# Characterization of Biochars and Their Use as an Amendment to Acid Soils

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**Abstract:** Biochar, because of its porous nature, calcium carbonate ($CaCO_3$) equivalent, surface functional groups, and other properties, could serve as an acid soil amendment. To investigate the liming potential of biochars, laboratory characterization and greenhouse and field experiments were conducted in Hawaii and West Java, Indonesia, respectively. Six wood-derived biochars were characterized and amended to a Hawaiian acid soil (pH 4.6, exchangeable aluminum [Al] 1.8 cmolc $kg^{-1}$) at 2% and 4% alone or in combination with 2 cmolc $kg^{-1}$ of lime and then planted with *Desmodium intortum* (a forage legume sensitive to Al) twice in a greenhouse trial. To the Indonesian acid soils (pH 3.9–4.0, exchangeable Al 8–14 cmolc $kg^{-1}$), a rice husk and a lac tree biochars at 4% and 8% alone or in combination with lime at 4 and 8 cmolc $kg^{-1}$ and compost at 0.1 and 0.2% were applied and then planted with soybean (*Glycine max*) cv. Anjasmoro twice in field trials. Biochar effects on soil properties and plant growth were measured. The results indicated that the six biochars varied in pH, ash content, $CaCO_3$ equivalent, total basic cations, cation exchange capacity, and other properties (pore size, surface functional groups). Soil pH was increased, soil exchangeable Al was reduced, and plant nutrients were enriched to different degrees upon additions of biochars. Total dry weights of *Desmodium* were increased 2- to 4-fold over the control or lime treatment upon applications of biochar. Similar effects on soils and soybean were obtained for the Indonesian field trials. It was concluded that $CaCO_3$ equivalent and total basic cations were among the most important properties of biochar responsible for improving acid soil productivity and plant growth.

**Key Words:** Biochar, exchangeable Al, liming potential, soil acidity

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Biochar is a relatively new term that is used to define the product of thermal decomposition of biomass or biowaste under a limited supply of oxygen. Its production was inspired by the discovery of the anthropogenic Amazonian Dark Earth or Terra Preta, which had a higher nutrient content, cation exchange capacity (CEC), and organic matter than the surrounding soils (Glaser et al., 2000; Lehmann et al., 2002; Lehmann and Joseph, 2009). Recently, biochar has often been produced in a pyrolytic process for specific purposes, such as soil amendment, carbon sequestration, or environmental management (Glaser et al., 2009; Lehmann and Joseph, 2015; Marris, 2006).

Because biochars can be produced from a variety of feedstocks and under different production processes and conditions, they have different physical and chemical properties (Antal and Grønli, 2003). Thus, they potentially have different effects when applied to soils. Singh et al. (2010) reported significant differences in pH, CEC, ash content, surface basidity and acidity, lime equivalent, and nutrient content of 11 biochars made from wood, manure, leaf, papermill sludge, and poultry litter produced at 400°C and 500°C, with and without steam activation. Enders et al. (2012) showed a wide range of ash content, volatile matter (VM), and various forms of carbon (fixed, organic, total, and inorganic C), total O, N, H, pH, and exchangeable bases of 94 biochars made from various feedstocks and temperatures. Keiluweit et al. (2010) also found that wood pine biochar differed from tall fescue grass biochar with respect to their VM; fixed C; ash content; C, N, H, and O content; H:C and O:C ratios, and surface area. In addition, Mukherjee et al. (2011) observed a different surface chemistry represented by pH, VM, ash content, surface area, CEC, anion exchange capacity, point of zero net charge, zeta potential, isoelectric point, and the distribution of surface functional groups of oak, pine, and grass biochars laboratory produced at different pyrolysis temperatures. Lee et al. (2010) showed that biochar produced from the same cornstover under fast pyrolysis at 450°C had CEC and O:C ratio higher than those obtained from gasification at 700°C. Such results show a need for biochar characterization before its use as a soil amendment.

Biochar has been shown as a promising and environmental-friendly soil amendment for sustainable agriculture and climate change mitigation (Glaser et al., 2002). Among the beneficial effects is liming potential—the capacity of biochar to increase soil pH and reduce Al toxicity in acid soils. These effects of biochars reportedly depend on their ash and VM content (Deenik et al., 2011). Soluble salts, such as potassium and sodium carbonates and oxides, can cause an increase in pH in the water film around biochar particles (Joseph et al., 2010). The liming effect of crop residue biochars was more pronounced when the pyrolysis temperature was increased to 500°C to 700°C (Wan et al., 2014). Liming effect could also be due to functional groups on biochar surface, such as phenolic and carboxylic acids, especially at lower (350°C–450°C) pyrolysis temperatures (Boehm, 1994; Rutherford et al., 2008; Cheng et al., 2008; Keiluweit et al., 2010; Wan et al., 2014). The objectives of this study were to characterize selected biochars produced from different feedstocks and to evaluate their liming effects on Hawaiian and Indonesian acid soils.

## MATERIALS AND METHODS

### Biochar Collection and Characterization

Six biochars for characterization and greenhouse trial were collected from Indonesia and Hawaii. Five biochars were made by farmers in an open fire process with a 300°C to 450°C range of temperatures in West Timor, Indonesia. They are leucaena (*Leucaena leucocephala*), lac tree (*Schleichera oleosa*) referred to as lac tree 450, she oak (*Casuarina junghuhniana*), mahogany (*Swietenia macrophylla*), and mountain gum (*Eucalyptus urophylla*) wood-derived biochars. A mixed wood (scrapped wood and tree trimmings)–derived biochar was produced by Landscape Ecology Corporation, Hilo, Hawaii. All coarse biochars were air dried followed

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by oven drying at 70°C for 48 h, crushed, sieved to pass a 60-mesh sieve (0.25 mm), and stored before use. Two other biochars for field trial were collected from Java, Indonesia. The lac tree wood biochar was made in a mound kiln at 500°C to 600°C by farmers in Ponorogo, East Java (referred to as lac tree 600), and the rice husk biochar produced at 290°C to 320°C by a traditional charcoal making process by farmers in Jasinga, West Java, Indonesia.

Biochar pH was measured after biochar and deionized water (1:5) were mixed and equilibrated for 1 h, whereas biochar electrical conductivities (ECs) were measured after the same mixtures were equilibrated for 24 h. Biochar moisture, ash, and VM contents were measured using the American Society for Testing and Materials method (D-1762-84) (ASTM, 1990). Moisture content was measured as the weight loss after heating biochars at 105°C for 24 h. The VM content was determined as the weight loss after heating biochars in a covered crucible at 950°C for 6 min. The ash content was determined as the weight loss after combusting biochars at 750°C for 6 h. Fixed C in a biochar was calculated as follows (Antal et al., 2003): fixed C (%) = (100 − VM − ash) %.

Biochars' calcium carbonate ($CaCO_3$) equivalent (CCE) was determined by a rapid titration according to the procedure described by Rayment and Higginson (1992). Briefly, 2.00 g (dry weight) of biochar was added to 50 mL of 1 M HCl, shaken for 1 h, equilibrated overnight, and shaken again for 1 h before titrating with 0.5 M NaOH.

To measure functional groups on the surface of biochars, three procedures were used. First, a Fourier transform infrared (FTIR) spectrometer (Nicolet 6700) was used to generate the unique peak of functional groups on each biochar. Briefly, biochar powder was placed and pressed into a sample cell (Smart ZnSe 450), then scanned from 4,000 to 700 wavenumbers. The setup consisted of atmosphere air as the background; number of scan, 256; and resolution, 8.The peak of functional groups obtained from the FTIR scan was identified with the aid of the OMNIC program (Thermo Fisher Scientific, Inc., Waltham, MA) for Windows. Second, a solid-state $_{13}C$ magic angle spinning nuclear magnetic resonance (MAS $_{13}C$-NMR) was used to identify chemical compounds contained within biochars. The procedure was referred to McBeath et al. (2011). Spectra were obtained at a frequency of 100.6 MHz using a Varian Unity INOVA 400 NMR spectrometer (Varian Inc. [now is a part of Agilent Technologies], Palo Alto, CA). Samples were packed in 7-mm diameter cylindrical zirconia rotors with Kel-F rotor end caps (Doty Scientific, Inc., Columbia, SC) and spun at the magic angle (54.7°) at 6,000 ± 50 Hz in a Doty Scientific supersonic MAS probe (Doty Scientific, Inc.). Free induction decays were acquired with a sweep width of 45.454 kHz; 1,216 data points were collected over an acquisition time of 12 ms. All spectra were zero filled to 8,192 data points and processed with a 50-Hz Lorentzian line broadening and a 0.010-s Gaussian broadening. Chemical shifts were externally referenced to the methyl resonance of hexamethylbenzene at 17.36 ppm. Cross polarization spectra represent the accumulation of 4,000 scans and were acquired using a 90° 1H pulse of 5- to 6-μs duration, a 1-ms contact time, and a 2-s recycle delay. Third, the Boehm titration method (Boehm, 1994) was used to quantify the carboxylic and phenolic functional groups on biochar. Water-soluble salts and carbonates in biochar were removed before the titration. Briefly, 0.50 g of fine biochar was added to 50 mL of each of the three 0.05 M bases: $NaHCO_3$, $Na_2CO_3$, and NaOH. The mixtures, along with a control solution without biochar, was shaken for 24 h and then filtered (Whatman no. 42 paper) to remove particles. Then, a 5-mL aliquot from each filtrate was mixed with 10 mL of 0.05 M HCl to ensure complete neutralization of bases and then back-titrated with 0.05 M NaOH solution. The endpoint was determined using a pH meter and phenolphthalein color indicator. The total surface acidity was calculated as the quantity neutralized by NaOH, the carboxylic acid fraction as the quantity neutralized by $NaHCO_3$, and the lactonic fraction as that neutralized by $Na_2CO_3$. The difference between quantity of NaOH and $Na_2CO_3$ is assumed to be the phenolic functional group content (Rutherford et al., 2008).

Total C, nitrogen (N), and hydrogen (H) were determined using an Exeter Analytical CE 440 Elemental Analyzer (Exeter Analytical, Inc., North Chelmsford, MA). Oxygen (O) was calculated as follows: percentage of O = (100-C-H-N) %. Atomic ratio of O:C and H:C was obtained by calculation. Other nutrients in biochars were analyzed as follows. Briefly, 0.50 g of biochar was weighed and transferred into a 50-mL digestion tube. Five milliliters of the acid mix (70% of $HNO_3$ and 30% of $HClO_4$) was added. The tube was placed in a block digester and heated at 150° C for 1.5 h. After cooling, the volume was brought to 50 mL with deionized water, filtered through a Whatman no. 42 paper, and then transferred into a specialized tube for reading with an inductively coupled plasma (ICP) spectrometer (Perkin-Elmer model Optima 7000DV; PerkinElmer, Inc., Waltham, MA).

Surface structure and porosity of biochars were measured by mounting the biochar powders (passed through a 60-mesh sieve) onto aluminum (Al) stubs (sample holder) with conductive carbon double tape, then reading with a scanning electron microscope (SEM) (HITACHI; Hitachi High-technologies, Corp., Tokyo, Japan). Pore diameters were measured from 2,000-time magnified SEM images. Chemical composition of selected biochars was further analyzed with an energy-dispersive spectroscopy (EDS) that was attached to the SEM.

Cation exchange capacity of biochars was measured in three replicates by using the ammonium acetate ($NH_4OAC$) pH 7.0 method (Chapman, 1965). Briefly, 2.00 g of biochar was added to 100 mL of 1 M $NH_4OAC$, pH 7.0, occasionally shaken for 24 h; vacuum filtered using a Buchner funnel lined with filter paper no. 6S; then rinsed with an additional 20 mL of $NH_4OAC$. Biochar in the Buchner funnel was washed four times with 10 mL of methyl alcohol and transferred into an Erlenmeyer; 50 mL of 4% KCl was added, shaken for 30 min, filtered using another Buchner funnel, and washed three times with 4% KCl. Forty-milliliter aliquot was transferred to a micro-Kjeldahl flask. To the flask, 1 mL of 1 M NaOH was added, the solution was distilled, and the distillate was titrated with standardized 0.04 M HCl.

## Greenhouse Experiment

Acidity amending capacity of biochars was studied in a greenhouse experiment using an acid Ultisol (Ustic Kanhaplohumults, Leilehua series), from Hawaii. Soil samples were air dried and sieved to pass a 4-mm sieve for the pot experiment and a 0.5-mm sieve for chemical analysis. Six biochars (leucaena, lac 450 tree, she oak, mahogany, mountain gum, and Hilo mixed wood) were crushed, sieved to pass a 60-mesh sieve, and stored before use. A hydrated lime (Bandini; Bandini Fertilizer Co., Los Angeles, CA) with a CCE of 108% (pure $CaCO_3$ was assigned to 100%) was dried and sieved through a 60-mesh sieve before use. In its natural state, the soil had a pH of 4.6, 2.4 cmolc $kg^{-1}$ acidity, 1.8 cmolc $kg^{-1}$ exchangeable Al, and 16.8 cmolc $kg^{-1}$ CEC.

The treatments, consisting of biochar and lime, were arranged in a 6 × 3 × 2 factorial completely randomized design with three replicates. The six biochars were applied at three rates: control (soil without biochar), 2% biochar, and 4% biochar. The lime treatments were control (soil without lime) and 2 cmolc $kg^{-1}$ factorially superimposed on the biochar treatments. A 2-kg soil was mixed with biochar and/or lime, watered, and then transferred into a pot. Basal nutrients were added to all treatments (in mg $kg^{-1}$):

160 N, 160 $P_2O_5$, and 160 $K_2O$ from a 16-16-16 commercial fertilizer. After 4 weeks of incubation, all pots were planted twice with *Desmodium intortum* cv. Greenleaf, an Al-sensitive forage legume, as the test plant (one plant per pot). *Desmodium* was harvested after 37 days of growth. The shoots were cut, and the roots were carefully removed from the soil. Both were washed with tap water and then with deionized water three times before oven drying at 70°C for 50 h. Soil samples were collected from each pot, air dried, crushed, and passed through a 0.5-mm sieve before analysis. Selected soil chemical properties, namely, soil pH ($H_2O$ 1:1), total acidity, exchangeable Al, and CEC, were measured using the same methods described previously.

Shoot and root dry weights were recorded and then ground separately for tissue analysis. A 0.10-g sample was dry digested in a muffle furnace at 500°C for 4 h. Four milliliters of 1 M $HNO_3$ was added to dissolve the ash and then heated at 150°C on a hotplate until dry. Fifteen milliliters of 0.1 M HCl was added, stirred, and filtered into an ICP tube for analysis.

## Field Experiment

To verify the amending capacity of biochars to acid soils at field level, field experiments were conducted in West Java, Indonesia, in 2013–2014 seasons. The first site was at Guradog, Lebak district, Banten province (6°30′45.65″ S, 106°22′43.38″ E). The soil at this site was a Typic Paleudult, pH 4.0, and exchangeable Al 8.0 cmolc $kg^{-1}$. The second site was at Jasinga, Bogor district, West Java province (6°28′1.24″ S, 106°28′34.62″ E). The soil at this site was a Typic Hapludult, pH 3.9, and exchangeable Al 14.0 cmolc $kg^{-1}$.

Two biochars, lac tree wood 600 and rice husk, with properties as described in Table 1, were selected based on a pot trial with five biochars (lac tree wood 600, rice husk, leucaena wood, she oak wood, mountain gum wood) and two soils. The pot experiment was conducted at the University Farm, Bogor Agriculture University, Darmaga, Bogor, Indonesia, in 2013.

Results from this pot trial showed that lac tree wood 600 biochar was superior to the others for soil productivity improvement and soybean growth. The rice husk biochar resulted in poor soybean growth. To compare the liming value of the biochars, a local lime (CaO with CCE of 104%) was used alone or in combination with the biochars. Because of low nutrients in these two highly weathered tropical soils, a local thermo-compost (pH [1:5]: 7.8, EC: 0.61dS $m^{-1}$, CEC: 38 cmolc $kg^{-1}$, C/N: 8.3, N: 1.79%, P: 0.22%, K: 0.25%, Ca: 0.69%, Mg: 0.11%, Fe: 3,792 mg $kg^{-1}$, Mn: 335 mg $kg^{-1}$, Al: 7,702 mg $kg^{-1}$) was also used in various combinations with the biochars and lime.

The biochars were applied at 4% and 8% (96 and 192 Mg $ha^{-1}$) alone or in combination with lime at 4 and 8 cmolc $kg^{-1}$ (3.5 and 7 Mg $ha^{-1}$) and compost at 0.1% and 0.2% (2.5 and 5 Mg $ha^{-1}$). All treatments were arranged in a randomized complete block design with four replicates. The sites were prepared by cutting reed grass, the original vegetation cover, followed by plowing, harrowing, and then establishing 52 plots. Plot size was 280 × 150 × 20 cm. Biochars alone or in combination with lime or compost were poured, spread, and mixed thoroughly with soils, watered, and incubated for 34 days (October 30 to December 2, 2013) before sampling and planting. Soybean (*Glycine max*) cv. Anjasmoro, a local cultivar that is highly sensitive to soil acidity, was planted twice as the test plant. The first planting started on December 3, 2013, and plants were harvested on January 13, 2014. The second planting at the Jasinga site was started on February 1, 2014, and the plants were harvested on March 11, 2014, whereas the second planting at Guradog site was started on March 10, 2014, and harvested on April 16, 2014.

Soil samples were collected from each plot, air dried, crushed, and passed through a 2-mm sieve before analysis. Soil pH, exchangeable Al, and CEC were measured. Plant dry weights were recorded separately for shoot and root after being washed with tap water and then deionized water and oven dried at 70°C for 4 days. Dry shoot and root samples were then collected, crushed, sieved, and ashed for chemical analysis with an ICP spectrometer.

## Statistical Analysis of Data

Descriptive analysis was used to calculate means and S.E.M. from three or four replicates of measured soil pH and CEC, plant dry weight, and nutrient content in plant tissues. The relationships between biochar properties and soil properties or plant growth

**TABLE 1.** Mean and S.E. of Selected Properties of Eight Biochars

| | Biochars | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Selected Properties** | **Leucaena Wood** | **Lac tree Wood 450** | **Hilo Mixed Wood** | **She Oak Wood** | **Mahogany Wood** | **Mountain Gum Wood** | **Lac Tree Wood 600** | **Rice Husk** |
| pH (1: 5) | 10.3 | 10.4 | 9.5 | 10.2 | 5.0 | 4.2 | 9.0 | 7.6 |
| EC (dS $m^{-1}$) | 2.1 ± 0.04 | 2.3 ± 0.02 | 2.4 ± 0.01 | 0.9 ± 0.02 | 0.1 ± 0.00 | 0.1 ± 0.00 | 1.9 ± 0.02 | 1.3 ± 0.01 |
| CEC (cmolc(+) $kg^{-1}$ | 20.1 ± 0.5 | 17.3 ± 1.3 | 14.7 ± 0.2 | 13.9 ± 0.6 | 45.1 ± 0.5 | 44.9 ± 0.5 | 17.7 ± 0.45 | 33.1 ± 0.85 |
| CCE (%) | 28.6 ± 1.5 | 24.1 ± 0.4 | 8.4 ± 0.4 | 8.4 ± 0.4 | 5.0 ± 0.4 | 4.2 ± 0.4 | 13.7 ± 0.46 | 1.2 ± 0.07 |
| Phenolics (mg $kg^{-1}$) | 27.4 ± 0.8 | 35.2 ± 0.3 | 25.1 ± 0.2 | 28.3 ± 0.5 | 532.4 ± 7.5 | 9,621.9 ± 341.9 | — | — |
| Ash content (%) | 30.7 ± 0.03 | 15.8 ± 0.04 | 19.6 ± 0.2 | 4.6 ± 0.1 | 7.3 ± 0.1 | 0.7 ± 0.0 | 11.7 ± 0.03 | 48.9 ± 015 |
| Volatile matters (%) | 33.3 ± 1.2 | 28.1 ± 0.03 | 23.7 ± 0.5 | 22.9 ± 1.8 | 53.3 ± 1.0 | 36.4 ± 0.2 | 40.5 ± 0.63 | 43.7 ± 0.17 |
| Fixed C (%) | 35.9 ± 1.2 | 56.1 ± 0.1 | 56.7 ± 0.4 | 69.8 ± 1.7 | 39.3 ± 1.2 | 62.9 ± 0.2 | 47.8 ± 0.12 | 7.4 ± 0.11 |
| Total C (%) | 55.2 ± 0.6 | 68.2 ± 0.9 | 64.5 ± 0.9 | 71.2 ± 5.1 | 61.9 ± 1.6 | 69.1 ± 2.1 | 72 ±0.98 | 37 ± 0.68 |
| H (%) | 1.0 ± 0.07 | 1.4 ± 0.04 | 1.5 ± 0.06 | 2.0 ± 0.04 | 3.0 ± 0.04 | 2.3 ± 0.09 | 2.2 ± 0.06 | 1.4 ± 0.04 |
| O (%) | 43.2 ± 0.6 | 29.9 ± 0.9 | 33.6 ± 0.9 | 26.4 ± 5.2 | 34.7 ± 1.7 | 28.3 ± 2.2 | 25.4 ± 0.23 | 48.2 ± 0.69 |
| O:C | 0.59 ± 0.01 | 0.33 ± 0.02 | 0.39 ± 0.02 | 0.30 ± 0.08 | 0.42 ± 0.03 | 0.31 ± 0.03 | 0.26 ± 0.01 | 0.98 ± 0.01 |
| H:C | 0.22 ± 0.01 | 0.24 ± 0.01 | 0.27 ± 0.01 | 0.34 ± 0.02 | 0.59 ± 0.01 | 0.40 ± 0.01 | 0.37 ± 0.02 | 0.45 ± 0.02 |

EC, electric conductivity; lac tree 450, produced at 300°C to 450°C; lac tree 600, produced at 500°C to 600°C.

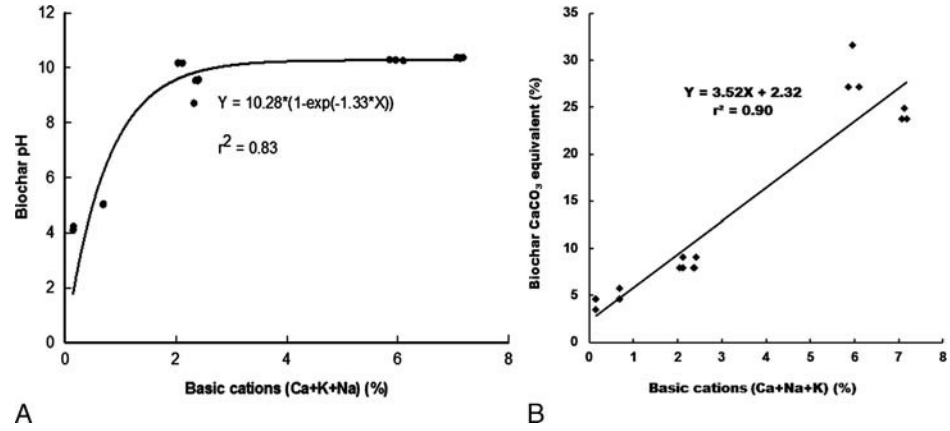


**FIG. 1.** Relationship between biochar pH and basic cations (A) and between biochar CCE and basic cations (B).

were analyzed using regression analysis and presented in the combined scatter and fitted line graphs using Microsoft Excel 2010 (Microsoft Corp., Redmond, WA) and Sigmaplot 11.0 software (Systat Software, Inc., San Jose, CA). Histogram figures of soil property changes and plant growth differences resulting from the biochar application were drawn using Microsoft 2010 Excel software. Treatment effects on soil properties and plant growth were analyzed by a two-way analysis of variance using PROC ANOVA GLM of the SAS 9.2 software, and Tukey tests at $P \leq 0.05$ were performed to test the significance.

## RESULTS AND DISCUSSION

### Selected Properties of Biochars

Selected properties of biochars are shown in Table 1. Biochars' pH ranged from 4.2 to 10.4 and positively correlated with base cations (Fig. 1A), which in turn positively correlated with CCE (Fig. 1B). Biochars' EC ranged from 0.1 to 2.4 dS $m^{-1}$, their CEC varied from 13.9 to 45.1 cmolc $kg^{-1}$, and VM content from 22.9% to 55.3%. Total phenolics varied from 25.1 to 9,621.9 μg $g^{-1}$ (based on gallic acid equivalent), with the highest content being from the mahogany and mountain gum biochars. Leucaena-, lac tree 450–, mix wood–, and she oak wood–derived biochars had alkaline pH and high CCE and EC. In contrast, the mountain gum and mahogany wood–derived biochars had acid to slightly acid pH and high CEC, VM, and total phenolic content.

Elemental content varied with the type of biochars (Table 2). Leucaena, lac tree 450, Hilo mixed wood, and she oak biochars had higher content of N, P, Na, K, Mg, Ca, Si, B, Cu, and Zn than mountain gum and mahogany biochars. The results reflect the differences in feedstock. In fact, nutrient content of biochars was reportedly determined mostly by feedstock mineral composition, not by the pyrolysis temperature (Brewer, 2012; Gaskin et al., 2008; Zhao et al., 2013).

Total C, H, and O ranged from 55.2% to 71.2%, from 1.0% to 3.0%, and from 26.4% to 43.2% respectively (Table 1), and calculated O:C and H:C ratios ranged from 0.2 to 0.6. Based on the O:C ratio and fixed C content (Spokas, 2010), the stability order of the test biochars was leucaena < mahogany < mixed wood < lac tree 450 ≈ mountain gum ≈ she oak. The use of O:C ratio as an indicator of stability was supported by the work of Crombie et al. (2013), who showed a strong correlation among three stability indicators (including O:C ratio) for pine wood–, rice husk–, and wheat straw–derived biochars.

Surface functional groups of biochars measured by FTIR and Boehm titration are shown in Fig. 2 and Table 3, respectively. FTIR bands at 3,400 $cm^{-1}$ are assigned to OH stretching of phenol; C-H stretching of aliphatic CHx at 2,800 to 2,400 $cm^{-1}$; C = O carboxylic and ketones at 1,700 $cm^{-1}$; C = C stretching aromatic components and C = O conjugated ketones and quinones at 1,600 $cm^{-1}$; aliphatic C-H bending vibration at 1,420 $cm^{-1}$; C-H stretching at 1,030 $cm^{-1}$, which is associated with undecomposed cellulosic and lignous C (cellulose, hemicelluose, and lignin); and C-H bending aromatic CH out-of-plane deformation at 874 $cm^{-1}$. The peak intensity also reflecting the quantity of surface functional groups is shown in Table 3. The band of carboxylic groups (1,700 $cm^{-1}$) for leucaena and lac tree biochars, for

**TABLE 2.** Mean Concentration of Selected Elements in the Biochars

| | N | P | K | Ca | Mg | Na | Fe | Mn | Zn | Cu | B | Mo | Al | Co | Si |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Biochars** | % | | | | | | mg $kg^{-1}$ | | | | | | | | |
| Leucaena wood | 0.6 | 0.08 | 0.85 | 5.1 | 0.55 | 0.03 | 3894.4 | 214.2 | 18.9 | 16.2 | 26.1 | 0.30 | 3710.8 | 0.04 | 38.1 |
| Lac tree wood 400 | 0.3 | 0.13 | 0.73 | 6.3 | 0.32 | 0.06 | 571.0 | 87.9 | 15.1 | 10.1 | 12.7 | 0.04 | 448.1 | 0.00 | 45.7 |
| Hilo mixed wood | 0.5 | 0.09 | 0.47 | 1.6 | 0.22 | 0.35 | 12259.5 | 153.8 | 13.3 | 20.9 | 12.8 | 0.18 | 9766.9 | 0.06 | 15.7 |
| She oak wood | 0.3 | 0.01 | 0.50 | 1.5 | 0.11 | 0.08 | 284.0 | 41.9 | 5.1 | 5.2 | 8.4 | 0.38 | 129.4 | 0.01 | 58.8 |
| Mahogany wood | 0.2 | 0.01 | 0.17 | 0.5 | 0.12 | 0.01 | 2728.6 | 62.4 | 9.4 | 8.7 | 2.9 | 0.20 | 1779.2 | 0.02 | 28.3 |
| Mountain gum wood | 0.1 | 0.02 | 0.03 | 0.1 | 0.03 | 0.04 | 206.2 | 12.9 | 8.7 | 2.5 | 3.2 | 0.02 | 155.3 | 0.00 | 28.0 |
| Lac tree wood 550 | 0.4 | 0.06 | 0.33 | 3.13 | 0.13 | 0.12 | 684 | 55 | 18 | 6 | 8 | — | — | — | 77.9 |
| Rice husk | 0.8 | 0.1 | 0.48 | 0.17 | 0.09 | 0.03 | 413 | 189 | 94 | 4 | 9 | — | — | — | 353.9 |

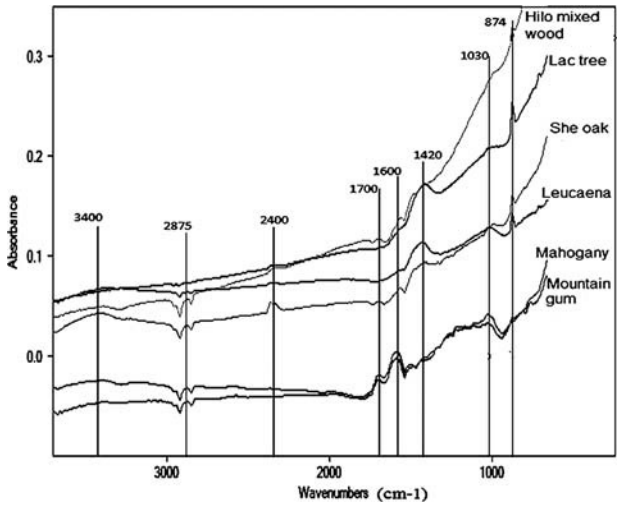


**FIG. 2.** Fourier transform infrared bands characterizing surface functional groups of six biochars.

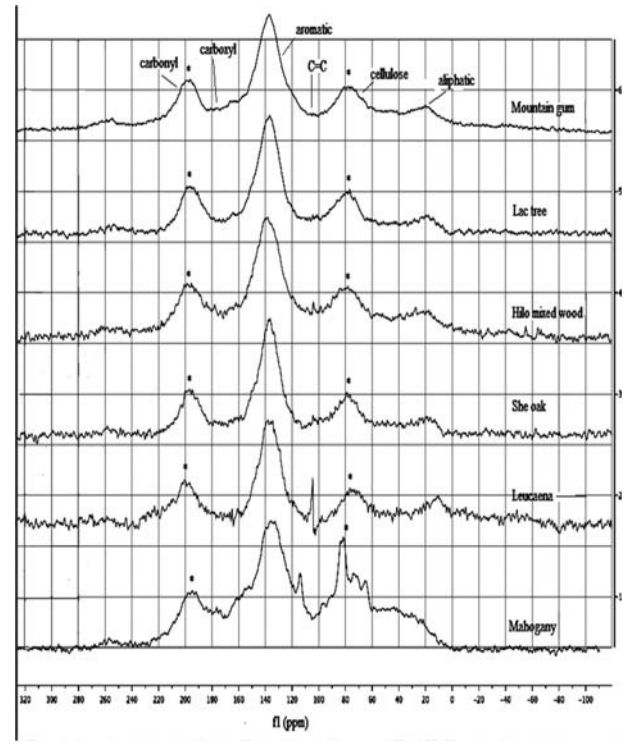


**FIG. 3.** Cross Polarization Angle Spinning NMR peaks of the chemical compounds and functional groups of six biochars. *Spinning side band (SSB). Peaks of six biochars were stacked to show the aromatization resulted from the pyrolysis process of each biochar.

example, nearly disappeared, as shown in Fig. 2. These values are in agreement with those reported by Sharma et al. (2004), Brewer et al. (2009, 2011), Lee et al. (2010), and Kloss et al. (2012). The small quantity of carboxylic groups obtained from Boehm titration for those biochars was consistent with our FTIR results. Quantity of phenolic groups was very high in the mountain gum and mahogany biochars, consistent with the total phenolic content obtained from the Prussian blue assay (Table 1), broad bands FTIR at 1,600 $cm^{-1}$, and Boehm titration (Table 3).

Nuclear magnetic resonance bands from the six biochars had similar patterns (Fig. 3), a typical aromatic band at the center and two symetrical spinning side bands. The broad central band approximately 120 to 160 ppm is aromatic and is the main component of biochar. This was the result of the rearrangement and aromatization of thermochemically degraded cellulose, hemicellulose, and lignin during pyrolysis (Amonette and Joseph, 2009; Brewer, 2012; Keiluweit et al., 2010). The presence of the aromatic compound is supported by the FTIR peak at 1,600 $cm^{-1}$ and the low values of biochar H:C ratio. On the left side, there are aldehyde (190–200 ppm) and a small band of carboxylic (160–170 ppm), whereas CH-O, CH-N, CH-X (70–90 ppm) and other aliphatic (10–30 ppm) bands are on the right.

Scanning electron microscope graphs show the porous nature of biochars (Fig. 4A). The ranges of pore diameter for leucaena, Hilo mixed wood, she oak, mahogany, lac tree 450, and mountain gum biochars are 2.25 to 27.60 μm, 2.26 to 15.90 μm, 4.28 to 12.60 μm, 2.88 to 15.50 μm, 1.69 to 12.60 μm, and 2.24 to 6.92 μm, respectively. These pores (> 50 nm) could hold water and provide habitat for microbiota. Porosity developed from the rearrangement of fused-ring carbons during the heating process. The aggregated fused-ring carbons are stacked to form small lamellar crystallites, then the crystallites were randomly orientated, leaving voids between them (Rutherford et al., 2004). Scanning electron microscope or electron microprobe analysis (EMPA) can be combined with EDS analysis for detailed study of the chemical composition of a sample. An example of biochar chemical composition obtained from EDS analysis following the EMPA records is shown in Fig. 4B. The EDS analysis on two spots of white crystalline material from the EMPA image showed the typical peaks of C, O, and Ca. Thus, the white crystalline material was likely CaO or $CaCO_3$. This result is in line with an early report by Chia et al. (2010), who used SEM and EDS to analyze a synthetic Terra Preta soil.

**TABLE 3.** Mean and S.E. of Functional Groups on Six Biochars Obtained by the Boehm Titration

| | Total | Carboxylic | Phenolic | Lactonic |
|---|---|---|---|---|
| **Biochars** | | **mmol $g^{-1}$** | | |
| Leucaena wood | 0.38 ± 0.01 | 0.07 ± 0.02 | 0.21 ± 0.04 | 0.10 ± 0.03 |
| Lac tree wood 400 | 0.38 ± 0.02 | 0.12 ± 0.02 | 0.20 ± 0.04 | 0.07 ± 0.02 |
| Hilo mixed wood | 0.58 ± 0.03 | 0.22 ± 0.02 | 0.27 ± 0.03 | 0.10 ± 0.03 |
| She oak wood | 0.43 ± 0.01 | 0.24 ± 0.01 | 0.06 ± 0.01 | 0.13 ± 0.01 |
| Mahogany wood | 2.64 ± 0.03 | 0.31 ± 0.01 | 1.57 ± 0.05 | 0.76 ± 0.03 |
| Mountain gum wood | 2.15 ± 0.03 | 0.55 ± 0.04 | 0.88 ± 0.04 | 0.72 ± 0.03 |

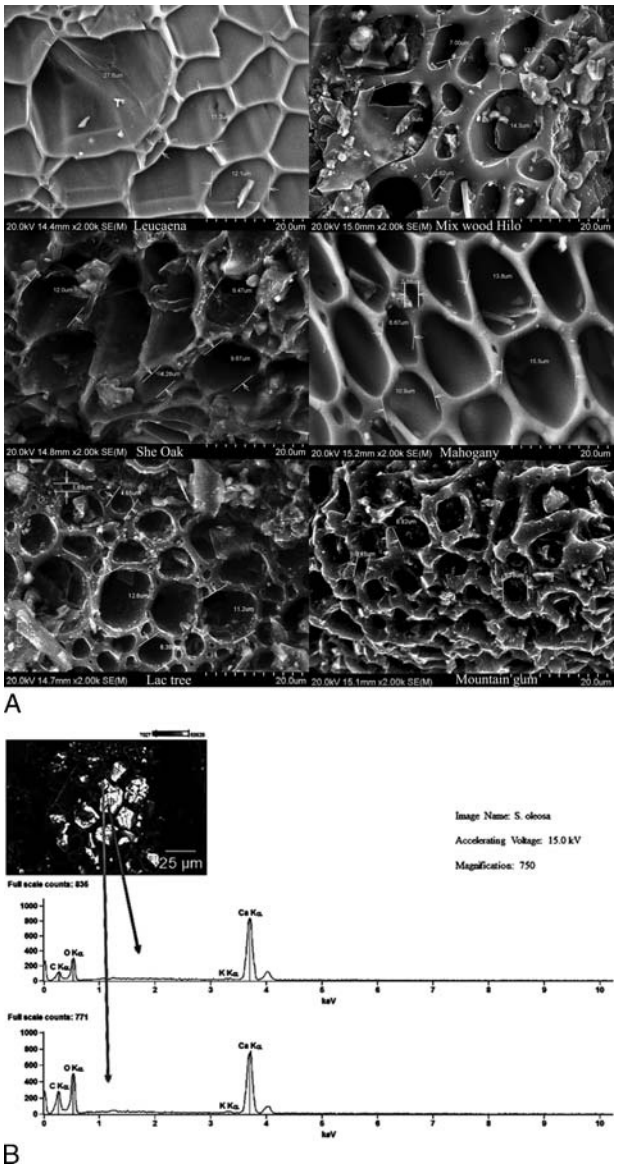


**FIG. 4.** Scanning electron microscope images of the surface structure and porosity of six biochar (A) and EDS analysis of white spots on an electron microprobe image of lac tree 450 biochar (B).

Regarding the two biochars used in field trials, the ash content, CCE, pH, EC, and VM content of the lac tree wood 600 biochar were higher than those of the rice husk biochar (Table 1). Such differences were attributed to both higher pyrolysis temperature and feedstock (Zhao et al., 2013). The CEC of rice husk was higher because it was produced at lower temperature (300°C) than lac tree biochar 600 (produced at 600°C). This could be related to the greater presence of –OH functional groups at 300°C. It is known that the highest treatment temperature greater than 500°C resulted in a loss of carboxylic groups (Harvey et al., 2012). This finding agrees with those of Kloss et al. (2011) and Budai et al. (2014), who reported that CEC decreases with increasing pyrolysis temperature. Also, wood-derived biochar is low in CEC because defragmentation of lignocellulose (cleavage of OH–O-type) H-bonding of wood biochar and the subsequent oxidation to carboxyl are low (Harvey et al., 2012). Plant nutrients such as N, P, K, Mn, and Zn were higher in rice husk biochar than lac tree wood 600 biochar, whereas conversely, Ca, Mg, and Fe were higher in the lac tree wood 600 biochar. The Si content in rice husk biochar was quite high because rice plants require high Si. Being formed at 500 to 600°C, lac tree biochar contained low N because of the volatilization of N at that temperature range (Gaskin et al., 2008; Rajkovich et al., 2012; Wu et al., 2012). However, it contained higher Ca and Mg because such nutrients vaporize only at greater than 1,000°C (Knicker, 2007).

The liming effect is attributed to the biochar ash content or inorganic phase for the short-term effects and its oxygenated functional groups for the long-term effects. Because most biochars are produced at a relatively high temperature, the mineral elements, basic cations in particular, have been transformed into their carbonates or oxides, which are basic as reflected in their CCE. For example, the measured CCE of the lac tree wood 600 biochar was 13.7%; the basic cation contents were 0.33% K, 3.13% Ca, 0.13% Mg, and 0.12% Na. Assuming that these cations were in their oxide forms, then 0.33% K would be equivalent to 0.0423 moles $CaCO_3$ or 0.42% $CaCO_3$ per kg biochar. Similarly, 3.13% Ca, 0.13% Mg and 0.12% Na would be equivalent to 7.8%, 0.54%, and 0.26% $CaCO_3$, respectively. Then the total CCE estimated from the basic cations (8.65%) was close to the measured CCE.

Biochars have their surface functional groups (Table 3 and Fig. 2) that carry negative charge when added to soils. Decarboxylation of these functional groups consumes proton and increases soil pH (Wang et al. 2014). These surface oxygenated functional groups can also complex Al in the soil solution and reduced Al toxicity to plants.

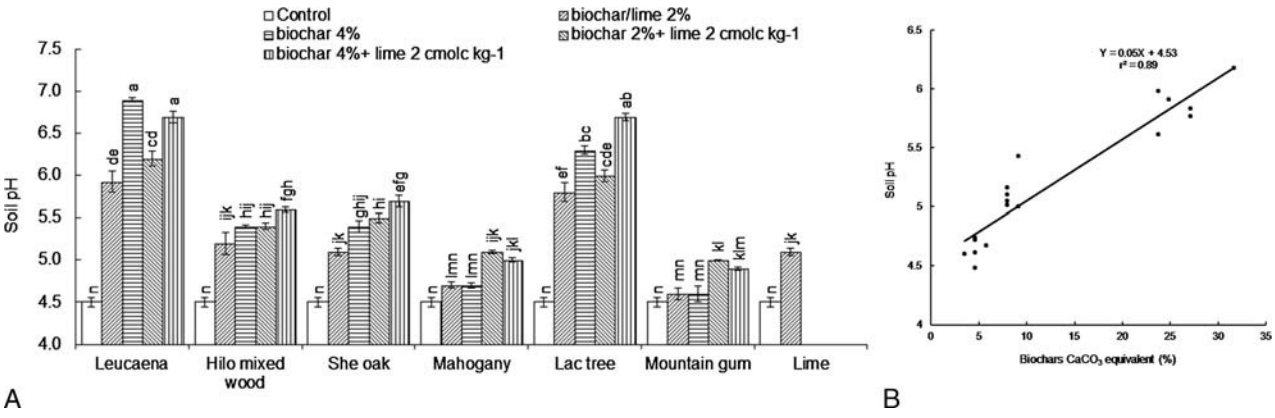


**FIG. 5.** A, Effect of biochars and lime on pH of an acidic soil of Hawaii. B, Correlation between soil pH and biochar CCE.

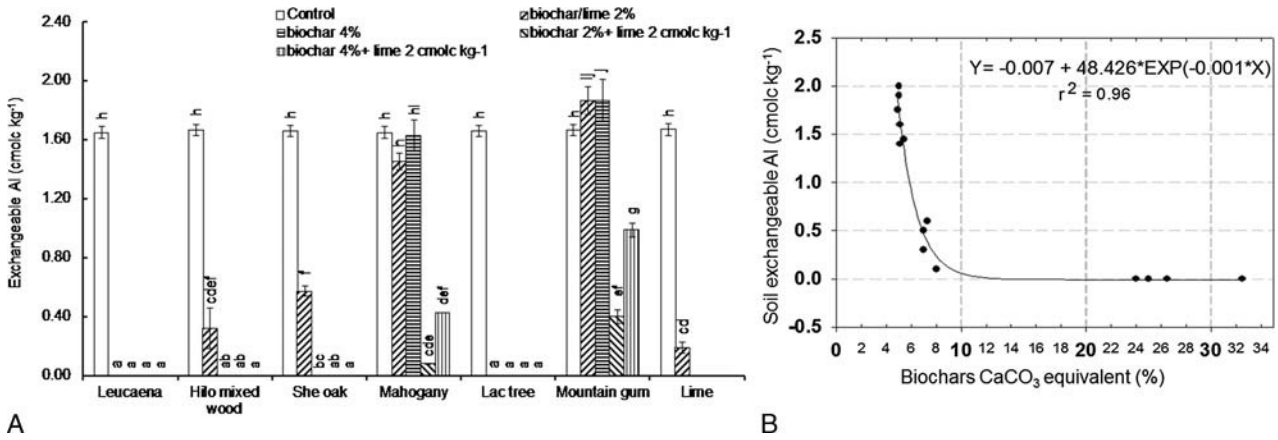


**FIG. 6.** A, Effect of biochars and lime on exchangeable Al of an acidic soil of Hawaii. B, Relationship between biochars CCE and exchangeable Al in an acid Hawaiian soil amended with 2% biochars.

Nutrient content and availability are the other beneficial effect (beside the liming effect) of biochars used as soil amendments. For example, the rice husk biochar produced at 300°C contained more N than the lac tree wood 600 biochar produced at 500°C to 600°C. In contrast, the lac tree 600 produced at higher temperature contained more Ca than the rice husk biochar. This

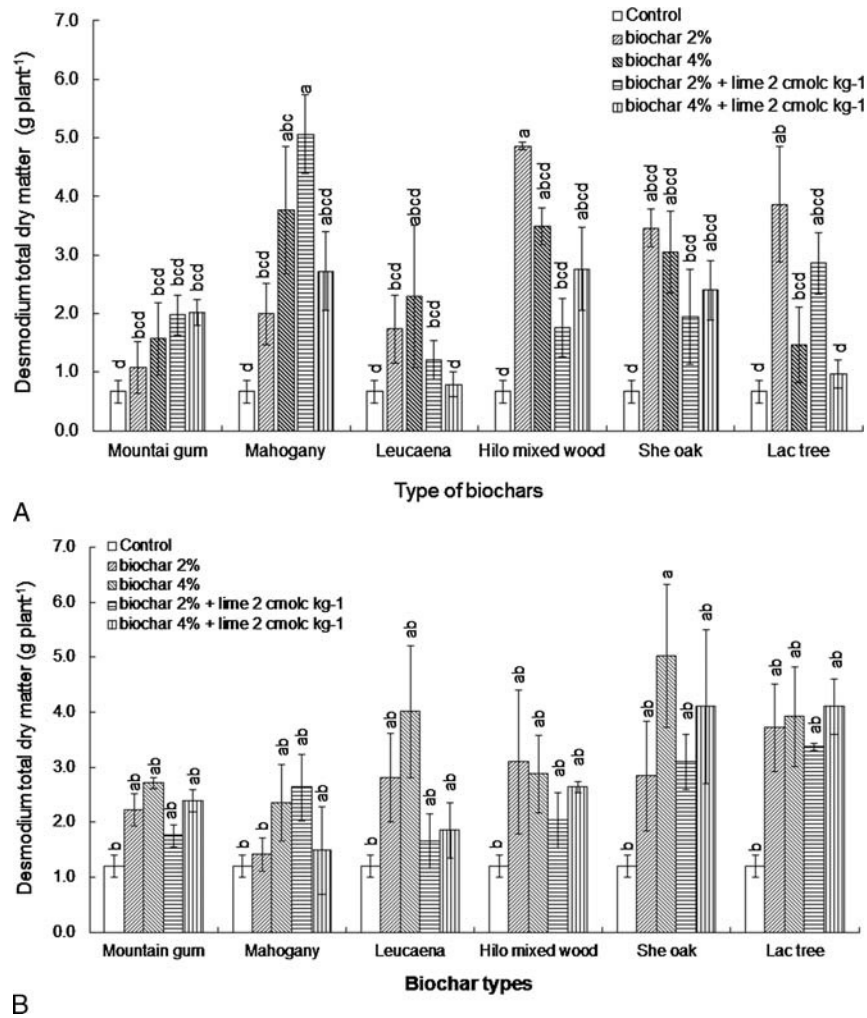


**FIG. 7.** *Desmodium intortum* dry matter as affected by biochars and lime: (A) first planting and (B) second planting.

finding agrees with Keiluweit et al. (2010) and Kim et al. (2012), who found that some nutrients were volatilized at the high pyrolysis temperature.

## Greenhouse Trial

### *Effect of Biochars on Soil Properties*

Additions of biochars at 2% and 4% alone or in combination with 2 cmolc $kg^{-1}$ of lime raised soil pH to different values, depending on the type and proportion of biochars (Fig. 5A). Lac tree and leucaena biochars increased soil pH from 4.5 to 5.8 and 5.9 when applied at 2%, and increased the soil pH further to 6.3 and 6.9, respectively, at 4%. However, the extent of pH increase was lowered when applied in combination with lime, perhaps because of the overliming effect. Hilo mixed wood and she oak biochars raised the soil pH moderately to 5.1 and 5.4 when applied at 2% and 4% alone and to 5.6 to 5.7 when applied in combination with lime. Mahogany and moutain gum biochars increased soil pH only slightly to 5.0 when applied with lime. The magnitude of soil pH increases correlates well with the biochar CCE ($r^2 = 0.89$) (Fig. 5B). This finding agrees with those of Chintala et al. (2013), Butterly et al. (2013), Deenik et al. (2011), Novak et al. (2010), Singh et al. (2010), Tryon (1948), Yamato et al. (2006), Yuan and Xu (2012).

The type and amount of biochars affected soil exchangeable Al differently (Fig. 6A). Soil exchangeable Al was decreased from 1.8 cmolc $kg^{-1}$ to undetectable by the addition of leucaena and lac tree biochars at 2% and by Hilo mixed wood and she oak biochars at 4%. In contrast, mahogany and mountain gum biochars decreased soil exchangeable Al only when applied in combination with lime. The result is similar to that of Deenik et al. (2011), who reported that the kiawe charcoal was capable of increasing soil pH and reducing exchangeable Al in a Hawaiian Ultisol. Our finding is also consistent with those of Yuan and Xu (2012) in China, Singh et al. (2010) in Australia, and Yamato et al. (2006) in Indonesia.

Biochar CCE is likely responsible for soil pH increases and exchangeable Al reduction (Fig. 5B and Figs. 6A, B). Specifically, with the highest CCE and basic cations, the leucaena and lac tree 450 biochars increased the soil pH the most, followed by moderate pH increase with the Hilo mixed wood and she oak wood biochars, and the least pH increase with mahogany and mountain gum wood biochars. For example, applications of 2% leucaena and lac tree 450 biochars yielded 28.6% and 24.1% CCE, respectively, and 4% Hilo mixed wood and she oak biochars yielded 8% CCE. Thus, $OH^-$ ions released from biochar were likely responsible for soil pH increase and precipitation of "active" Al (Smider and Singh, 2014; Yuan and Xu, 2011; van Zwieten et al., 2010). More specifically, addition of 2% leucaena wood biochar reduced the exchangeable Al of the Hawaiian Ultisol from 1.8 cmolc $kg^{-1}$ to undetectable, caused by 28.6% $CaCO_3$ of this biochar. Applied at 2%, it required 28.60 g leucaena biochar for 1 kg of soil; 28.60 g leucaena biochar could contain 5.72 g $CaCO_3$. If 100 g $CaCO_3$ can produce 100 to 200 cmol of $OH^-$, then 5.72 g $CaCO_3$ could produce 5.72 to 11.44 cmol $OH^-$, more than the $OH^-$ that would be required to neutralize 1.8 cmolc exchangeable Al per kg soil. This

**TABLE 4A.** Mean Concentration of Selected Elements in the Plant Tissues (Shoot) of *D. intortum*

| | P | K | Ca | Mg | Na | Mn | Fe | Zn | Al |
|---|---|---|---|---|---|---|---|---|---|
| **Biochars and Lime Rates** | | | g $kg^{-1}$ | | | | mg $kg^{-1}$ | | |
| Leucaena wood 2% | 2.2 | 25.3 | 3.3 | 5.4 | 3.6 | 5.3 | 599.8 | 16.9 | 397.3 |
| Leucaena wood 4% | 1.9 | 26.2 | 3.8 | 6.4 | 3.6 | 5.7 | 941.6 | 12.1 | 651.5 |
| Leucaena wood 2% with lime 2 cmolc $kg^{-1}$ | 2.0 | 24.1 | 3.6 | 5.4 | 3.3 | 3.5 | 755.4 | 13.2 | 506.9 |
| Leucaena wood 4% with lime 2 cmolc $kg^{-1}$ | 1.8 | 30.5 | 4.6 | 6.7 | 3.4 | 3.2 | 674.8 | 10.5 | 457.4 |
| Lac tree wood 2% | 1.4 | 19.6 | 2.8 | 4.5 | 2.8 | 4.6 | 734.5 | 6.2 | 487.5 |
| Lac tree wood 4% | 1.5 | 24.8 | 3.6 | 4.8 | 3.8 | 2.6 | 889.0 | 14.3 | 616.3 |
| Lac tree wood 2% with lime 2 cmolc $kg^{-1}$ | 2.1 | 23.7 | 3.8 | 5.6 | 3.2 | 4.7 | 822.6 | 13.5 | 506.8 |
| Lac tree wood 4% with lime 2 cmolc $kg^{-1}$ | 1.6 | 23.6 | 3.7 | 5.9 | 3.4 | 2.5 | 642.1 | 9.2 | 426.5 |
| Hilo mixed wood 2% | 1.9 | 25.9 | 2.9 | 5.1 | 4.3 | 9.6 | 939.8 | 12.8 | 704.8 |
| Hilo mixed wood 4% | 2.1 | 22.8 | 3.4 | 5.9 | 4.2 | 10.8 | 1144.7 | 10.8 | 788.3 |
| Hilo mixed wood 2% with lime 2 cmolc $kg^{-1}$ | 1.7 | 19.6 | 7.1 | 4.4 | 4.6 | 5.7 | 1145.8 | 11.4 | 918.7 |
| Hilo mixed wood 4% with lime 2 cmolc $kg^{-1}$ | 1.7 | 16.0 | 3.5 | 3.7 | 3.3 | 5.2 | 918.0 | 9.2 | 614.3 |
| She oak wood 2% | 1.8 | 25.2 | 3.2 | 3.8 | 3.9 | 6.2 | 1093.0 | 12.6 | 789.8 |
| She oak wood 4% | 2.1 | 21.0 | 3.1 | 4.0 | 3.5 | 5.9 | 833.9 | 9.1 | 595.9 |
| She oak wood 2% with lime 2 cmolc $kg^{-1}$ | 1.8 | 14.8 | 3.5 | 4.7 | 2.7 | 6.0 | 1326.1 | 11.5 | 990.5 |
| She oak wood 4% with lime 2 cmolc $kg^{-1}$ | 2.1 | 19.5 | 3.4 | 4.7 | 3.2 | 6.2 | 1221.9 | 15.7 | 855.1 |
| Mahogany wood 2% | 2.1 | 23.5 | 2.7 | 4.3 | 4.4 | 11.6 | 694.7 | 8.9 | 506.1 |
| Mahogany wood 4% | 1.9 | 22.4 | 2.5 | 4.5 | 4.2 | 13.6 | 571.2 | 10.1 | 421.4 |
| Mahogany wood 2% with lime 2 cmolc $kg^{-1}$ | 1.8 | 22.6 | 2.7 | 5.6 | 3.2 | 10.2 | 756.1 | 6.0 | 536.9 |
| Mahogany wood 4% with lime 2 cmolc $kg^{-1}$ | 2.0 | 23.8 | 2.8 | 5.1 | 4.0 | 7.0 | 648.0 | 13.5 | 460.9 |
| Mountain gum wood 2% | 1.7 | 19.0 | 2.3 | 3.3 | 4.4 | 11.4 | 664.3 | 11.2 | 490.8 |
| Mountain gum wood 4% | 2.3 | 19.9 | 2.5 | 4.7 | 4.4 | 12.7 | 743.3 | 8.1 | 549.3 |
| Mountain gum wood 2% with lime 2 cmolc $kg^{-1}$ | 1.7 | 26.1 | 3.2 | 4.1 | 3.8 | 8.7 | 594.0 | 9.9 | 423.5 |
| Mountain gum wood 4% with lime 2 cmolc $kg^{-1}$ | 2.0 | 19.6 | 2.9 | 3.6 | 3.6 | 9.3 | 558.4 | 13.5 | 412.9 |
| Lime 2 cmolc $kg^{-1}$ | 1.5 | 20.6 | 2.5 | 3.8 | 3.1 | 9.7 | 571.4 | 7.2 | 420.7 |
| Control | 1.7 | 26.2 | 2.7 | 6.9 | 3.6 | 6.8 | 640.1 | 6.5 | 433.5 |

finding is in line with works by Joseph et al. (2010) and Yuan and Xu (2012). Surface adsorption and coprecipitation (as $KAlSi_3O_8$) of Al onto silicate particles at the biochar surface were another possible (although minor, in our opinion) mechanism proposed by Qian et al. (2013) and Qian and Chen (2013).

Incoporations of biochars into soil increased soil CEC to different values, depending on the type and proportion of biochars. Leucaena, Hilo mixed wood, and lac tree 450 biochars increased soil CEC from 16.8 cmolc $kg^{-1}$ to 21.6, 20.7, and 25.2 cmolc $kg^{-1}$, respectively, when applied at 2%. At this rate and in combination with lime, leucaena biochar increased soil CEC to 26.0 cmolc $kg^{-1}$. Increases in soil CEC could be attributed to the negative charge of surface functional groups, especially carboxylates (Boehm, 1994; Chan and Xu, 2009; Glaser et al., 2001; Liang et al., 2006; Yuan and Xu, 2011). Additions of she oak, mahogany, or mountain gum biochars have no significant effect on soil CEC, perhaps because of low soil pH in those treatments. Similar observations were reported by Yuan and Xu (2012) for an Ultisol soil from Hainan, China; by Novak et al. (2009) for a southeastern coastal plain soil, United States; and by Steiner et al. (2007) for a highly weathered central Amazonian upland soil.

### *Effect of Biochars on Plant Growth*

*Desmodium* growth expressed as total dry weight increased with biochar additions (Figs. 7A, B). The highest growth in the first planting was obtained from the application of Hilo mixed wood, lac tree 450, and she oak at 2% and mahogany biochars at 2% with lime. However, plant dry matter was lowered in the second planting, particularly in the mahogany biochar. In the second planting, the highest growth was obtained from the application of leucaena, lac tree 450 and she oak biochars at 4%. Such growth enhancement could be attributed to the lowering of Al toxicity (Figs. 6A, B), increases in soil pH (Figs. 5A, B), soil CEC (data not shown), and nutrients (Tables 4A and B) upon the incorporation of biochars. With high basic cations and CCE, leucaena, lac tree 450, Hilo mixed wood, and she oak biochars improved productivity of the Hawaiian acid Ultisol and subsequently enhanced *D. intortum* growth more than the mahogany and mountain gum biochars. The combination of biochars and lime lowered plant growth in the second planting for most biochars tested, perhaps because of overliming.

## Field Trials

### *Effect of Biochars on Soil Properties*

Soil pH increases differed by treatments and experimental sites (Fig. 8), by approximately 0.5 to 1.1 units at Jasinga and 0.3 to 2.0 units at Guradog. At Guradog site, soil pH was raised from 4.0 to 5.1 and 6.0 by addition of lac tree wood 600 biochar at 8% alone and in combination with lime at 8 cmolc $kg^{-1}$ and compost at 0.2%, respectively. In contrast, rice husk biochar at the same rate increased the soil pH only from 4.0 to 4.7 and 5.1, respectively. At the Jasinga site, soil pH increased only moderately from 3.9 to 4.7 and 5.0 by addition of lac tree wood 600 biochar at 8% alone and in combination with lime 8 cmolc $kg^{-1}$ and compost 0.2%, respectively. Rice husk biochar in the same proportion increased soil pH only from 4.0 to 4.4 and 4.7,

**TABLE 4B.** Mean Concentration of Selected Elements in the Plant Tissues (Root) of *D. intortum*

| | P | K | Ca | Mg | Na | Mn | Fe | Zn | Al |
|---|---|---|---|---|---|---|---|---|---|
| **Biochars and Lime Rates** | | | **g $kg^{-1}$** | | | | **mg $kg^{-1}$** | | |
| Leucaena wood 2% | 1.9 | 24.6 | 17.0 | 3.1 | 1.3 | 83.3 | 212.9 | 52.3 | 111.7 |
| Leucaena wood 4% | 1.6 | 23.5 | 19.3 | 3.0 | 1.4 | 49.5 | 231.4 | 23.9 | 118.5 |
| Leucaena wood 2% with lime 2 cmolc $kg^{-1}$ | 2.0 | 24.7 | 19.8 | 3.3 | 1.4 | 67.1 | 201.3 | 34.7 | 123.0 |
| Leucaena wood 4% with lime 2 cmolc $kg^{-1}$ | 1.9 | 25.0 | 20.9 | 3.7 | 1.2 | 59.1 | 395.2 | 25.1 | 405.7 |
| Lac tree wood 2% | 1.8 | 25.1 | 17.5 | 3.2 | 1.2 | 110.9 | 154.0 | 47.0 | 68.7 |
| Lac tree wood 4% | 1.8 | 24.8 | 16.3 | 3.7 | 1.5 | 55.7 | 348.0 | 39.5 | 196.2 |
| Lac tree wood 2% with lime 2 cmolc $kg^{-1}$ | 2.0 | 24.6 | 19.1 | 3.5 | 1.2 | 96.0 | 192.4 | 50.2 | 250.8 |
| Lac tree wood 4% with lime 2 cmolc $kg^{-1}$ | 2.0 | 23.5 | 19.7 | 3.4 | 1.4 | 58.1 | 201.4 | 37.6 | 144.1 |
| Hilo mixed wood 2% | 1.8 | 22.4 | 11.8 | 4.2 | 1.5 | 170.9 | 351.0 | 49.3 | 224.4 |
| Hilo mixed wood 4% | 2.4 | 24.6 | 16.1 | 4.5 | 1.4 | 174.3 | 361.3 | 42.8 | 189.9 |
| Hilo mixed wood 2% with lime 2 cmolc $kg^{-1}$ | 2.0 | 24.7 | 15.5 | 4.1 | 1.5 | 132.0 | 289.2 | 43.9 | 171.3 |
| Hilo mixed wood 4% with lime 2 cmolc $kg^{-1}$ | 2.4 | 26.3 | 18.0 | 3.9 | 1.5 | 163.1 | 253.8 | 90.4 | 223.5 |
| She oak wood 2% | 2.1 | 25.8 | 9.8 | 4.0 | 1.7 | 131.4 | 190.7 | 52.6 | 157.0 |
| She oak wood 4% | 2.2 | 26.3 | 13.3 | 3.9 | 1.5 | 134.6 | 393.9 | 57.3 | 242.4 |
| She oak wood 2% with lime 2 cmolc $kg^{-1}$ | 2.0 | 26.3 | 17.3 | 4.0 | 1.6 | 137.9 | 343.6 | 59.2 | 237.5 |
| She oak wood 4% with lime 2 cmolc $kg^{-1}$ | 2.3 | 26.0 | 17.5 | 3.6 | 1.4 | 128.9 | 189.1 | 52.1 | 74.6 |
| Mahogany wood 2% | 1.8 | 26.1 | 7.7 | 5.4 | 1.5 | 173.6 | 301.4 | 44.0 | 221.0 |
| Mahogany wood 4% | 2.0 | 25.1 | 8.5 | 4.9 | 1.2 | 215.6 | 225.2 | 86.9 | 150.8 |
| Mahogany wood 2% with lime 2 cmolc $kg^{-1}$ | 1.9 | 23.1 | 12.8 | 4.2 | 1.5 | 170.9 | 184.3 | 110.0 | 101.4 |
| Mahogany wood 4% with lime 2 cmolc $kg^{-1}$ | 2.2 | 25.1 | 14.8 | 4.5 | 1.4 | 173.7 | 289.3 | 59.4 | 186.6 |
| Mountain gum wood 2% | 2.3 | 25.7 | 8.4 | 6.5 | 1.4 | 212.5 | 194.9 | 66.8 | 120.7 |
| Mountain gum wood 4% | 2.3 | 26.5 | 8.2 | 6.4 | 1.4 | 156.6 | 237.2 | 43.8 | 138.8 |
| Mountain gum wood 2% with lime 2 cmolc $kg^{-1}$ | 2.0 | 24.7 | 12.7 | 4.3 | 1.2 | 187.8 | 427.2 | 64.9 | 301.7 |
| Mountain gum wood 4% with lime 2 cmolc $kg^{-1}$ | 1.9 | 21.2 | 9.8 | 4.2 | 1.2 | 162.7 | 231.9 | 51.5 | 161.5 |
| Lime 2 cmolc $kg^{-1}$ | 1.9 | 25.7 | 9.3 | 5.8 | 1.7 | 164.9 | 256.4 | 52.5 | 171.9 |
| Control | 2.1 | 23.1 | 12.0 | 4.8 | 1.7 | 155.4 | 478.3 | 41.3 | 320.4 |

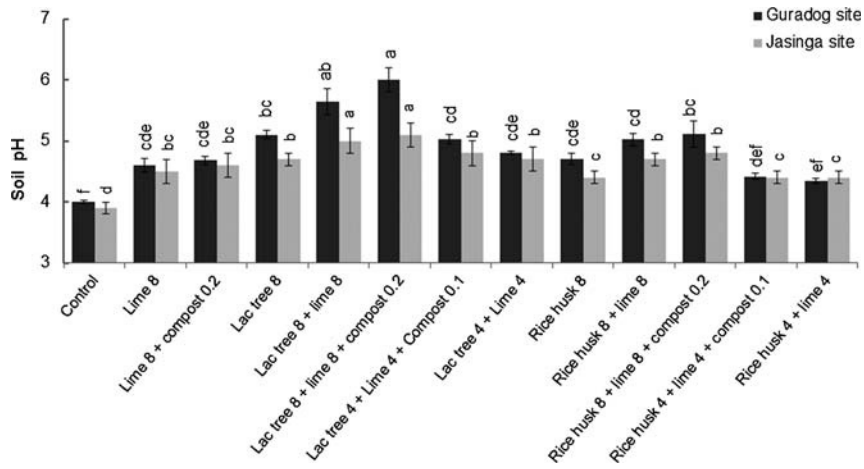


**FIG. 8.** Effect of biochars, lime, and compost on pH of acidic soils (sites) of Indonesia. Units for biochars, lime, and compost were %, cmolc $kg^{-1}$, and %, respectively. Means followed by the same letter(s) were not significantly different by Tukey test at α = 5%. The mean comparison is valid only for each site.

respectively. Those differences could be attributed to the very high exchangeable Al of the Jasinga soil and the low CCE of the rice husk biochar.

Exchangeable Al was reduced by the addition of lac tree wood 600 and rice husk biochars at both sites (Fig. 9). With 13.7% CCE, lac tree wood 600 biochar markedly reduced soil exchangeable Al more than rice husk biochar (1.2% CCE), although their effects were not significantly different at Guradog site ($P > 0.05$). For example, additions of rice husk and lac tree wood 600 biochars at 8% alone decreased exchangeable Al of Guradog soil from 8 cmolc $kg^{-1}$ to 2 and 1 cmolc $kg^{-1}$, respectively, and further decreased exchangeable Al to 1 and undetected, respectively, when applied in combination with lime at 8 cmolc $kg^{-1}$ and compost at 0.2%. Approximately 6 to 7 cmolc $kg^{-1}$ exchangeable Al was reduced by the addition of 8% lac tree wood 600 or rice husk biochar, likely as a result of the biochars' CCE. This finding was in line with the works of van Zwieten et al. (2010) on an Australian Ferrosol, Deenik et al. (2011) on a Hawaiian Ultisol, Chintala et al. (2013) on an acidic Entisol (Grummit soil series), and Yuan and Xu (2012) on Ultisols and Oxisols in China. Complexing Al by insoluble oxidized organic functional groups, particularly carboxylics and phenolics at the surface of biochar, would be another mechanism that could explain the capacity of rice husk biochar to reduce exchangeable Al. Specifically, rice husk biochar at 8% alone reduced approximately 6 cmolc $kg^{-1}$ of exchangeable Al of Guradog soil. This Al reduction could not be explained by the 1.2% CCE of rice husk biochar alone because the maximum OH produced from the carbonates was only 4 moles. At least an additional 2 moles $OH^-$ is needed, which could come from oxygenated functional groups of the rice husk biochar. Similar finding was reported by Yuan et al. (2011), who found that crop residue biochars produced at 300°C contributed to a greater extent of the alkalinity than those made at higher temperatures.

Soil CEC was slightly increased upon addition of lac tree wood 600 and rice husk biochars (Table 5). The highest CEC was obtained from the addition of rice husk biochar at 8% in combination with lime and compost. This could be attributed to the higher CEC of rice husk biochar. Low temperature biochar is more easily oxidized, and compost addition supplies organic matter to the soil, then both materials contribute to development of surface oxidized functional groups, the source of negative charge. This would explain why the addition of the rice husk biochar (produced at 300°C) increased CEC of the Indonesian acid soils more than lac tree wood 600 biochar (500–600°C). Similar results were reported by Kloss et al. (2012) and Budai et al. (2014), showing that CEC decreases with increasing pyrolysis temperature. Also,

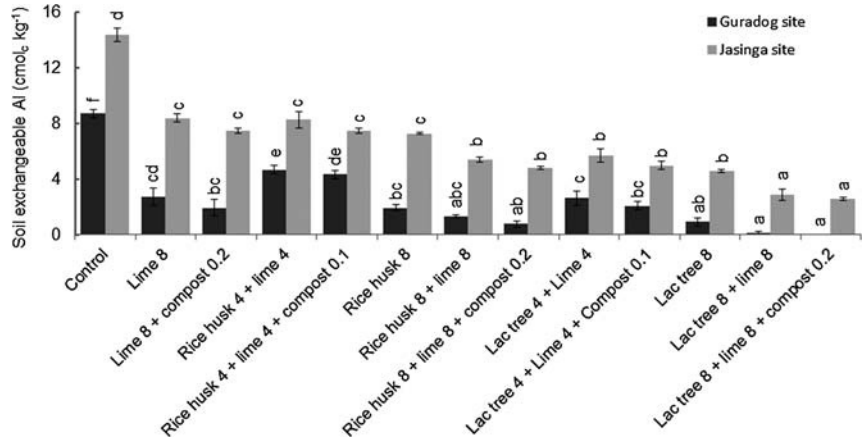


**FIG. 9.** Effect of biochars, lime, and compost on exchangeable Al of acidic soils (sites) of Indonesia. Units for biochars, lime, and compost were %, cmolc $kg^{-1}$, and %, respectively. Means followed by the same letter(s) were not significantly different by Tukey test at α = 5%. The mean comparison is applicable only to each site.

**TABLE 5.** Means and S.E. of Soil CEC as Affected By Biochars, Lime, and Compost Additions (n = 4)

| | Soil CEC (cmolc kg$^{-1}$) | |
|---|---|---|
| **Treatments** | **Guradog** | **Jasinga** |
| Control | 36.34 ± 0.34 d | 33.19 ± 0.56 f |
| Lime 8 | 37.35 ± 0.45 cd | 36.68 ± 0.23 d |
| Lime 8 + compost 0.2 | 37.80 ± 0.23 bcd | 37.46 ± 0.34 bcd |
| Lac tree 8 | 37.46 ± 0.34 cd | 34.43 ± 0.45 ef |
| Lac tree 8 + lime 8 | 39.04 ± 0.34 abc | 38.81 ± 0.34 abc |
| Lac tree 8 + lime 8 + compost 0.2 | 39.83 ± 0.23 ab | 39.04 ± 0.56 ab |
| Lac tree 4 + lime 4 + compost 0.1 | 37.91 ± 0.34 bcd | 37.35 ± 0.45 bcd |
| Lac tree 4 + lime 4 | 37.46 ± 0.56 cd | 36.79 ± 0.34 cd |
| Rice husk 8 | 38.36 ± 0.11 abcd | 36.34 ± 0.11 de |
| Rice husk 8 + lime 8 | 39.15 ± 0.45 abc | 38.93 ± 0.23 ab |
| Rice husk 8 + lime 8 + compost 0.2 | 40.28 ± 0.23 a | 39.83 ± 0.45 a |
| Rice husk 4 + lime 4 + compost 0.1 | 38.59 ± 0.11 abc | 37.69 ± 0.34 bcd |
| Rice husk 4 + lime 4 | 37.69 ± 0.79 bcd | 37.35 ± 0.23 bcd |

Units for biochars, lime, and compost are %, cmolc kg$^{-1}$, and %, respectively.

wood biochar is often low in CEC because of defragmentation of lignocellulose (cleavage of OH–O-type) H-bonding of wood biochar, and the subsequent oxidation to carboxyl is low (Harvey et al., 2012).

### *Effect of Biochar on Soybean Growth*

Soybean shoot and root dry weights were significantly increased upon biochar additions at both experimental sites (Figs. 10A, B; Table 6). Relative to the control, addition of lac tree wood 600 and rice husk biochars at 8% to the Guradog soil increased soybean shoot and root dry matter 206% and 167%, and 169% and 157%, respectively, in the first planting, and further increased to 248% and 208%, and 220% and 203% when applied in combination with lime and compost. Such growth increases could be attributed to the decrease in soil exchangeable Al (Figs. 11A, B) and plant Mn (Fig. 12A), nutrient enhancement such as K (Fig. 12B), and other indirect effects, such as increased soil pH, CEC (Table 5), and unmeasured factors, such as microbial activity that provided favorable conditions for growth (Graber et al., 2010). In the second planting, shoot dry weights were approximately 20% higher than in the first planting, but there was no increase in root dry weights. The increased soybean growth in the second planting suggested additional, and perhaps

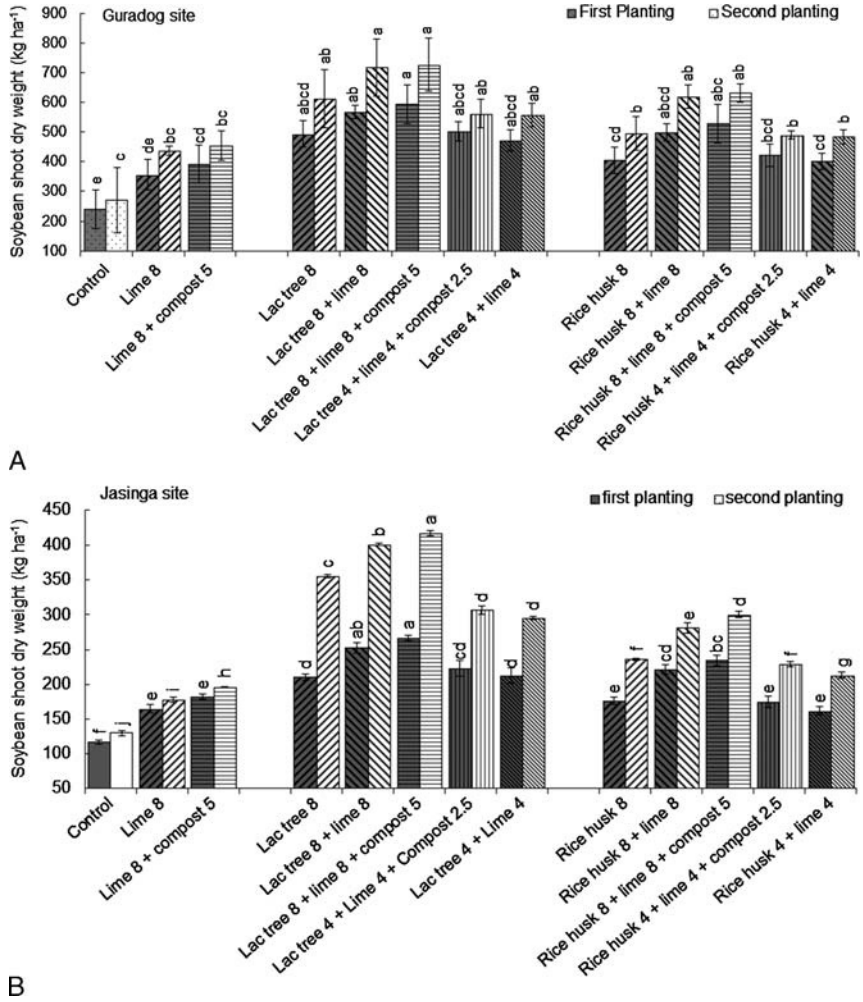


**FIG. 10.** Soybean shoot dry matter as affected by biochars, lime, and compost. Units for biochars, lime, and compost were %, cmolc kg$^{-1}$, and %, respectively: (A) Guradog site and (B) Jasinga site. Means followed by the same letter(s) were not significantly different by Tukey test at α = 5%. The mean comparison is applicable only to each planting.

**TABLE 6.** Means and S.E. of Soybean Root Dry Matters as Affected By Biochar, Lime, and Compost Amendments (n = 4)

| | Guradog | | Jasinga | |
|---|---|---|---|---|
| | First Planting | Second Planting | First Planting | Second Planting |
| Treatments | kg ha$^{-1}$ | | | |
| Control | 55.2±10.8 b | 59.9±16.8 b | 38.5±0.9 g | 43.6±1.3 f |
| Lime 8 | 89.3±9.7 ab | 91.1±9.8 ab | 48.7±0.8 fg | 56.5±2.1 e |
| Lime 8 + compost 0.2 | 93.5±14.4 ab | 95.8±13.8 ab | 51.4±2.3 ef | 58.7±1.0 e |
| Rice husk 4 + lime 4 | 92.2±7.1 ab | 101.9±6.5 ab | 60.2±2.2 cdef | 72.8±2.5 d |
| Rice husk 4 + lime 4 + compost 0.1 | 101.8±10.9 ab | 97.3±5.9 ab | 61.1±3.7 cde | 85.9±2.8 c |
| Rice husk 8 | 86.6±8.7 ab | 88.9±8.5 ab | 52.8±3.4 def | 57.7±1.1 e |
| Rice husk 8 + lime 8 | 117.4±3.6 a | 102.9±2.5 ab | 91.7±4.0 a | 101.1±2.9 b |
| Rice husk 8 + lime 8 + compost 0.2 | 124.0±11.9 a | 114.7±4.7 a | 97.4±0.7 a | 112.9±1.0 a |
| Lac tree wood 4 + lime 4 | 100.4±7.4 ab | 106.4±6.4 ab | 63.9±0.9 cd | 69.1±1.5 d |
| Lac tree wood 4 + lime 4 + compost 0.1 | 105.7±6.5 ab | 102.3±7.1 ab | 64.9±2.2 c | 90.0±1.6 c |
| Lac tree wood 8 | 92.2±14.4 ab | 95.8±12.7 ab | 53.5±1.8 cdef | 59.2±1.1 e |
| Lac tree wood 8 + lime 8 | 103.4±4.9 ab | 110.2±6.5 a | 76.9±1.3 b | 88.2±1.2 c |
| Lac tree wood 8 + lime 8 + compost 0.2 | 114.9±14.5 a | 116.4±14.4 a | 79.6±2.3 b | 110.7±2.4 a |

Units for biochars, lime, and compost were %, cmolc kg$^{-1}$, and %, respectively. Means within column followed by the same letter were not significantly different by Tukey test at $\alpha = 5\%$.

delayed, effects (e.g., aging effect) of biochars, such as improving soil nutrient and water retention (Novak et al., 2009) and stimulating microbial activities (Graber et al., 2010), which were not measured in this experiment.

The best soybean growths expressed in shoot dry weights were 593.6 and 726.2 kg ha$^{-1}$ for the first and second plantings, respectively. These were obtained from the application of lac tree wood 600 biochar at 8% in combination with lime at 8 cmolc kg$^{-1}$ and compost 0.2%. However, the effect of lac tree wood 600 biochar was not significantly different from that of rice husk biochar on soybean shoot dry matter, in both plantings. The result could be explained by the fact that although the lac tree wood biochar had a higher liming potential than the rice husk biochar, the latter contained more easily degradable fractions because of its lower temperature treatment.

Also, the soybean shoot dry matter affected by biochars at 8% in combination with lime and compost was not statistically different from the effect of biochars added alone. This suggests that either lac tree wood 600 or rice husk biochar at 8% can be applied alone for soybean growth in the Guradog soil, depending on the availability of biochars and the local situations based on a benefit-cost analysis (Table S1, Supplemental Digital Content 1, http://links.lww.com/SS/A42). Although soybean growth in biochar-amended soil at Guradog was higher with the lac tree wood 600 biochar than the rice husk biochar, the former and its feedstock were not locally available, and its cost was nearly twice as high. In contrast, the latter was locally available and cheaper; making its use more profitable than the other biochar (Table S1, Supplemental Digital Content 1, http://links.lww.com/SS/A42).

Soybean growth expressed as shoot dry matter at the Jasinga site that received the same proportion of biochars either alone or in combination with lime and compost, also increased, but it was approximately 50% lower than growth observed at the Guradog site (Fig. 10B). This was probably because of the higher exchangeable Al in the Jasinga soil. It seemed that the liming effect of applied biochars at 8% alone or in combination with lime and compost was not sufficient to correct soil acidity at the Jasinga site. Specifically, soybean shoot and root dry matters increased from 116 and 38 kg ha$^{-1}$ (control) to 176 and 53 kg ha$^{-1}$ and to 210 and 54 kg ha$^{-1}$ upon application of rice husk and lac tree wood 600 biochars alone at 8%, respectively. Shoot and root dry weights were further increased to 234 and 97 kg ha$^{-1}$ and to 267 and 80 kg ha$^{-1}$ by the rice husk and lac tree wood biochars at 8% when applied in

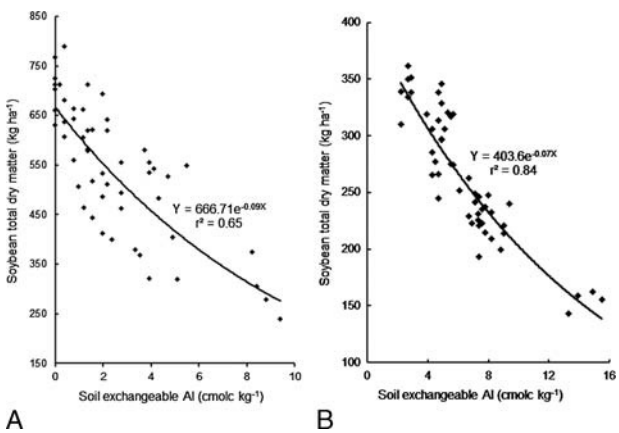


**FIG. 11.** Relationship between total soybean dry matter and soil exchangeable Al at the (A) Guradog site and the (B) Jasinga site.

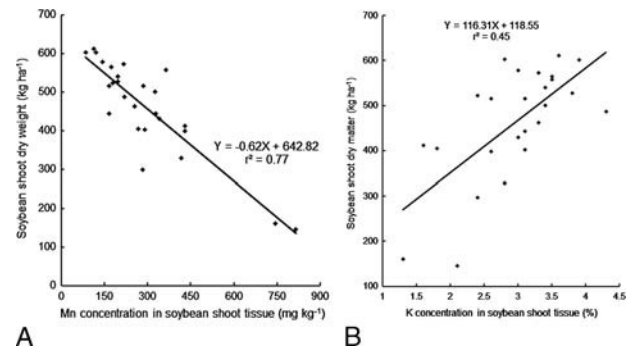


**FIG. 12.** Correlation between soybean shoot dry weight and Mn concentration (A) and K concentration (B) in shoot tissue.

**TABLE 7.** Means and S.E. of Plant Nutrients in Soybean Shoot Tissue First Planting Guradog Soil (n = 2)

| | N | P | K | Ca | Mg | Fe | Mn | Al |
|---|---|---|---|---|---|---|---|---|
| **Treatments** | | | **%** | | | | **mg kg$^{-1}$** | |
| Control | 3.41 ± 0.10 | 0.34 ± 0.05 | 1.71 ± 0.38 | 1.25 ± 0.01 | 0.57 ± 0.00 | 571 ± 81 | 779 ± 36 | 1064 ± 98 |
| Lime 8 | 3.27 ± 0.01 | 0.30 ± 0.02 | 2.12 ± 0.29 | 1.94 ± 0.08 | 0.58 ± 0.00 | 632 ± 77 | 275 ± 8 | 1082 ± 186 |
| Lime 8 + compost 0.2 | 3.03 ± 0.32 | 0.41 ± 0.06 | 2.10 ± 0.54 | 2.29 ± 0.23 | 0.73 ± 0.04 | 457 ± 110 | 429 ± 1 | 796 ± 184 |
| Rice husk 4 + lime 4 | 3.01 ± 0.21 | 0.32 ± 0.03 | 2.71 ± 0.30 | 1.58 ± 0.01 | 0.44 ± 0.06 | 509 ± 51 | 260 ± 81 | 812 ± 87 |
| Rice husk 4 + lime 4 + compost 0.1 | 3.41 ± 0.45 | 0.40 ± 0.02 | 3.13 ± 0.01 | 1.71 ± 0.19 | 0.54 ± 0.01 | 455 ± 9 | 309 ± 19 | 749 ± 33 |
| Rice husk 8 | 3.58 ± 0.27 | 0.36 ± 0.03 | 2.71 ± 0.14 | 1.19 ± 0.03 | 0.45 ± 0.02 | 381 ± 87 | 352 ± 66 | 590 ± 214 |
| Rice husk 8 + lime 8 | 3.09 ± 0.08 | 0.42 ± 0.00 | 3.49 ± 0.04 | 1.98 ± 0.08 | 0.58 ± 0.01 | 832 ± 265 | 344 ± 19 | 969 ± 622 |
| Rice husk 8 + lime 8 + compost 0.2 | 3.26 ± 0.27 | 0.41 ± 0.01 | 3.63 ± 0.19 | 1.54 ± 0.17 | 0.47 ± 0.02 | 659 ± 73 | 198 ± 0.3 | 1249 ± 98 |
| Lac tree wood 4 + lime 4 | 2.99 ± 0.06 | 0.34 ± 0.01 | 3.08 ± 0.02 | 1.57 ± 0.11 | 0.46 ± 0.00 | 682 ± 161 | 166 ± 0.3 | 1119 ± 295 |
| Lac tree wood 4 + lime 4 + compost 0.1 | 3.42 ± 0.03 | 0.39 ± 0.01 | 3.30 ± 0.02 | 1.98 ± 0.01 | 0.56 ± 0.04 | 827 ± 90 | 237 ± 19 | 1400 ± 161 |
| Lac tree wood 8 | 3.30 ± 0.35 | 0.37 ± 0.07 | 3.67 ± 0.64 | 1.62 ± 0.18 | 0.50 ± 0.07 | 426 ± 50 | 182 ± 37 | 665 ± 58 |
| Lac tree wood 8 + lime 8 | 3.51 ± 0.28 | 0.34 ± 0.06 | 3.12 ± 0.33 | 1.68 ± 0.24 | 0.43 ± 0.10 | 455 ± 46 | 130 ± 44 | 701 ± 106 |
| Lac tree wood 8 + lime 8 + compost 0.2 | 3.65 ± 0.05 | 0.39 ± 0.02 | 3.74 ± 0.14 | 1.78 ± 0.08 | 0.50 ± 0.02 | 538 ± 84 | 117 ± 3 | 848 ± 124 |

combination with lime and compost. The highest growth in both plantings was from the treatment receiving 8% lac tree wood biochar + 8 cmolc kg$^{-1}$ lime + 0.2% compost. In contrast to shoot growth, the highest root dry matter in the first planting was after the application of the rice husk biochar at 8% + lime and compost (Table 6). This could be related to easier root penetration into the rice husk biochar pores, which were volumetrically more numerous than the lac tree biochar (bulk density of the rice husk biochar was lower than that of the lac tree wood biochar). Both soil properties and soybean growth in the Jasinga soil were not significantly improved upon applications of the biochars at 8%, either alone or in combination with lime and compost. Thus, further investigations would be necessary before biochar could be recommended for use at the Jasinga site.

The likely reasons for the increased soybean growth in tropical acid soils upon addition of biochars are (1) correction of soil acidity (increasing soil pH, decreasing exchangeable Al and Mn) (Figs. 8, 9, 10A and B, and 12A) that provides better rhizosphere conditions for root development and (2) increasing soil CEC that enhances nutrient retention and regulates nutrient release (Tables 3, 5, and 6) increasing plant nutrients, such as K (Fig. 12B) probably due to better K uptake. Our findings were similar to those of Major et al. (2010), who reported increases in grass, forb, and legume biomass by 93%, 292%, and 1916% over the control 5 months after addition of 23.2 t ha$^{-1}$ of black carbon (biochar) to a Colombian Typic Haplustox (a sandy clay loam soil). Similar results were also reported by Tagoe et al. (2008) in Japan, Suppadit et al. (2012) in Thailand, and Smider and Singh (2014) in Australia. Some contradictory findings were also reported. For example, van Zwieten et al. (2010) added paper mill waste biochar at 10% to a Ferrasol that resulted in an increase in pH from 4.20 to 5.93 and a decrease in exchangeable Al from 2.0 cmolc kg$^{-1}$ to virtually zero, yet there was no effect on soybean growth when the biochar was applied without additional fertilizers.

### *Effect of Biochars on Plant Nutrients*

Addition of the lac tree wood and rice husk biochars increased some nutrients, but decreased other nutrients in soybean tissues (Table 7). For example, K was clearly increased by the addition of the biochars, perhaps because K is the most available nutrient in the biochars. In contrast, Mn was markedly decreased by adding biochars compared with the control. Processes that suppress Mn in soybean tissue grown in biochar-treated soils could be the precipitation of soluble Mn by increased soil pH upon biochar addition, and/or competition for uptake between Mn and Ca, or the complexation of Mn ions by oxygenic functional groups on the biochar surface (Hue et al., 2001). Calcium, P, and Mg were also increased to some extent, whereas Fe and Al were decreased by biochar additions (Table 7).

Tissue N was higher in the treatment with the rice husk biochar than the lac tree 600 biochar. This was probably due to the higher N in the former. Similar explanation is applicable to the higher Ca and Mg in soybeans grown in soils amended with the lac tree 600 biochar. Higher N, P, K, Ca, and Mg concentrations were also found in soybeans grown in the treatment with biochar and compost. Undoubtedly, compost additions provided significant nutrients to nutrient-poor soils, especially highly weathered acid soils of the Tropics.

Nutrients in soybean shoots indicated that most were sufficient for adequate growth, except for N, which was 2.99% to 3.65%. This range is considered "low" for soybean in common situations, but Jumro (2011) and Sudarsono et al. (2013) reported that the best growth and yield of soybean cv. Anjasmoro in Indonesia were achieved with 3.3% and 3.7% N, respectively. The concentrations of P, K, Ca, and Mg were at values of sufficient to high, whereas Fe and Mn concentrations were high. Aluminum concentrations in soybean shoots were very high, but very loosely correlated with soybean growth in Guradog soil. A similar result was reported by Jackson (1967), who concluded that good correlations between Al content in the foliage of crop plants and Al toxicity were more the exception than the rule.

## SUMMARY AND CONCLUSIONS

Biochar characterization of physicochemical properties allowed us to choose appropriate biochars for improving soil productivity and enhancing plant growth. Six biochars derived from different woody materials were quantitatively shown to differ in ash; VM; fixed C; total C, H, N, and O; O:C and H:C ratios; pH; CCE; CEC; EC; elemental content; surface functional groups; and surface structure and porosity.

The higher capacity to improve the productivity of a Hawaiian acid soil and to support plant growth of the leucaena-, lac tree 450–, Hilo mixed wood–, and she oak wood–derived biochars than the mahogany and mountain gumbiochars could be attributed to the liming potential and nutrient content of the former group of biochars. More specifically, the additions of leucaena and lac tree

450 at 2% and Hilo mixed wood or she oak derived biochars at 4%, clearly increased soil pH and CEC and lowered the soil exchangeable Al to a nontoxic level, thereby increasing the growth of D. intortum, a forage legume sensitive to Al.

Additions of the lac tree wood 600 or rice husk biochars alone or in combination with lime and compost significantly improved the productivity of two Indonesian acid soils and enhanced soybean growth. Soil pH, CEC, and plant nutrients were markedly increased, and soil exchangeable Al was noticeably reduced upon applications of these biochars at 8% on the Guradog soil either alone or in combination with lime at 8 cmolc $kg^{-1}$ and compost at 0.2%, resulting in the highest growth of local soybean cv. Anjasmoro. Benefit-cost analysis showed that application of the rice husk biochar to the Guradog soil was more profitable than the lac tree wood 600 biochar.

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