

## *Cordyline fruticosa* Growth and Soil Microbial Quality with Topical Application of Coal Combustion By-Products Aggregates

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### ABSTRACT

Coal combustion by-products aggregates (CCAs) are a solidified composite of fly and bottom ashes (2:1, by wt). Here, we assessed the feasibility of beneficial use of CCAs as a soil amendment or conditioner by conducting an outdoor experiment with *Cordyline fruticosa* (lucky plant) to determine its growth and soil microbial quality under the influence of topical CCA application (95 tons ha<sup>-1</sup>). Enhanced growth of *C. fruticosa* with CCA application was noted, with respect to plant height, growth rate, leaf size, and leaf chlorophyll intensity. Soil dehydrogenase activity and total heterotrophic bacteria count were greater with topical CCA application, especially in the 5–15-cm layer, than in the control system without CCAs. The stimulated soil microbial quality due to the influence of CCAs was believed to be responsible, at least in part, for enhanced growth and health of *C. fruticosa*. Therefore, CCAs can be construed as beneficial as a soil amendment or conditioner for decorative plants such as *C. fruticosa*.

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### 1. Introduction

Many studies have been conducted to evaluate the effects of the application of coal combustion by-products (CCPs) on growth of plants and crop production (Mitra et al., 2005; Jala and Goyal, 2006). Fly ash (FA) is the most frequently tested CCP for its beneficial use, followed by bottom ash (BA) (Pandey and Singh, 2010). Reported benefits of CCP application include increase in water-holding capacity that could decrease irrigation frequency and subsequently increase water savings (Pandey et al., 2009), reduction of soil acidity-related constraints (Pandey and Singh, 2010), and supply of essential micronutrients for growth and development of plants (Hwang et al., 2010). Negative aspects of CCP application to agricultural use also have been reported. Examples are excessive trace element loadings that may increase

food-chain metals (Peralta-Videa et al., 2009), high soluble salt loadings that may reduce initial plant growth (Palumbo et al., 2004), and leaching of toxic substances into the ground water (Singh and Paul, 2001).

Coal combustion by-products aggregates (CCAs) are a solidified composite of FA and BA (2:1, by wt) that are mixed in water and then air dried. They gain strength with time due to cementitious reactions. CCAs can have better engineering properties than either FA or BA because they are structurally stronger and chemically more stable (Pando and Hwang, 2006). Hwang et al. (2010) recently reported enhanced growth of *Phaseolus vulgaris* (common bean) under the influence of CCAs as a micronutrient source. In addition, CCAs were documented to show sorptive removal of aqueous trinitrotoluene (Hwang and Hernandez, 2010).

Expanding the previous study (Hwang et al., 2010), the current study aimed to assess feasibility of beneficial use of CCAs as a soil amendment or conditioner for a larger sized plant than *P. vulgaris*. Thus, an outdoor experiment was conducted with *Cordyline*

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**Table 1**  
Metal oxide composition of the coal combustion by-products aggregates

Major component	%, wt	Minor component	%, wt
Silica, SiO <sub>2</sub>	34.5	Sodium oxide, Na <sub>2</sub> O	1.5
Lime, CaO	29.7	Magnesia, MgO	1.1
Sulfur trioxide, SO <sub>3</sub>	14.7	Potassium oxide, K <sub>2</sub> O	0.8
Alumina, Al <sub>2</sub> O <sub>3</sub>	12.0	Titania, TiO <sub>2</sub>	0.5
Ferric oxide, Fe <sub>2</sub> O <sub>3</sub>	4.2	Phosphorus pentoxide, P <sub>2</sub> O <sub>5</sub>	0.8
		Strontium oxide, SrO	
		Barium oxide, BaO	
		Manganese oxide, Mn <sub>3</sub> O <sub>4</sub>	

*fruticosa* (lucky plant) to evaluate its growth and soil microbial quality with topical CCA application.

## 2. Materials and Methods

### 2.1. Plant, soils, and CCP aggregates

*Cordyline fruticosa* (50–70 cm in height) were acquired from a local nursery farm. The target species was selected for its abundance in tropical and subtropical regions as a decorative plant. Potting soil (Rain Forest™) was purchased from a local nursery farm and used as received. Potting soil pH was  $9.69 \pm 0.02$  ( $n = 4$ ) and water-soluble total organic carbon (TOC) concentration was  $46.5 \pm 17.4 \text{ mg L}^{-1}$  ( $n = 4$ ) in 1:10 (wt/vol) deionized water, with a soil organic matter (SOM) concentration of  $24 \pm 4\%$  ( $n = 3$ ), a cation exchange capacity (CEC) of  $145 \pm 14 \text{ mEq/100 g}$  ( $n = 2$ ), and a water content of  $0.50 \pm 0.02\%$  ( $n = 2$ ). An organic-rich loamy sand soil (sand [75.1  $\pm$  2.0%], silt [13.3  $\pm$  0.2%], and clay [11.6  $\pm$  2.1%];  $n = 2$ ) was sampled from a local site. This soil had a pH of  $6.49 \pm 0.01$  ( $n = 2$ ) and  $32.1 \pm 14.3 \text{ mg water-soluble TOC L}^{-1}$  ( $n = 2$ ) in 1:10 (wt/vol) deionized water,  $5.69 \pm 0.15\%$  SOM ( $n = 3$ ),  $22 \pm 4 \text{ mEq/100 g CEC}$  ( $n = 2$ ), and a moisture content of  $0.32 \pm 0.01\%$  ( $n = 2$ ). The two soils were mixed at 1:1 (wt/wt) and used for all experiments.

CCAs were collected from a coal-burning power plant (AES Puerto Rico, Guayama, PR). The combustion occurs in a circulating fluidized bed. Selective noncatalytic reactions, a circulating dry scrubber with lime, and an electrostatic precipitator are used to reduce NO<sub>x</sub>, SO<sub>x</sub>, and particulate matter, respectively, in the flue gases. The main chemical components of the CCAs were (SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>), CaO, and SO<sub>3</sub>, representing 51%, 30%, and 15% by weight, respectively (Table 1). Although the American Society for

Testing and Materials (ASTM) classification of FA (ASTM Standard C618, ASTM International 2008) is not strictly applicable to CCAs, they can be regarded as a Class C CCP based on their chemical properties. However, the 15% SO<sub>3</sub> concentration in CCAs exceeds the maximum concentration of 5% for a Class C or F FA. Before use, they were crushed mechanically and sieved to collect particle sizes in the range of 2.36 to 9.53 mm.

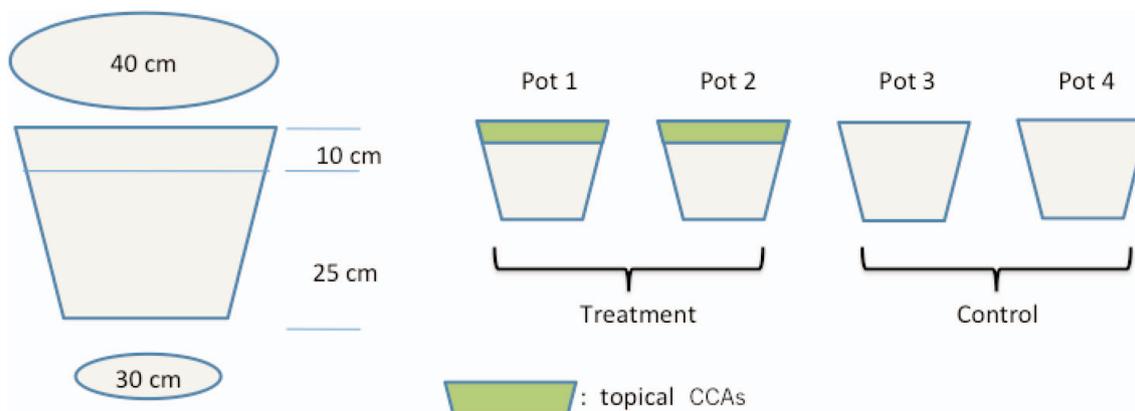
The synthetic precipitation leaching procedure (SPLP) was conducted with the CCAs to assess the risk of ground water contamination that might be posed by the CCA application to the land. For SPLP, CCA specimens (2.36–9.53 mm) were placed in a bottle container before the addition of leaching solution nitric/sulfuric acid (40/60% [wt]; pH 4.2) to provide a ratio of 20:1 mass ratio of leaching solution to CCA specimens. The containers were then agitated at  $30 \pm 2 \text{ rpm}$  for  $18 \pm 2 \text{ hours}$ . Extractant pH was measured at the end of the extraction period. The extractant was then filtered with a glass fiber filter (0.8- $\mu\text{m}$  pore size), and the filtrate was acidified with nitric acid to pH < 2.0 and stored under refrigeration (<4°C) before heavy metal analysis by inductively coupled plasma mass spectrometry.

### 2.2. Setup of plant pots

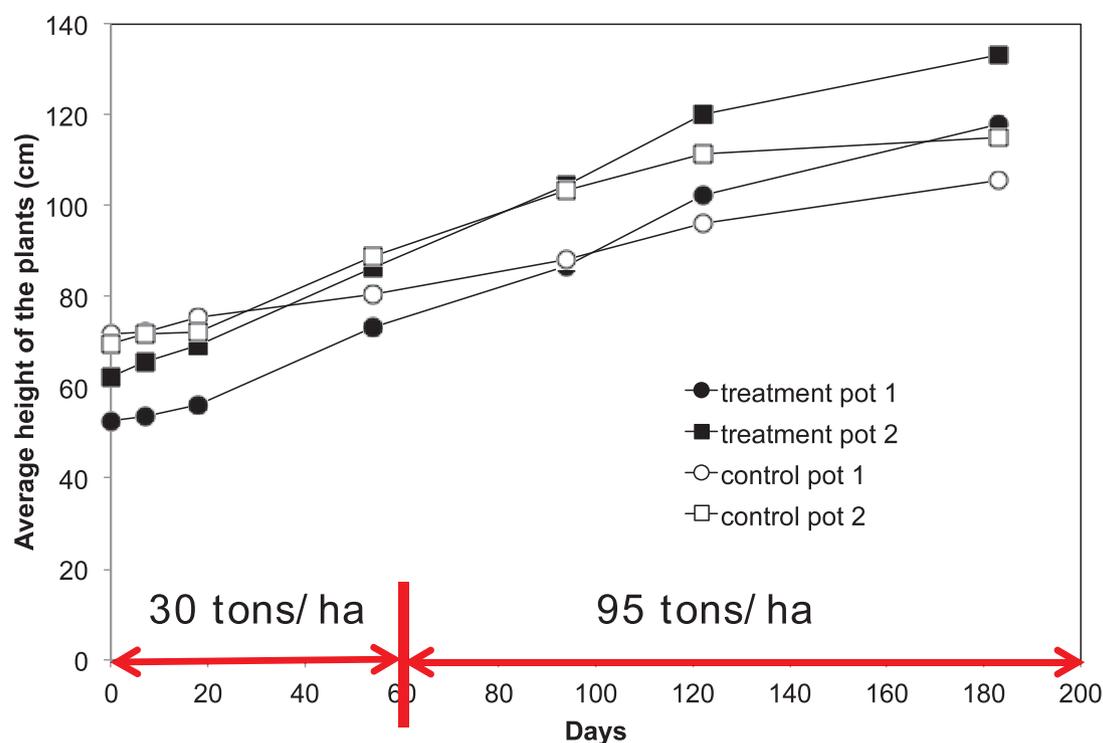
An outdoor experiment was designed to evaluate the effects of topical CCA application on *C. fruticosa* growth and soil microbial quality. Plant pots (40-cm top diameter  $\times$  35-cm height  $\times$  30-cm bottom diameter) were arranged in a trapezoidal shape (Fig. 1). The soil was packed 25 cm in depth at a bulk density of  $0.67 \pm 0.16 \text{ g cm}^{-3}$  ( $n = 4$ ) in each pot. Three *C. fruticosa* were transplanted into each pot. Then, CCAs were placed at an application rate of 30 tons ha<sup>-1</sup> on the perforated plates on the top of the soil in two pots (treatment pots). After 60 days, the CCA application rate was increased to 95 tons ha<sup>-1</sup>. The other two pots without CCAs were run as the control pots. The pots were put outside and subjected to natural weather. During the 180-day experiment, temperature (degrees Celsius), humidity (percentage), rain intensity (millimeters per hour), solar radiation (Watts per square meter), and ultraviolet index were in the range of 20–34, 49–97, 0–195, 0–1133, and 0–16, respectively.

### 2.3. Biochemical analysis

*Cordyline fruticosa* height was monitored with a tape measure, and plant health was monitored by measuring leaf chlorophyll



**Fig. 1.** Dimension and setup of the treatment and control pots. Each pot had three *Cordyline fruticosa*. CCAs = coal combustion by-products aggregates.



**Fig. 2.** *Cordyline fruticosa* growth in the treatment and control pots. Coal combustion by-products aggregates were topically applied at a rate of 30 tons ha<sup>-1</sup> and increased to 95 tons ha<sup>-1</sup> after 60 days. Values are averages of three *C. fruticosa* heights.

intensity with a chlorophyll meter (SPAD-502, Konica Minolta Sensing, Inc., Osaka, Japan) for 180 days. Lead concentration in leaves was determined at 180 days by the LeadTrak™ Fast Column Extraction method (detection limit, 5 µg L<sup>-1</sup>) following the Hach Digesdahl digestion process (method 8317). Specific conductivity was analyzed with the Orion specific conductivity meter (model 162, Thermo Fisher Scientific, Waltham, MA). Soil pH (in 0.1% [wt/vol] deionized water) was measured with a pH meter (Orion) in the supernatant after a vigorous mixing of soil slurry for 10 min. Soil organic matter was quantified by the Loss-on-Ignition (Sparks, 1996). Water-soluble TOC was determined with the Hach TOC Reagents Set (HACH, Loveland, CO) following a membrane filtration (0.45 µm) of the soil slurry supernatant after soil pH measurement. CEC was measured at pH 7 with ammonium acetate (Sparks, 1996).

Soil dehydrogenase activity and total heterotrophic bacteria (THB) count at different layers (each 5 cm) were analyzed at 30 and 180 days. For the sampling at 30 days, a thin-walled polyvinyl chloride tube (30 cm in length × 1 mm in thickness × 2.5 cm in diameter) was carefully penetrated through the center of the pot soil to take samples. The resulting void hole was refilled with the fresh potting soil with the same packing density (0.67 g cm<sup>-3</sup>). A composite sample from different layers was prepared for the 180-day samples. Soil dehydrogenase activity was measured via a colorimetric determination of 2,3,5-triphenyltetrazolium formazan produced by reduction of 2,3,5-triphenyltetrazolium chloride by soil microorganisms (Weaver et al., 1994). THB was monitored via a membrane filtration technique and incubation on the HPC growth medium (catalog no. 28124-50, Hach) for 48 h at 35°C.

#### 2.4. Statistical analysis

Significant differences in *C. fruticosa* growth, leaf chlorophyll, leaf width, soil dehydrogenase activity, and THB count between the

CCA-applied treatment pots and the control pots were determined by Student's *t* test. Differences between means at a confidence level of 5% ( $p < 0.05$ ) were considered to be statistically significant.

### 3. Results and Discussion

#### 3.1. *Cordyline fruticosa* growth

*Cordyline fruticosa* grown in the pots with CCAs were taller at the end of the experiment, despite smaller heights at the beginning of the experiment (Fig. 2), compared with controls. The enhanced *C. fruticosa* growth in the treatment pots was probably attributed to the micronutrients that leached from the CCAs. The concentrations of individual micronutrients were not quantified in this study. Instead, a subset of batch experiments analyzed specific conductivity of varying CCA quantities in deionized water (0–50 g L<sup>-1</sup>) to obtain a rapid estimate of the dissolved solids concentrations (Sawyer et al., 2003). The final conductivities after 72-h reaction time were 770 µS cm<sup>-1</sup> (5 g CCAs L<sup>-1</sup>) and 3090 µS cm<sup>-1</sup> (50 g CCAs L<sup>-1</sup>). Therefore, the CCAs, a solidified composite of FA and BA, probably provided various ionic micronutrients (e.g., Ca, K, P, and S) that enhanced *C. fruticosa* growth in the current study (Elseewi et al., 1978).

A growth rate ( $[K]$ , centimeters per day) was calculated as follows:  $K = (H_t - H_0)/t$ , where  $H_t$  and  $H_0$  are the *C. fruticosa* height (centimeters) at time  $t$  (day) and at the beginning of the experiment, respectively. *Cordyline fruticosa* grew faster in the CCA-applied treatment pots than in the control pots (Table 2), suggesting that the topical CCA application at 95 tons ha<sup>-1</sup> enhanced *C. fruticosa* growth. In a previous study, *P. vulgaris* grew better under the influence of CCAs at application rates of 65 to 800 tons ha<sup>-1</sup>, having taller shoots, more leaves, and higher leaf chlorophyll intensity (Hwang et al., 2010).

**Table 2**  
*Cordyline fruticosa* growth rate ( $K$ ) in treatment and control pots

Pot	$K$ (cm day <sup>-1</sup> )	Initial height, $H_0$ (cm)	Coefficient of determination, $R^2$
Treatment 1	0.38a	51.8a	0.99
Treatment 2	0.41a	63.6b	0.98
Control 1	0.19b	71.3b	0.99
Control 2	0.28b	71.3b	0.94

Note:  $K = (H_t - H_0)/t$ , where  $H_t$  and  $H_0$  are the *C. fruticosa* height (centimeters) at time  $t$  (day) and at the beginning of the experiment, respectively. Values are averages of three *C. fruticosa* in each pot. Values with different letters in each column are significantly different from each other ( $p < 0.05$ ).

### 3.2. Leaf chlorophyll intensity

Chlorophyll is one of the major chloroplast components for photosynthesis and allows plants to obtain energy from light. Therefore, relative chlorophyll intensity has a positive relationship with photosynthetic rate and hence plant health (Valdez-Pérez et al., 2011).

*Cordyline fruticosa* grown in the CCA-applied pot had statistically greater chlorophyll intensities ( $p < 0.05$ ), maintaining ~40 SPAD from 20 to 180 days (Fig. 3). In comparison, *C. fruticosa* grown in the control pots showed a gradual decrease of chlorophyll intensity, resulting in <30 SPAD at the end of the experiment.

The three largest leaves of each *C. fruticosa* in each pot were measured for their width at the end of the experiment. The plants in the CCA-applied treatment pots had significantly wider leaves ( $9.8 \pm 0.2$  cm;  $n = 18$ ) than the control plants ( $8.0 \pm 0.5$  cm;  $n = 18$ ).

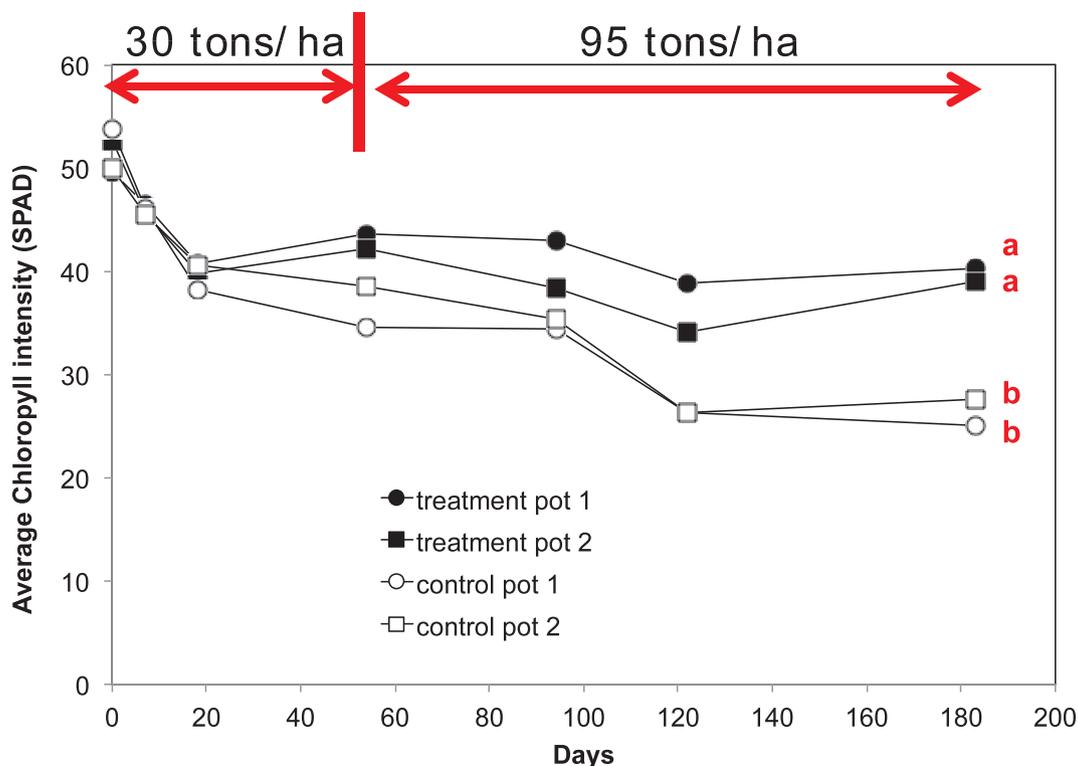
### 3.3. Heavy metals

Lead concentration in *C. fruticosa* leaves grown in the treatment pots was zero or below the detection limits ( $5 \mu\text{g L}^{-1}$ ), indicative of no inhibitory effect of toxic heavy metals (at least lead) by CCAs in *C. fruticosa* leaves. Instead, *C. fruticosa* grew more, faster, and healthier with topical CCA application. Manios et al. (2003) reported that an inhibitory effect of heavy metal accumulation on *Typha latifolia* (cattail) leaves was correlated with a decrease in chlorophyll intensity. However, it should be noted that coal ash, particularly FA, might leach potentially toxic heavy metals at concentrations above (Dutta et al., 2009) or below (Mullen, 1992) permissible limits, depending on their chemical properties and site-specific characteristics (Soco and Kalembkiewicz, 2007; Jegadeesan et al., 2008).

The concentrations of heavy metals in the filtrate from the SPLP with CCAs were below the maximal contaminant levels and secondary levels, except for those of selenium and thallium (Table 3). Selenium is toxic at high levels to living organisms, although it is a micronutrient essential for normal cellular function (MacFarquhar et al., 2010). Probably, thallium is highly toxic to living organisms, comparable to the toxicity of lead and mercury (Peter and Viraraghavan, 2005). Therefore, special caution should be given to the fate and transport of these heavy metals once they leach out of CCAs in soils.

### 3.4. Soil microbial quality

Dehydrogenase activity has been used as a valid biomarker of soil microbial quality (Reddy and Faza, 1989). Generally, greater dehydrogenase activities were observed in the CCA-applied treatment pots than control pots and were substantially greater



**Fig. 3.** Leaf chlorophyll intensity of *Cordyline fruticosa* grown in the treatment and control pots. Values with different letters at 180 days are significantly different from each other ( $p < 0.05$ ). Values are averages of the nine largest leaves (three from each *C. fruticosa*).

**Table 3**

Heavy metal concentrations from synthetic precipitation leaching procedure (SPLP)

Group 1 <sup>1</sup>	MCL <sup>2</sup> (mg L <sup>-1</sup> )	SPLP (mg L <sup>-1</sup> )	Group 2 <sup>3</sup>	Secondary standard (mg L <sup>-1</sup> )	SPLP (mg L <sup>-1</sup> )
Antimony	0.006	0.0022 (0)	Aluminum	0.05–0.2	0.0023 (0)
Arsenic	0.01	0.0014 (0)	Copper	1.0	0.0026 (0.0003)
Barium	2	0.48 (0.02)	Iron	0.3	0.018 (0)
Beryllium	0.004	0.0014 (0.0001)	Manganese	0.05	0.0008 (0.0007)
Cadmium	0.005	0.0004 (0)	Silver	0.1	0.016 (0)
Chromium	0.1	0.0343 (0.008)	Zinc	5	0.0014 (0)
Lead	0.015 <sup>4</sup>	0.0089 (0.0023)			
Selenium	0.05	0.0547 (0.0189)			
Thallium	0.002	0.0018 (0)			

Note: Values are averages of six measurements; standard deviations are shown in parentheses. pH values at the end of the SPLP were  $10.1 \pm 0.2$  ( $n = 6$ ).

<sup>1</sup> Heavy metals under National Primary Drinking Water Regulations.

<sup>2</sup> Maximum contaminant level.

<sup>3</sup> Heavy metals under National Secondary Drinking Water Regulations.

<sup>4</sup> TT: action level = 0.015.

in 5–15-cm layers of the treatment pots (Table 4). Dehydrogenase activity was constant over time (30 vs. 180 days) in both the treatment and control pots.

A logarithmic correlation was found between the soil dehydrogenase activity and THB count (Fig. 4): the greater the soil dehydrogenase activity, the higher the THB count. Thus, THB count had a very similar trend to the dehydrogenase: greater THB in the treatment pots than in the control pots and the greatest THB count in 5–15-cm layers of the treatment pots. Unlike the dehydrogenase activity, THB count was decreased over time in the control pots.

Soil pHs in 5–25-cm layers were in the range of 6.4–7.0 in the control pots and 6.8–7.0 in the CCA-applied treatment pots (Table 4). Like soil dehydrogenase activity and THB count, soil pHs were maintained constant over time (30 vs. 180 days). Despite greater soil microbial population and activity that could result in acidic soil pH, narrower pH ranges were observed in the CCA-applied treatment pots. This finding was attributed to liming effect (i.e., dissolution of calcium oxides) exerted by the applied CCAs on soil acidity (McCarty et al., 1994; Manoharan et al., 2010; Pandey and Singh, 2010). In 0–5-cm layers, substantially higher pHs were measured in the treatment pots (~7.6) than in the control pots (~7.0). In a separate experiment, aqueous pHs in the CCA extractant with deionized water were at 10.5–10.8 after 2 hours of reaction with a batch application rate of 5–30 g L<sup>-1</sup>. These high pHs can inhibit

plant growth (Marcar et al., 2002). However, soil pHs in the first 0–5-cm layers were close to neutral perhaps due to the soil's buffering capacity (i.e., cation exchange capacity). Similarly, pH values at the end of the SPLP were at  $10.1 \pm 0.2$  ( $n = 6$ ).

Several researchers found that soil microbial populations and enzymatic activity after organic amendments derived from wastes stimulated crop productivity and yields (Pascual et al., 1997; Arancon et al., 2004). Probably, improved soil dehydrogenase activity and THB count that were benefited from CCAs could be responsible for the increased *C. fruticosa* growth rate and health in the CCA-applied treatment pots.

#### 4. Conclusions

CCAs, a solidified mixture of FA and BA (2:1, by wt), were tested for their potential use as an alternative for soil amendments or conditioners. *Cordyline fruticosa* grew better with topical CCA application at 95 tons ha<sup>-1</sup>, producing taller plants with faster growth rate and greater leaf chlorophyll intensity ( $p < 0.05$ ). An enhanced soil microbial quality also was achieved with topical CCA application. Greater dehydrogenase activity and THB count were found in the CCA-applied treatment pots than in the control pots. The greatest values of dehydrogenase activity and THB count were determined in 5–15-cm layers of the CCA-applied treatment pots. CCAs are believed to stimulate soil microbial population and

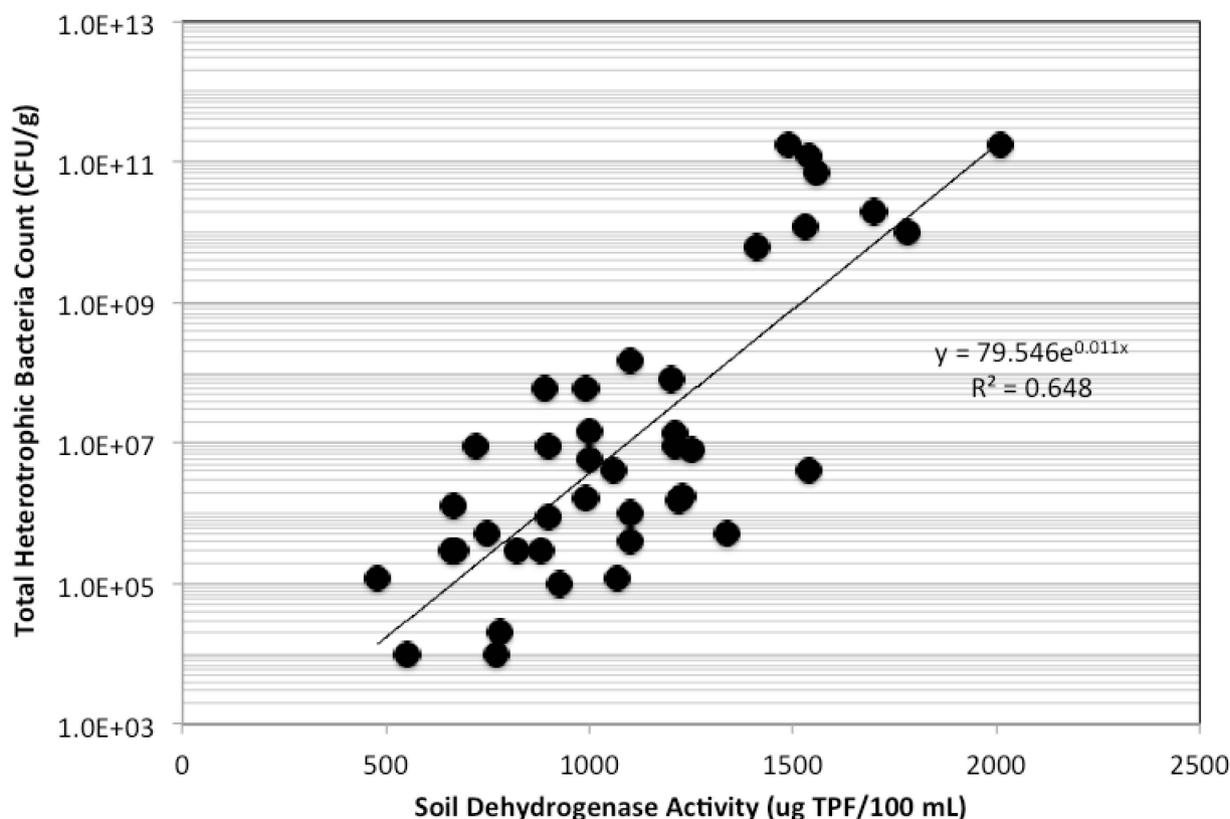
**Table 4**

Soil dehydrogenase activity, total heterotrophic bacteria count, and soil pH at different layers

Layer (cm)	After 30 days				After 180 days			
	Dehydrogenase activity (µg TPF <sup>1</sup> 100 mL <sup>-1</sup> )		pH		Dehydrogenase activity (µg TPF 100 mL <sup>-1</sup> )		pH	
	Control	Treatment	Control	Treatment	Control	Treatment	Control	Treatment
0–5	890 ± 240a	1055 ± 219a	7.1 ± 0.2a	7.6 ± 0.1b	668 ± 4a	1280 ± 85b	7.0 ± 0.2a	7.5 ± 0.2b
5–10	1095 ± 149a	1470 ± 85b	6.4 ± 0.1a	6.9 ± 0.1b	1045 ± 78a	1525 ± 50b	6.5 ± 0.1a	7.0 ± 0.1b
10–15	995 ± 149a	1895 ± 163b	6.8 ± 0.1a	6.8 ± 0.1a	1000 ± 0a	1620 ± 113b	6.9 ± 0.1a	6.8 ± 0.1a
15–20	985 ± 120a	1240 ± 14a	6.9 ± 0.1a	6.8 ± 0.1a	815 ± 92a	1375 ± 233b	7.0 ± 0.2a	6.9 ± 0.1a
20–25	720 ± 85a	1013 ± 124a	6.8 ± 0.1a	6.9 ± 0.1a	515 ± 50a	795 ± 35b	6.8 ± 0.1a	6.9 ± 0.1a

Note: Values are averages ± standard deviations of three measurements. Statistical comparison was made with the values between the control and treatment pots for each parameter. Values with different letters are significantly different from each other ( $p < 0.05$ ).

<sup>1</sup> TPF = 2,3,5-triphenyltetra zolium formazan.



**Fig. 4.** Correlation between soil dehydrogenase activity and total heterotrophic bacteria count. Values are from both the control and treatment pots. CFU = colony-forming unit; TPF = 2,3,5-triphenyltetra zolium formazan.

activity that subsequently enhance *C. fruticosa* growth and health. Therefore, it is construed that CCAs at a topical application rate of 95 tons ha<sup>-1</sup> could improve the growth and quality of *C. fruticosa* and stimulate soil microbial quality when they are applied as a soil amendment or conditioner.

However, it is warranted to conduct more detailed experiments with greater numbers of replicates in different CCA application modes to the soil (e.g., mixed with soils, added as subsoil substitute) and to analyze other trace elements in the soil and plant tissue because lead may not be the only potentially toxic risk-driver of concern.

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