C at 10°C min⁻¹ under an air atmosphere. Density measurements were determined in triplicate and averaged using helium pycnometry on a Micromeritics Accupyc II 1340 using a 10 mL sample cup (Norcross, GA). Densities reported here are more specifically defined as the skeletal density, as helium can penetrate accessible pores, but not internally sealed pores (Brewer et al., 2014).

2.5 Water sorption measurement

Water sorption of the biochar as a function of relative humidity (RH) was measured using a Q5000 SA (TA Instruments, New Castle, DE). The sample was initially dried in the atmospheric chamber under nitrogen at 60°C and 0% humidity for 2 h, followed by treatment at 25°C and 0% humidity for 2 h or until it was stable (less than 0.02% weight change for 15 min). Then the sample was cycled from 0 to 90% relative humidity and then back to 0% RH in 10% increments, holding at each step for 6 h or stabilization of < 0.02% for 20 min was achieved. This allowed both water sorption and the water holding behavior to be observed.

2.6 Fourier transform infrared spectroscopy (FT-IR)

The functionality of ground samples of the original wood, corresponding biochar, and treated biochar samples were examined by FT-IR. The spectra were recorded using a Nicolet iS10 (Thermo Fisher Scientific Inc., Waltham, MA) 370 spectrometer equipped with a dATR SensIR DuraScope, with a single-bounce diamond stage using a scanning range of 550-4000 cm^{-1} for 128 scans averaged at a spectral resolution of 4 cm^{-1} . Spectra were acquired and analyzed with Nicolet Omnic software by subtracting the background air spectrum.

2.7 Temperature programmed oxidation/mass spectrometry (TPO/MS)

In a typical TPO analysis, about 10 mg of sample were placed in a sample tube sandwiched between packings of quartz wool and dried and degassed at 120°C under vacuum for 20 min. Gas flow was then started using a mixture of 5% O_2 in helium (ILMO gas, Jacksonville, IL) set at a flow rate of 40 ml/min and the cell purged for 20 min prior to the start of the measurement. Under this gas flow and at this starting temperature, the sample was heated at 10°C/min to 700°C. The mass spectrometer outputs representing the following m/e were recorded: 18 (H_2O), 28 (CO), 32 (O_2), and 44 (CO_2).

3. Results and Discussion

3.1 Production of biochar and physical properties

A temperature profile of a typical run reached during production of PE biochar in the TLUD stove is shown in [Figure 1](#).

[Figure 1 here](#)

The peak temperature reached during pyrolysis in any run was 1047 °C, although the majority of the temperatures during pyrolysis were lower, between 600 °C and 1000 °C.

Pyrolysis temperature is extremely important in determining several important biochar characteristics, including BET surface areas, CEC and EC values. It has been shown in several studies that biochar porosity increases as pyrolysis temperature is increased with a variety of substrates, including sewage sludge, poultry litter, wheat straw, corn stover, walnut shells, and wood from a variety of species ([Bagreev et al., 2001](#); [Kloss et al., 2012](#); [Mukome et al., 2013](#); [Zhou et al., 2013](#); [Peterson and Jackson, 2014](#); [Tian et al., 2016](#)). These increases in porosity have been up to several orders of magnitude. For example, BET surface areas for biochars produced from pine needles increased from a low value of $0.65 \text{ m}^2 \text{ g}^{-1}$ to an extremely high value of $490.8 \text{ m}^2 \text{ g}^{-1}$ as pyrolysis temperatures were raised from 100°C to 700°C ([Chen et al., 2008](#)).

Data for surface areas, micropore surface areas, and % micropore surface areas, pH, CEC and EC values are shown in [Table 1](#).

[Table 1 here](#)

Untreated biochar had a relatively high BET surface area of $310 \text{ m}^2 \text{ g}^{-1}$, with the 10% H_2O_2 -treated biochar having the lowest value. Micropore surface area and %micropore surface area were also higher in the untreated biochar, with the 30% oxalic acid treated biochar having the lowest values. It has been suggested that biochar suitable as a soil amendment have a surface area of at least $100 \text{ m}^2 \text{ g}^{-1}$ ([Schimmelpfenning and Glaser, 2012](#)), which all of the biochars in this study exceed. While some biochars have been found to have surface areas exceeding $350 \text{ m}^2 \text{ g}^{-1}$ ([Chen et al., 2008](#); [Kaudal et al., 2015](#); [Vaughn et al., 2015c](#)), most reported values are considerably lower, especially at pyrolysis temperatures below 400°C . Increasing fast pyrolysis temperature from 400°C to 500°C raised the BET surface area of wood stock biochars from 2.82 to $156 \text{ m}^2 \text{ g}^{-1}$ ([Mukome et al., 2013](#)). This has not always been found, however, as surface areas of poultry litter and hardwood pellets pyrolyzed at temperatures at 500°C were all found to

be less than $100 \text{ m}^2 \text{ g}^{-1}$ (Tian et al., 2016). CEC, EC and pH values decreased for all of the treated biochars, while density increased with the two H_2O_2 treatments while decreasing with the two acid treatments. While the pH value for the untreated biochar is moderately basic, we found previously that PE biochar incorporated into sand at 5% (v/v) compared to a 100% sand control increased the growth of hydroponically-fertilized creeping bentgrass (Vaughn et al., 2015a). Optimum pH values for soilless substrates have been reported to be between 5.4-6.0, optimal CEC values to be between 6 and 15 cmol kg^{-1} , and optimal EC values from 2.0 to 4.5 dS m^{-1} (Nelson, 2011). For sand-based root zones, typical pH values have been found to range from 7.2 to 8.4, CEC values ranging from 2.0-3.3, and EC values from 0.07 to 0.21, depending on the soil depth measured (McClelland et al., 2009). Brewer et al. (2011) found that the addition of various biochars to an agricultural loam soil had significant increases in EC, but little effect on CEC, as unamended soil had a relatively high (26 cmol kg^{-1}) CEC. Although we found decreases in CEC with the various oxidants, previous research found increased CEC values when pinewood biochar was treated with H_2O_2 , due to the addition of acidic oxygen functional groups (Huff and Lee, 2016).

Transverse SEM images of the untreated and oxidant-treated biochars (Figure 2)

Figure 2 here

do not show large differences in structural characteristics between treatments. Evidently, chemical treatment had little physical effect on PE biochar structure. This suggests that PE biochar structure is stable to chemical treatments which contrasts to its chemical stability as shown previously. Both untreated and oxidant-treated biochars have a mixture of large ($50\text{-}120 \text{ }\mu\text{M}$) and small ($5\text{-}50 \text{ }\mu\text{M}$) vascular vessels with generally smooth, intact walls. Some evidence of degradation of the vessel walls can be seen in the 30% H_2SO_4 (Figure 2c) and 30% oxalic acid (Figure 2d) treated biochars, but this did not

occur in either of the H₂O₂ treatments compared to untreated biochar. Due to the extensive washing of all the biochars after pyrolysis there is also little or no visible ash in the images.

3.2 Chemical properties

The ash, CHNO analysis values, H/C and O/C ratios for the biochars are shown in [Table 2](#).

[Table 2 here](#)

As expected, percentage ash was highest in the untreated biochar and lowest in the 10% H₂O₂ biochar. Clearly the chemical treatments and subsequent washings reduced ash contents. Carbon values were similar among treatments, with only the 30% H₂O₂ biochar having a significantly lower value. Guidelines for biochar H/C and O/C ratios recommend an H/C ratio <0.6 and an O/C ratio <0.4 ([Schimmelpfenning and Glaser, 2012](#)). All of the values we found in our study followed these guidelines, with the O/C ratio of the untreated PE biochar being especially low. Lower O/C ratios are positively correlated with greater chemical stability and hence longevity in soils.

The water sorption results for all biochar samples are summarized in [Table 3](#), [Table 3 here](#)

arranged by decreasing peak moisture content. From these results it can be seen that treating Paulownia biochar with the oxidants in this study reduced its overall water sorption capacity; note how the untreated control has the highest water sorption at 90% relative humidity ([Figure 3](#)).

[Figure 3 here](#)

Water retention of the biochar control was minimal compared to previously studied coppiced wood samples ([Vaughn et al., 2015c](#)), and after treatment with acid was smaller

yet, with virtually no appreciable water retention capacity for those samples treated with 30% H₂O₂, sulfuric, or oxalic acid.

Figure 4 shows the FT-IR spectra of the starting PE wood species, its

Figure 4 here

corresponding biochar, and the four chemically treated biochars. The FTIR spectrum of the starting PE wood sample (Figure 4, A) exhibited peaks that correspond to the cellulosic, hemicellulosic, and lignin functionalities found in wood (Schwanninger et al., 2004) and were similar to those determined for various coppiced wood samples we have reported previously (Vaughn et al., 2015). Although a simple interpretation of the spectrum is challenging, the broad peak centered at 3333 cm⁻¹ is indicative of the hydroxy O-H stretching vibration contained in cellulose and hemicelluloses structures. The band at 2850-3000 cm⁻¹ corresponds to the aliphatic sp³ hybridized C-H stretching vibrations of methylene methyl, and methoxyl groups (Schwanninger et al., 2004). The broad peak centered at 1026 cm⁻¹ accompanied by the smaller peak at 1236 cm⁻¹ is representative of various C-O and O-C-O stretching vibrations associated with carbohydrate structures. The carbonyl C=O of carboxyl, ester, aldehyde, and ketone stretching vibrations can be observed at 1738 cm⁻¹ while various types of aromatic C=C stretching vibrations, CH₂ scissoring, and C-H plane deformations were observed between 1400-1600 cm⁻¹.

In contrast, the FT-IR spectrum of untreated biochar (Figure 4, B) was devoid of many spectral signals corresponding to the hydroxyl O-H, aliphatic C-H, carbonyl C=O, and carbohydrate C-O stretching observed in the starting PE wood sample indicating that much of the functionality had been consumed during the preparation of the biochar and converted into a carbon-rich product. The biochar exhibited weak peaks at 1585 cm⁻¹ corresponding to corresponding to C=C stretching in addition to bands at 872, 804, and

747 cm^{-1} which are characteristic for aromatic C-H out of plane deformations (Yakout, 2015) that suggest the formation of aromatic structures during pyrolysis of the PE wood.

Treatment of biochar with chemical oxidants such as H_2O_2 , sulfuric acid or oxalic acid has been shown as a method to introduce oxygen-containing functional groups onto the surface of the biochar (Huff and Lee, 2016; Vithanage, M. et al., 2015; Xue et al., 2012; Yorgun and Yildiz, 2015). Accordingly, the PE biochar prepared in this study was treated with 10 and 30 % H_2O_2 (Figure 4, C and D), 30 % sulfuric acid (Figure 4, E), and 30 % oxalic acid (Figure 4, F). As in the untreated biochar spectrum, all the treated biochar samples showed absorbances at 1585 cm^{-1} and at 872, 804, and 747 cm^{-1} , representing the presence of aromatic functionality. In the case of H_2O_2 treatment (Figure 4, C and D), the spectra are nearly the same as the untreated biochar and suggest that the H_2O_2 only minimally altered the biochar surface. Huff and Lee (2016) obtained similar results when they treated biochar derived from pinewood biomass with H_2O_2 . The FT-IR spectra of the biochar treated with either 30 % sulfuric acid or oxalic acid (Figure 4, E and F) were similar in appearance and the peaks at 872, 804 and 747 cm^{-1} appear to have become more pronounced. One would expect that successful functionalization by sulfuric acid would introduce HSO_3 groups onto the biochar surface for which the corresponding S=O stretching vibrations at approximately 1034 cm^{-1} and 1140 cm^{-1} may be observable. Although, there is some evidence that a peak at 1034 cm^{-1} may be present, unfortunately, it is very weak and masked by the broad peak located in the general vicinity.

Feng et al. (2013) esterified the hydroxy functionality present in straw biomass using oxalic acid as a method to introduce carboxylic acid groups. By FT-IR treatment of the PE biochar with oxalic acid appears to be minimal as there was likely no residual hydroxyl groups present in the biochar to be esterified by oxalic acid.

The biochars were also examined using temperature programmed oxidation studies. In these measurements, the samples are heated under a stream of 5% O₂ in helium and the resulting gases of combustion are recorded. The temperatures at which oxidation occurs and the relative quantities of the product gases are indicative of oxidative stability (Jackson et al., 2013; Elkasabi, 2015). The results are shown in Figure 5. Untreated PE biochar shows a simple gas evolution profile with a peak oxidation temperature of 509°C in which the gases are produced roughly in parallel. This indicates that the char contains a high level of homogeneity in carbon structure. The very low level of H₂O produced indicates that there is little hydrogen in the biochar and since the H₂O evolution occurs at high temperature shows that the hydrogen is bound to aromatic carbon. The profile shows a CO₂/CO ratio of 4.0. The chemical treatments all resulted in chars with higher temperatures of oxidation and lower CO₂/CO ratios and suggests that these chars are more resistant to oxidation. In terms of improving PE biochar for use in greenhouse and field applications, this added oxidative stability is more attractive for the latter where its long-term stability makes it an attractive replacement for peat moss.

4. Conclusions

The results found in the present study indicate that chemical modification is unnecessary for PE biochar, and the unmodified biochar has more desirable characteristics than any of the modified biochars. The high porosity and CEC of the untreated biochar would indicate that it would have excellent characteristics for potting medium, and would be expected to have enhanced oxidative stability due to the TPO profiles. We are conducting further experiments with PE biochar as a component of horticultural potting substrates and as a component of sand-based root zones.

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Table 1

Surface areas, densities, pHs, CECs and ECs of biochars

Sample	Surface area (m ² g ⁻¹)	Micropore surface area (m ² g ⁻¹)	% Micropore surface area	pH	Density (g cm ⁻³)	CEC (cmol kg ⁻¹)	EC (dS m ⁻¹)
Untreated biochar	310	249	80	9.4	1.77	27.4	4.1
Treated with 10% H ₂ O ₂	165	118	72	7.1	1.83	4.4	0.1
Treated with 30% H ₂ O ₂	272	192	71	6.9	1.86	7.2	0.1
Treated with 30% H ₂ SO ₄	208	141	68	2.7	1.68	10.3	1.7
Treated with 30% oxalic acid	197	110	56	7.2	1.60	2.2	0.1

Table 2Ash and CHNO analysis of biochars

Sample	Ash (%)	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Oxygen (%) ^a	H/C ratio	O/C ratio
Untreated PE biochar	4.08	88.08 ± 0.83	1.23 ± 0.37	0.17 ± 0.10	6.44	0.014	0.073
Treated with 10% H ₂ O ₂	1.47	86.68 ± 2.02	0.73 ± 0.18	0 ^b	11.12	0.008	0.128
Treated with 30% H ₂ O ₂	2.29	82.27 ± 1.51	0.60 ± 0.13	0.02 ± 0.02	14.82	0.007	0.180
Treated with 30% H ₂ SO ₄	1.88	87.03 ± 1.88	1.06 ± 0.16	0 ^b	10.03	0.012	0.115
Treated with 30% oxalic acid	1.61	84.98 ± 3.65	0.85 ± 0.17	0 ^b	12.56	0.010	0.148

^aOxygen was calculated as (100-(ash + carbon + hydrogen + nitrogen)).^bNo detectable nitrogen measured.

Table 3

Water sorption properties of biochars

Sample	Peak moisture content g H ₂ O/g sample	Maximum difference	RH @ maximum difference
Untreated biochar	0.1324	0.0063	40
Treated with 10% H ₂ O ₂	0.1141	0.0051	50
Treated with 30% H ₂ O ₂	0.1021	0.0016	50
Treated with 30% H ₂ SO ₄	0.0896	0.0019	60
Treated with 30% oxalic acid	0.0880	0.0018	50

Figure 1. Temperature profile of pyrolysis of PE wood in a TLUD stove equipped with three thermistors. Temperature of thermistors 1-3 are shown as a function of elapsed time from ignition of the feedstock.

Vaughn et al., Figure 1

Figure 2. Transverse SEM images of: untreated PE biochar (A); PE biochar treated with 10% H_2O_2 (B); PE biochar treated with 30% H_2SO_4 (C); PE biochar treated with 30% oxalic acid (D).

Vaughn et al., Figure 2

Figure 3. Moisture sorption characteristics of : (A) untreated biochar; (B) 30% sulfuric acid treated biochar; (C) 10% H_2O_2 treated (circles) and 30% H_2O_2 treated (diamonds) biochar; and (D) 30% oxalic acid treated biochar. Humidity is controlled and increased from 0 to 90%, then brought back down to 0 (see solid arrows in plot A). For each sample the maximum difference for a given relative humidity was measured. The peak moisture content was the value at 90% relative humidity for each sample.

Figure 4. FT-IR spectra of: (A) PE wood flour; (B) Untreated PE biochar; (C) PE biochar treated with 10 % H_2O_2 ; (D) PE biochar treated with 30 % H_2O_2 ; (E) PE biochar treated with 30 % H_2SO_4 ; (F) PE biochar treated with 30 % oxalic acid.

Figure 5. Gas evolution profiles from the temperature programmed oxidations of PE biochars that were: untreated (A); treated with 30% H_2SO_4 (B); treated with 30% oxalic acid (C); and treated with 30% H_2O_2 (D).