

Hindawi Publishing Corporation  
Applied and Environmental Soil Science  
Volume 2011, Article ID 475185, 11 pages  
doi:10.1155/2011/475185

## Research Article

# Fly Ash and Composted Biosolids as a Source of Fe for Hybrid Poplar: A Greenhouse Study

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Received 16 November 2010; Revised 8 February 2011; Accepted 10 February 2011

Academic Editor: Silvana I. Torri

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Soils of northwest New Mexico have an elevated pH and CaCO<sub>3</sub> content that reduces Fe solubility, causes chlorosis, and reduces crop yields. Could biosolids and fly ash, enriched with Fe, provide safe alternatives to expensive Fe EDDHA (sodium ferric ethylenediamine di-(o-hydroxyphenyl-acetate)) fertilizers applied to *Populus* hybrid plots? Hybrid OP-367 was cultivated on a Doak sandy loam soil amended with composted biosolids or fly ash at three agricultural rates. Fly ash and Fe EDDHA treatments received urea ammonium nitrate (UAN), biosolids, enriched with N, did not. Both amendments improved soil and plant Fe. Heavy metals were below EPA regulations, but high B levels were noted in leaves of trees treated at the highest fly ash rate. pH increased in fly ash soil while salinity increased in biosolids-treated soil. Chlorosis rankings improved in poplars amended with both byproducts, although composted biosolids offered the most potential at improving Fe/tree growth cheaply without the need for synthetic inputs.

## 1. Introduction

The New Mexico State University Agricultural Science Center at Farmington, San Juan County, has been exploring short rotation hybrid poplar trees for fiber and timber production, biofuel, and phytoremediation purposes. Adaptability trials involving numerous *Populus* crosses have produced a range of responses. Of these, Fe deficiency chlorosis (interveinal yellowing of juvenile leaves) has been observed because soil pH can exceed 8 with moderate to high CaCO<sub>3</sub> levels. Under these conditions, soil iron is mostly in the form of well-crystallized iron oxides (e.g., hematite and goethite) and almost insoluble and unavailable to plants [1]. On our research plots, chelated iron fertilizer in the form of Fe EDDHA is applied to alleviate chlorosis symptoms. Considering that 5 kg Fe EDDHA material—enough to cover approximately 1 ha season<sup>-1</sup>—costs approximately \$200, fertilizing large-scale plantations may be cost prohibitive.

On the other hand, fly ash, a byproduct from coal combustion, can provide plant-available Fe and other micronutrients [2–7]. Fly ash exits the combustion chamber with the flue gas and is captured by electrostatic precipitators, wet scrubbers, or other mechanical/chemical trap [8]. Particle sizes range from 0.01 to 100 μm allowing a large amount of surface area to mass [9]. Nearly 3.9 million Mg of coal combustion products (ash + flue gas desulfurization products) are produced in San Juan County each year by two coal fired generating plants, and both power plants are actively seeking recycling options (Salisbury, 2003, personal communication).

Biosolids (dewatered sewage sludge) also increase levels of plant-available Fe on calcareous soils [6, 10, 11] and are a source of other plant-essential elements including N and P [12, 13]. Iron enhancement in biosolids results from multiple factors at the wastewater treatment facility. When washed into treatment plants through storm runoff, iron oxides can

be reduced and reprecipitated as weakly crystalline plant-available iron phosphates [14]. Salts of  $\text{FeCl}_3$  or  $\text{FeCl}_2$  used to capture phosphorus from the waste stream during the treatment process also increase the iron phosphate content of biosolids [14, 15]. The city of Albuquerque, 290 km southeast of Farmington, produces 142 Mg of biosolids per day and is a regional leader in seeking land-use disposal and marketing options of processed, composted biosolids [16].

Environmental consequences for both byproducts also have been documented. Fly ash can contain elevated levels of heavy metals, increase boron to toxic levels, can act as liming agents because of their high Ca/Mg content, and can increase soil salinity [17, 18]. Biosolids also have the potential to increase salinity, heavy metals, and persistent organic pollutants such as antibiotics, and personal care products that enter the waste stream [19, 20]. If an environmentally responsible use can be established, recycling of these byproducts to agricultural lands may present an attractive disposal alternative because of the large land area devoted to crop production within a relatively short distance from the power plant or wastewater treatment facilities in the Farmington area. The objectives of this study were to pilot test the application of fly ash and composted biosolids at three rates to a high pH soil from Northwest, NM. Specific objectives were to

- (1) determine if Fe nutrition of soil and the growth of hybrid poplar clone OP-367 could be improved by amending soil with each amendment,
- (2) examine potential environmental issues, including heavy metal contamination, salinity, and pH changes in the soil, caused by each amendment.

## 2. Materials and Methods

### 2.1. Soil and Treatments

**2.1.1. 2004 Study Soil and Treatments.** A Doak sandy loam (fine-loamy, mixed, mesic Typic Haplargid) [21] was collected from the top 20–25 cm of the plow layer from an agricultural field located at the New Mexico State University Agricultural Science Center, Farmington (lat.  $36^\circ 41' 0''$  N; long.  $108^\circ 18' 36''$  W; elevation 1,700 m). Soil was sieved through 6-mm  $\times$  6-mm mesh to remove clods then transported to the NMSU Fabian Garcia horticulture farm greenhouse complex (Las Cruces, NM). Prior to container filling, a fiberglass mesh screen was used to line standard 7.5 L nursery containers to prevent soil loss through drainage holes. Each container was filled to a dry weight of 9 kg. Once filled, the surface area at the top of each container was 366 cm<sup>2</sup>.

Fly ash was collected from the APS Four Corners Power Plant (Farmington, NM). Fly ash at the power plant is stored in a lined ash impoundment area adjacent to plant. Composted biosolids were collected from the City of Albuquerque, NM Pilot Composting Facility. The biosolids were a 1 : 3 ratio of dewatered sewage sludge mixed with chipped yard waste that were composted in large windrows at 57°C for six weeks. The composting process reduces pathogen

concentrations to comply with USEPA standards for “Class A” classification [16]. After composting, the biosolids were drum sieved before trucking to Farmington. The fly ash required no sieving.

Composted biosolids and fly ash treatments were applied to the nursery containers February 24, 2005 at two agricultural rates: 22.75 Mg ha<sup>-1</sup> (82.1 g byproduct per container) and 45.5 Mg ha<sup>-1</sup> (164 g byproduct per container). A third rate was applied based on two criteria: (1) the amount of Fe in the Farmington soil after a DTPA- (diethylenetriaminepentaacetic acid-) extractable Fe baseline soil test was conducted and (2) the percent available Fe in each amendment that could be applied to the soil as a fertilizer to correct a potential Fe deficiency. The complete baseline soil chemical analysis used to determine DTPA application rates was established earlier for both byproducts and is presented in Table 1. The DTPA Fe content of the Farmington soil was 1.2 mg kg<sup>-1</sup>. For soils with a test report of 0.0–2.5 mg kg<sup>-1</sup> Fe, Jones and Jacobsen [22] recommend an application rate of 4.5 k Fe ha<sup>-1</sup> in order to overcome Fe deficiency in susceptible crops. The DTPA-extractable iron in the fly ash was 0.00609% Fe and 0.0329% Fe for the biosolids. Expressed as fractions, DTPA values determined for each byproduct were used as divisors to the 4.5 k Fe ha<sup>-1</sup> recommendation which yielded the equivalent of 74 Mg of fly ash ha<sup>-1</sup> (270.5 g applied per container) and 14 Mg biosolids ha<sup>-1</sup> (50.1 g per container) application rates. These were the highest and lowest rates for fly ash and composted biosolids, respectively. Treatments were incorporated by removing the top 10 cm of soil from each nursery container, placing the contents into a plastic bucket, and mixing in the amendment before returning the contents to the nursery container. An Fe fertilizer check, Sprint Sequestrene 138 (use of a trademarked product does not imply an endorsement by the NMSU Agricultural Experiment Station), 6% EDDHA chelated Fe (Becker Underwood, Ames Iowa), was applied as a soil drench once at week three at an application rate of 4.5 kg Fe ha<sup>-1</sup> (275 mg Fe EDDHA per container). The application rate was based on the soil test report and percent available Fe in the product (6%) to supply the literature recommendations as described previously. Unamended soil served as the control.

**2.2. Plant Material.** Hybrid poplar OP-367 (*Populus deltoides*  $\times$  *P. nigra*) is a commercial hybrid that performs well in Farmington but benefits from supplemental Fe. Uniform 30 cm long cuttings obtained from Broadacres Nursery (Hubbard, OR) were soaked for 3 days in tap water before transplanting (February 27, 2005) directly into nursery containers.

**2.3. Other Cultural Practices.** Greenhouse temperatures averaged 16°C (min) and 41°C (max). Containers were kept at or below field capacity and were not leached to examine the potential for salt buildup in the soil. The total amount of water applied to each container over the course of the study was 647 mm.

TABLE 1: Selected chemical properties of Albuquerque biosolids and APS fly ash.

Characteristic	Composted biosolids	Fly ash <sup>b</sup>
pH (1:2) <sup>c</sup>	7.4	12.4
EC (dS m <sup>-1</sup> ) <sup>d</sup>	14.0	6.7
SAR (mmol L <sup>-1</sup> ) <sup>d</sup>	4.75	2.04
NO <sub>3</sub> -N (mg kg <sup>-1</sup> ) <sup>e</sup>	71.3	2.71
TKN (mg kg <sup>-1</sup> ) <sup>f</sup>	1850.0	NT
P (mg kg <sup>-1</sup> ) <sup>c</sup>	231.3	17.0
K (mg kg <sup>-1</sup> ) <sup>c</sup>	5723.3	11.7
Zn (mg kg <sup>-1</sup> ) <sup>c</sup>	44.9	0.6
Fe (mg kg <sup>-1</sup> ) <sup>c</sup>	420.3	78.4
Fe by DTPA (mg kg <sup>-1</sup> ) <sup>z</sup>	329.0	60.90
Mn (mg kg <sup>-1</sup> ) <sup>c</sup>	20.6	8.1
Cu (mg kg <sup>-1</sup> ) <sup>c</sup>	15.0	1.2
Ca (mg kg <sup>-1</sup> ) <sup>c</sup>	3557.0	5650.0
Mg (mg kg <sup>-1</sup> ) <sup>c</sup>	657.7	31.0
Na (mg kg <sup>-1</sup> ) <sup>c</sup>	855.3	53.6
S (mg kg <sup>-1</sup> ) <sup>e</sup>	529.5	306.7
Al (mg kg <sup>-1</sup> ) <sup>e</sup>	807.2	348.7
As (mg kg <sup>-1</sup> ) <sup>e</sup>	18.5	10.3
B (mg kg <sup>-1</sup> ) <sup>e</sup>	40.1	59.5
Ba (mg kg <sup>-1</sup> ) <sup>e</sup>	211.8	904.1
Be (mg kg <sup>-1</sup> ) <sup>e</sup>	ND	ND
Cd (mg kg <sup>-1</sup> ) <sup>e</sup>	2.3	1.3
Co (mg kg <sup>-1</sup> ) <sup>e</sup>	3.4	0.8
Cr (mg kg <sup>-1</sup> ) <sup>e</sup>	13.9	2.9
Mo (mg kg <sup>-1</sup> ) <sup>e</sup>	ND	ND
Ni (mg kg <sup>-1</sup> ) <sup>e</sup>	8.6	1.9
Pb (mg kg <sup>-1</sup> ) <sup>e</sup>	18.8	5.0
Se (mg kg <sup>-1</sup> ) <sup>e</sup>	ND	ND
Tl (mg kg <sup>-1</sup> ) <sup>e</sup>	ND	ND
V (mg kg <sup>-1</sup> ) <sup>e</sup>	22.1	8.4
Bi (mg kg <sup>-1</sup> ) <sup>e</sup>	ND	ND
Li (mg kg <sup>-1</sup> ) <sup>e</sup>	8.6	3.8
Sr (mg kg <sup>-1</sup> ) <sup>e</sup>	163.8	37.9
Si (mg kg <sup>-1</sup> ) <sup>e</sup>	322.7	308.7
Ag (mg kg <sup>-1</sup> ) <sup>e</sup>	5.2	ND

<sup>a</sup>Mean of 6 samples for pH, P, K, Zn, Fe, Mn, Cu, Ca, Mg, and Na.

<sup>b</sup>Mean of 3 samples for S, Al, As, B, Ba, Be, Cd, Co, Cr, Mo, Ni, Pb, Se, Tl, V, Bi, Li, Sr, Si, and Ag.

<sup>c</sup>Analyzed at Soil Chemistry Research Laboratory, NMSU, Las Cruces, NM.

<sup>d</sup>Analyzed at Agricultural Testing and Research Laboratory, NAPI, Farmington, NM.

<sup>e</sup>Analyzed at the NMSU Soil, Water, and Air Testing Laboratory, Las Cruces, NM.

<sup>f</sup>Literature value supplied by Glass (personal communication, 2006).

ND = not detected. NT = not tested.

The control soil has a low N content (less than 1% organic matter). In addition, N is volatilized during combustion, making fly ash even lower in N content. Therefore, the fly ash- and Fe EDDHA-treated trees received the equivalent of 90 kg N ha<sup>-1</sup> N (split into 14 application times spread

over the course of the study, applied to trees in the irrigation water) in the form of urea ammonium nitrate (UAN 32-0-0) in order to maintain similar values of N in all treatments. This was necessary because the composted biosolids contained 85.5 kg N ha<sup>-1</sup> when applied at the 44.5 Mg ha<sup>-1</sup> rate determined from NO<sub>3</sub>-N using the ion-specific electrode method [23] and Kjeldahl N measured by the City of Albuquerque (Glass, 2006 personal communication) (Table 1).

**2.4. Chlorophyll Analysis.** Leaf chlorophyll content was monitored using a handheld Minolta SPAD- (soil plant analysis development-) 502 meter. The SPAD meter nondestructively measures transmittance of the leaf in red and infrared wavelengths (650 and 940 nm, resp.) giving a unitless leaf “greenness” value [24]. As SPAD values increase, leaf chlorosis decreases. For the clone OP-367, SPAD values were previously shown to correlate well with Fe ( $r^2 = 0.58$ ) and total chlorophyll analyzed by HPLC ( $r^2 = 0.85$ ) [25]. SPAD readings were made on April 12, and June 22 by measuring the first 10 fully expanded leaves (beginning 5-6 nodes down from the apical bud) on each tree.

**2.5. Postharvest Analysis.** The study was terminated July 6, 2005 at which point leaves were removed from each tree and passed through a leaf area meter. Leaves were decontaminated of Fe sources from dust/soil by dipping in a 0.01% phosphate-free detergent bath (0.1 mL detergent L<sup>-1</sup> tap H<sub>2</sub>O) [26] followed by rinsing with tap water under low pressure to remove soap residues. Leaves were then dipped into two baths of distilled water, bagged, dried for 24 hours at 70°C, and then weighed.

Stems were severed 2 cm from the top of the original cutting and measured for basal diameter and overall length. Soil was removed from the root ball (roots plus original cutting) then sieved through a 3 mm × 3 mm mesh to remove root pieces. Roots were then dipped in six water baths to remove residual soil. Roots were then severed from the original cutting and rinsed under low pressure. Stems and roots were then dried separately at 70°C for 72 hours before weighing.

**2.5.1. Plant Fe and N Analysis.** Dried leaves and stem material were ground to a fine powder using a stainless steel coffee grinder (cleaned thoroughly between samples) and stored in snap cap vials at room temperature until chemical analysis. Plant Fe was extracted with 20% trace metal grade HCl after dry ashing [27] and analyzed by inductively coupled plasma-optical emission spectroscopy (ICP-OES; Perkin-Elmer Optima 4300 DV ICP-OES). Plant total nitrogen (TN) was determined directly by combustion (LECO TruSpec CNS).

**2.5.2. Plant Tissue Heavy Metal Analysis.** Following the method described by Miller [28], microwave-assisted acid digestion using Teflon pressure digestion vessels was used to extract Cr, Pb, Se, As, Ag, Ba, and Cd from leaves. Acid digests were then analyzed by ICP-OES. All plant tissue macro elements, Fe, B, and heavy metals are expressed on a dry weight basis.

**2.5.3. Soil Analysis.** Soil was analyzed for pH (1:2, soil:water), extractable P, and Fe by ammonium bicarbonate-DTPA ( $1.0 \text{ mol L}^{-1} \text{ NH}_4\text{HCO}_3 + 0.005 \text{ mol L}^{-1} \text{ DTPA}$  at pH 7.6) [29]. Extracts were analyzed by ICP-OES. Soil  $\text{NO}_3\text{-N}$  was analyzed using the ion specific electrode method [23]. Electrical conductivity (EC) and sodium adsorption ratio (SAR) were measured on saturated paste extracts. Soil Cr, Pb, Se, As, Ag, Ba, and Cd concentrations were determined by ICP-OES following the USEPA 3051A [30] microwave-assisted acid digestion method for soil samples.

All laboratory analyses were conducted at the Navajo Agricultural Products Industry (NAPI) Agricultural Testing and Research Laboratory (Farmington, NM), NMSU Plant and Environmental Sciences Soils Research group laboratory (Las Cruces, NM), and the NMSU Soil, Water, and Air Testing (SWAT) laboratory.

**2.6. Experimental Design and Statistical Analysis.** The study was a randomized complete block design on two benches to compensate for temperature gradients within the greenhouse. There were eight containers per each treatment. Containers were redistributed once per week within blocks on the benches to help ensure that all trees received equal amounts of light exposure.

Analysis of variance was done in SAS (Cary, NC) using the PROC Mixed statement. All pairwise comparisons were made when significant differences were observed using Fisher's protected LSD at an alpha 0.05 level calculated by the method described by Littell et al. [31]. Concerning plant elements (Fe, N, B, and Ba), when significant accumulations of these elements were found in stems and leaves, the statistics were performed on the total plant accumulation (leaves + stems). In the case when no significant differences were detected in stems but were in leaves, only leaves are reported to simplify the data reporting. Correlation analysis using the PROC CORR command was performed to determine linear relationships between plant growth and environmental/plant toxicity parameters (i.e., pH, EC, SAR, and boron).

Codes for biosolid treatments are referenced the following way: ACB 22.75, ACB 44.5 (for Albuquerque composted biosolids at  $22.75 \text{ Mg ha}^{-1}$  and  $44.5 \text{ Mg ha}^{-1}$  application rates, resp.), ACB DTPA (biosolids applied at a rate based upon its DTPA extractable, plant-available Fe). Codes for fly ash plus urea ammonium nitrate fertilizer treatments are referenced as FA 22.75 + UAN, FA 44.5 + UAN, and FA DTPA + UAN (fly ash applied at a rate based upon its DTPA extractable, plant-available Fe). The Fe EDDHA plus urea ammonium nitrate fertilizer treatment is referenced as Fe EDDHA + UAN.

### 3. Results and Discussion

#### 3.1. Soil/Plant Nutrient Status and Tree Growth after Amending with Byproducts

**3.1.1. Soil Fe, Chlorosis, and Tissue Fe Responses.** Even though soil Fe in OP-367 trees receiving Fe EDDHA + UAN was

TABLE 2: Soil  $\text{NO}_3\text{-N}$ , P, and Fe a Doak sandy loam after amending with Fe EDDHA + UAN (urea ammonium nitrate), fly ash + UAN, and composted biosolids ( $n = 8$  per treatment). Means with the same letter are not significantly different at  $\alpha = 0.05$  level.

Treatments <sup>a</sup>	Fe ( $\text{mg kg}^{-1}$ )	$\text{NO}_3\text{-N}$ ( $\text{mg kg}^{-1}$ )	P ( $\text{mg kg}^{-1}$ )
Control	4.44 d	2.3 c	9.39 d
Fe EDDHA + UAN	4.84 d	3.4 a	8.55 d
ACB DTPA	16.63 c	2.4 c	23.23 c
ACB 22.75	22.13 b	2.5 bc	30.71 b
ACB 44.5	37.09 a	3.3 ab	52.05 a
FA 22.75 + UAN	5.27 d	3.8 a	8.87 d
FA 44.5 + UAN	5.51 d	3.5 a	8.90 d
FA DTPA + UAN	6.48 d	3.7 a	8.59 d
Mean	12.80	3.1	18.79
LSD	3.28	0.82	2.56
F Value	104.09	4.64	306.1
Pr > F	<.0001	0.0004	<.0001

<sup>a</sup>Codes for biosolid treatments are referenced the following way: ACB 22.75, ACB 44.5 (for Albuquerque composted biosolids at  $22.75 \text{ Mg ha}^{-1}$  and  $44.5 \text{ Mg ha}^{-1}$  application rates, resp.), ACB DTPA (biosolids applied at a rate based upon its DTPA extractable, plant-available Fe; equivalent to  $14 \text{ Mg ha}^{-1}$ ). Codes for fly ash plus urea ammonium nitrate fertilizer treatments are referenced as FA 22.75 + UAN, FA 44.5 + UAN, and FA DTPA + UAN (fly ash applied at a rate based upon its DTPA extractable, plant-available Fe; equivalent to  $74 \text{ Mg ha}^{-1}$ ). The Fe EDDHA plus urea ammonium nitrate fertilizer treatment is referenced as Fe EDDHA + UAN.

no different than the control (Table 2), these trees had the highest mean SPAD values ( $41.2 \text{ SPAD units}$ ;  $P < .0001$ ) and highest plant Fe ( $38.3 \text{ mg kg}^{-1}$ ;  $P < .0001$ ; Figure 1). Fly ash + UAN increased soil Fe concentration 18–46%, though these increases were also no different from the control soil (Table 2). However, chlorophyll and plant Fe in fly ash + UAN treatments increased in leaves according to the application rate in the following manner: SPAD values were highest in the FA DTPA + UAN rate ( $36.9 \text{ SPAD units}$  with a 64% increase in plant Fe), followed by the FA  $44.5 \text{ Mg ha}^{-1}$  + UAN rate (SPAD value 34.7 and 64% increase in plant Fe) and FA  $22.75 \text{ Mg ha}^{-1}$  + UAN rate (SPAD value 32.8 and 43% increase in plant Fe; Figure 1).

The increases in plant Fe with the presumed boost in SPAD values were consistent with carrot when grown at a fly ash landfill site [32]. We have shown in previous studies that both Fe and N play a role in influencing SPAD values for the hybrid OP-367 [25]. Fly ash- and Fe EDDHA + UAN-treated poplars were given the same amount of UAN during the study, holding N constant. Acid-forming fertilizers containing  $\text{NH}_4^+$ , such as UAN, are known to lower the pH of the rhizosphere, making Fe and other microelements more plant-available in alkaline soils [33–35]. So the addition of UAN probably had a role in increasing plant Fe and SPAD values in the Fe EDDHA- and fly ash-treated trees.

Biosolids, on the other hand, significantly improved soil Fe contents by 275–700% above the control soil ( $P < .0001$ ; Table 2), a response consistent with other studies

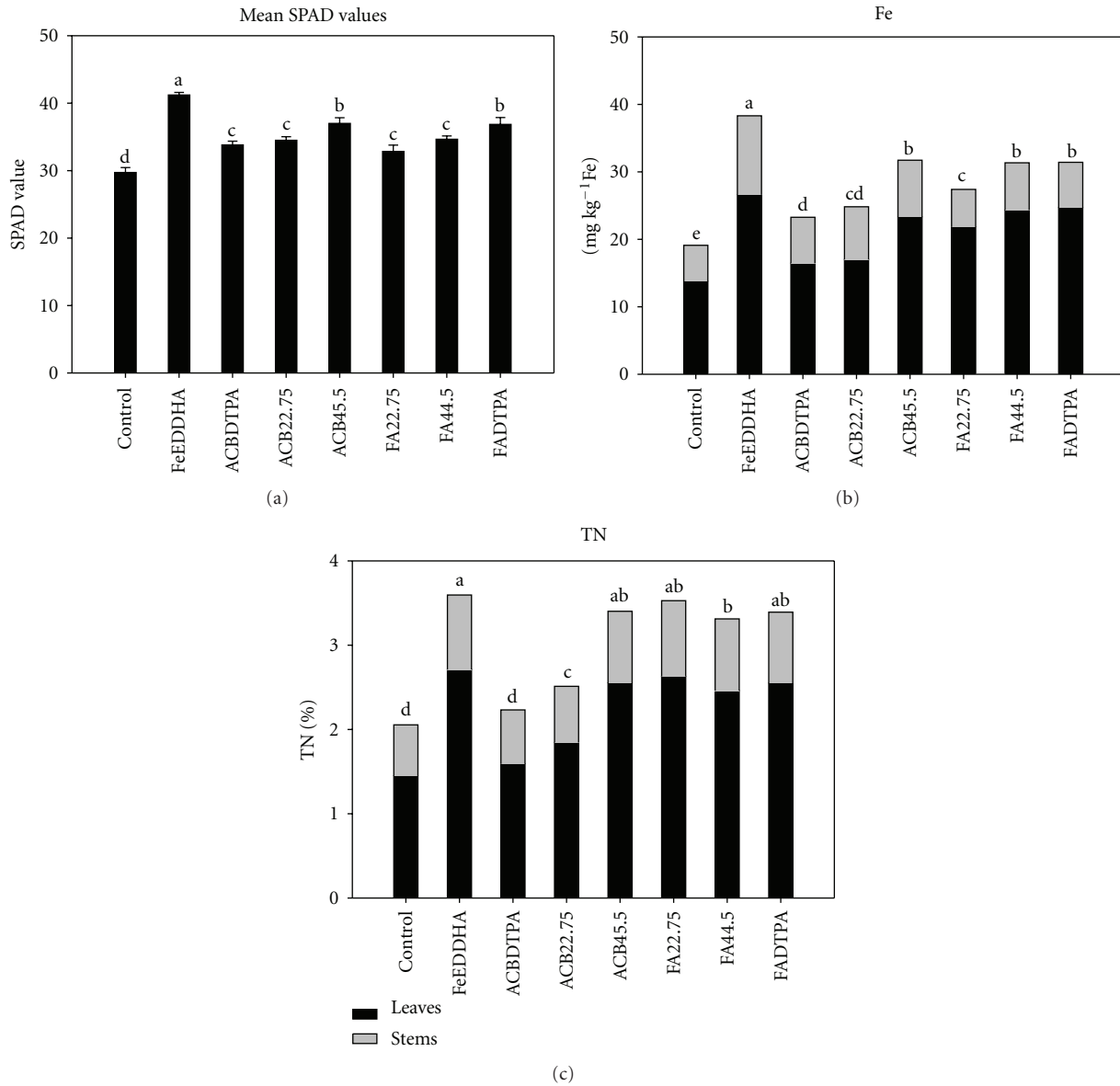


FIGURE 1: SPAD values (a), plant Fe (b), and total plant N (c) of the hybrid poplar clone OP-367 cultivated in soil amended with Fe EDDHA + UAN (urea ammonium nitrate), fly ash + UAN, and composted biosolids. SPAD means are from two measurement periods. Means with the same letter are not significantly different at  $\alpha = 0.05$  level for combined leaf and stem material. Refer to Experimental Design and Statistical Analysis section for treatment codes.

when wastewater has been treated with FeCl salts and the subsequent biosolids are applied to calcareous soils [6, 11]. These trees also had improved leaf greenness and plant Fe (leaves plus stems) in the following way: the ACB DTPA (14 Mg ha<sup>-1</sup>) gave SPAD values of 33.8 and a 22% plant Fe improvement while the ACB 22.75 Mg ha<sup>-1</sup> treatment raised SPAD values to 34.5 and plant Fe by 30% above control trees (Figure 1). Composted biosolids applied at the 44.5 Mg ha<sup>-1</sup> rate resulted in a 66% increase in plant Fe above the control ( $P < .0001$ ). Although statistically lower than the Fe EDDHA+UAN treated trees, SPAD values in the ACB 44.5 Mg ha<sup>-1</sup> trees increased to 37.0, 25% increase in leaf greenness above control trees ( $P < .0001$ ; Figure 1).

Nitrogen and phosphorus bear some mention because, from a producer standpoint, both elements can be expensive farm inputs. Both elements are inherently low in fly ash [36] but can be quite elevated in biosolids. Because UAN applications were held constant for all fly ash and Fe EDDHA trees, as expected, all trees that received UAN had equal and higher soil NO<sub>3</sub>-N when compared to control soil ( $P = .0004$ ; Table 2). Except for the FA 44.5 rate, total leaf N was also equal and highest for trees receiving UAN (Figure 1;  $P < .0001$ ). Only the ACB 44.5 rate equaled this response, increasing NO<sub>3</sub>-N by 43% (Table 2) and plant N by 70% (Figure 1). The two lower composted biosolid rates were similar to the control for soil NO<sub>3</sub>-N, but total plant N

TABLE 3: Biomass results of OP-367 (*P. deltoides* × *P. nigra*) showing significant differences after amending with industrial byproducts. Means with the same letter are not significantly different at the  $\alpha = 0.05$  level.

Treatments <sup>a</sup>	Leaf area (cm <sup>2</sup> )	Leaf dry weight (g)	Stem dry weight (g)	Stem length (cm)
Control	1419 d	15.0 b	14.0 d	124.6 abc
Fe EDDHA + UAN	1552 bc	15.1 b	14.5 bcd	124.0 abc
ACB DTPA	1563 bc	16.4 a	15.6 abc	127.6 ab
ACB 22.75	1584 abc	16.4 a	16.1 a	129.4 a
ACB 44.5	1464 cd	15.2 b	15.6 abc	126.3 ab
FA 22.75 + UAN	1527 bcd	15.2 b	14.3 cd	122.5 bc
FA 44.5 + UAN	1694 a	16.1 ab	14.8 bcd	124.2 abc
FA DTPA + UAN	1590 ab	15.3 ab	14.3 cd	119.5 c
LSD	121	1.1	1.3	5.5
F Value	3.79	2.3	2.84	2.5
Pr > F	0.002	0.0397	0.0133	0.0263

<sup>a</sup>Refer to Experimental Design and Statistical Analysis section for treatment codes.

significantly increased by 27% in ACB-22.75-treated trees (Figure 1). Phosphorus contents of fly ash- and Fe EDDHA-treated soil were no different than the control (Table 2) because these trees received no supplemental P. On the other hand, P was 147–450% higher in soil treated with composted biosolids because Albuquerque uses iron chloride salts to remove P from the waste stream during the treatment process. This fact had some effect on soil salinity (more below).

**3.1.2. Tree Growth.** The following tree growth measurements were unaffected by the treatments: stem diameters (mean 10 mm), root dry weights (mean 9.4 mg kg<sup>-1</sup>), total above-ground dry weights (mean 39.9 mg kg<sup>-1</sup>), and root-to-above ground-biomass ratios (mean 0.31; data not shown). Table 3 presents leaf area, leaf weight, stem weight, root weight, stem length, and stem diameter growth results. All treatments had greater leaf areas ( $P = .0020$ ) except for the FA 22.75 Mg ha<sup>-1</sup>, which compared equally to control trees (Table 3). The greatest leaf areas were from trees treated at the highest two fly ash application rates, followed by trees treated at the ACB 22.75 Mg ha<sup>-1</sup> rate. For leaf dry weight, composted biosolids at the DTPA (14 Mg ha<sup>-1</sup>) and 22.75 Mg ha<sup>-1</sup> rates had the greatest response followed by the FA 44.5 and FA DTPA + UAN treatments ( $P = .0397$ ; Table 3); the Fe EDDHA + UAN and ACB 44.5 Mg ha<sup>-1</sup> rates were no different than the control. For stem dry weight, biosolids at all rates had the greatest response compared to the control trees ( $P = .0133$ ); the control, Fe EDDHA, and fly ash plus UAN-treated trees were no different from one another (Table 3). Stem lengths were similar among the control soil, Fe EDDHA + UAN, composted biosolids, and FA 44.5 Mg ha<sup>-1</sup> trees while the FA 22.75 + UAN and FA

DTPA (74 Mg ha<sup>-1</sup>) rate had a nearly 2 and 4% reduction when compared to control trees ( $P = .0263$ ; Table 3).

Many complex factors influenced growth. Stem weights and stem lengths benefited from the added soil Fe while stem weights, stem lengths, and, to a lesser extent, stem diameters did not seem to benefit from the added NO<sub>3</sub>-N (Table 4). Increasing the soil salinity, pH, and leaf B content also contributed to reduced growth (more below).

### 3.2. Environmental Considerations

**3.2.1. Soil Sodium Adsorption Ratio, Electrical Conductivity, and pH.** Salinity is a concern in our region because we average approximately 200 mm of rainfall per year, which equates to a low salt leaching potential. Any amendment containing high amounts of soluble salts poses the risk of increasing sodic/saline soil conditions. The sodium adsorption ratio (SAR) measures the proportion of Na<sup>+</sup> ions compared to the concentration of calcium Ca<sup>2+</sup> plus Mg<sup>2+</sup> in the saturated paste extract (the higher the SAR value, the more that Na<sup>+</sup> is dominating the soil chemistry). Electrical conductivity (EC) measures total soluble salt content (which can include NaCl but also N, P, Ca, Mg, and other fertilizer salts). An EC above 4 is generally considered the threshold point at which most agricultural crops suffer reduced yields [37].

When compared to the control, SAR values were similar for all treatments except for the ACB 44.5 (4.95 mmol L<sup>-1</sup>), which experienced a 12% reduction in values from the control soil (5.65 mmol L<sup>-1</sup>;  $P < .0001$ ; Table 5). This is explained by the additional Mg and Ca contributions to the soil from the parent biosolids material (Table 1). All of the SAR values were considerably below 13–15 mmol L<sup>-1</sup> which is considered sodic and problematic for agricultural soils. Still, an inverse association was found between increasing SAR values and stem weight ( $r = -0.35$ ;  $P = .0047$ ) and stem length ( $r = -0.26$ ;  $P = .0389$ ; Table 4). Within plants, the ionic balance of Ca, Mg, Na, components of the sodium adsorption ratio in soil, are known to be influenced by nitrogen fertilizer source [38]. The UAN may have had an effect on influencing the components of the SAR test and tree growth given the inverse relationship between NO<sub>3</sub>-N versus growth parameters shown in Table 4.

The Fe EDDHA, FA 22.75, and FA 44.5 + UAN treatments were similar to control soil for EC. However, soil treated at the FA DTPA + UAN (74 Mg ha<sup>-1</sup>) had a significant decrease in conductivity compared to control soil for reasons unknown. On the other hand, composted biosolid-treated soil increased EC by 21–41% above the control soil ( $P < .0001$ ; Table 5). The increase in EC was expected because biosolids are high in soluble salts. In fact, at the ACB 44.5 Mg ha<sup>-1</sup> rate, EC reached 4 dS m<sup>-1</sup>. These levels were below the 5.5 dS m<sup>-1</sup> tolerance limit defined for hybrid poplar [39] and did not appear to affect above-ground growth negatively (i.e.,  $r = 0.27$ ;  $P = .0279$  for the relationship between EC and stem length; Table 4). Although no significant relationship was demonstrated between EC and root weight ( $r = -0.23$ ;  $P = .0656$ ; Table 4), the potential for decreasing this parameter with increasing salinity exists given

TABLE 4: Correlation matrix for 2005 Greenhouse Study showing growth versus soil and foliar parameter. Note: correlation coefficients ( $r$  values) are followed by  $P$  values; \*\* indicates significance  $P < .05$ ; \*\*\* indicates significance  $P < .001$ .

	Leaf area	Leaf Wt.	Stem Wt.	Root Wt.	Stem Lnth.	Stem Dia.
Fe soil	-0.08	0.09	0.42***	-0.11	0.33**	0.11
	0.5197	0.4885	0.0006	0.3987	0.0068	0.4026
NO <sub>3</sub> -N	-0.15	-0.21	-0.45***	-0.08	-0.46***	-0.28**
	0.2364	0.0983	0.0002	0.5246	0.0001	0.0234
pH	-0.04	-0.16	-0.54***	-0.18	-0.41***	-0.14
	0.7443	0.1990	<0.0001	0.1481	0.0007	0.2756
SAR	0.16	-0.04	-0.35**	-0.14	-0.26**	-0.16
	0.2213	0.7499	0.0047	0.2592	0.0389	0.1945
EC	-0.14	0.08	0.24	-0.23	0.27**	-0.04
	0.2900	0.5548	0.052	0.0656	0.0279	0.7498
B leaves	0.21	-0.08	-0.20	0.14	-0.44***	0.03
	0.0982	0.5331	0.1198	0.2605	0.0003	0.7922

TABLE 5: Soil sodium adsorption ratio (SAR), electrical conductivity (salinity), and pH of a Doak sandy loam amended with industrial byproducts.

Treatments <sup>a</sup>	SAR (mmol L <sup>-1</sup> )	EC (dS m <sup>-1</sup> )	pH saturated paste
Control	5.65 ab	3.24 d	8.62 b
Fe EDDHA + UAN	5.78 a	3.17 d	8.66 ab
ACB DTPA	5.42 b	3.47 c	8.58 bc
ACB 22.75	5.55 ab	3.77 b	8.50 cd
ACB 44.5	4.95 c	4.04 a	8.44 d
FA 22.75 + UAN	5.73 a	3.10 de	8.64 b
FA 44.5 + UAN	5.62 ab	3.16 d	8.67 ab
FA DTPA + UAN	5.67 a	3.03 e	8.76 a
Mean	5.54	3.37	8.6
LSD	0.24	0.18	0.1
$F$ Value	9.32	31.71	7.33
$Pr > F$	<0.0001	<0.0001	<0.0001

<sup>a</sup>Refer to Experimental Design and Statistical Analysis section for treatment codes.

that containers were not leached. In later field plot studies, we found no salinity increases in plots amended with composted biosolids at 44 Mg ha<sup>-1</sup> [40]. In the latter study, a total of 983 mm of water (irrigation + rainfall) was applied during the second growing season alone, which provided sufficient leaching potential; all biosolids field plots never exceeded an EC of 1 dS m<sup>-1</sup> when sampled at a depth of 30 cm.

Another concern is applying an amendment that may have the potential to raise soil pH in already calcareous conditions. The pH ranged from 8.4 in soil treated at the ACB 22.75 Mg ha<sup>-1</sup> rate to 8.8 in the FA DTPA + UAN-treated soil (equivalent of 74 Mg ha<sup>-1</sup>); the control soil had a pH of 8.6 ( $P < .0001$ ; Table 5). Low S-containing western US lignite coals typically produce alkaline ash [9, 41], which explains the pH increase in accordance with increasing application

rate of fly ash. The pH increase was related to a reduction in stem weights ( $r = -0.54$ ;  $P < .0001$ ) and stem length ( $r = -0.41$ ;  $P = .0007$ ), and the general trend was that as pH increased, leaf area, leaf weight, root weight, and stem diameter decreased (Table 4).

Composted biosolids, when applied at the 22.75 Mg ha<sup>-1</sup> rate, decreased pH below the control soil to 8.4. As salt concentration increases, soluble cations, such as Ca<sup>2+</sup> and Mg<sup>2+</sup>, replace acidic exchangeable cations (H<sup>+</sup> and Al<sup>3+</sup>) in the soil solution, lowering the pH of the soil extract solution [35, 43, 44]. The reduction in pH was probably related to the soluble salt content of the material. In field studies with the same biosolid application rates as in this study, under a leaching fraction, soil pH was equal among biosolid treatment [40].

**3.2.2. Other Environmental Considerations.** The following metals were analyzed in soils from acid digests: Cr, As, Ag, Se, Pb, Cd, and Ba. Arsenic, Ag, and Se were not detected in either soils or plants. Lead, Cr, and Cd also did not increase in soils in either study, averaging 7.71 mg kg<sup>-1</sup> for Cr, 5.08 mg kg<sup>-1</sup> for Pb, and 0.16 mg kg<sup>-1</sup> for Cd (Table 6). These levels were below USEPA (Part 503 Rule) and European Union (Directive 86/278/EEC) regulations for heavy metal loading rates for biosolids applied to agricultural lands [42].

Boron and Ba, however, did present potential environmental concerns. Boron increased in the leaves of fly ash + UAN amended poplars by 23% (22.75 Mg ha<sup>-1</sup> rate), 45% (44.5 Mg ha<sup>-1</sup> rate) to 85% (rate equivalent to 74 Mg ha<sup>-1</sup>) (Figure 2). Although B is a micronutrient needed by plants in trace amounts, toxicity symptoms and decreased crop yields result from the application of unweathered fly ash [9, 41, 45, 46]. At the highest fly ash application rate, leaf B reached 93.6 mg kg<sup>-1</sup>, which began to approach toxicity levels (above 141 mg B kg<sup>-1</sup> dwt) defined for OP-367 by Bañuelos et al. [47]. Indeed, the elevated B levels found in the leaves of fly ash-treated trees inversely correlated with stem lengths ( $r = -0.44$ ;  $P = .0003$ ; Table 4). As B moves

TABLE 6: Soil Cr, Pb, Ba, and Cd levels in a Doak sandy loam amended with industrial byproducts.

	Cr (mg kg <sup>-1</sup> )	Pb (mg kg <sup>-1</sup> )	Ba (mg kg <sup>-1</sup> )	Cd (mg kg <sup>-1</sup> )
Literature				
U.S. EPA 40 CFR 503 Rule <sup>a</sup>	—	300.0	—	39.00
European Union limit values <sup>b</sup>	—	750–1,200	—	20–40
EU proposed	1000.0	750.0	—	10.00
Treatments <sup>c</sup>				
Control	7.28 a	5.19 a	97.23 e	0.16 a
Fe EDDHA + UAN	7.60 a	5.19 a	98.18 de	0.17 a
ACB DTPA	8.34 a	5.07 a	105.83 cd	0.16 a
ACB 22.75	7.80 a	5.05 a	102.33 de	0.15 a
ACB 44.5	7.92 a	5.12 a	100.84 de	0.17 a
FA 22.75 + UAN	7.68 a	5.22 a	111.48 bc	0.17 a
FA 44.50 + UAN	7.51 a	4.93 a	119.25 b	0.17 a
FA DTPA + UAN	7.53 a	4.86 a	137.63 a	0.17 a
Mean	7.71	5.08	109.10	0.16
LSD	NS	NS	8.46	NS
F Value	0.34	1.02	20.93	0.95
Pr > F	0.9302	0.4274	<.0001	0.48

<sup>a</sup>Pollutant concentration limits and loading rates for land application in the United States.

<sup>b</sup>European Union limit values for concentrations of heavy metals in biosolids for use on land [42].

<sup>c</sup>Refer to Experimental Design and Statistical Analysis section for treatment codes.

easily with irrigation waters, accumulation of B may have been mitigated if the containers were leached. Thus, boron accumulations to toxic levels present an environmental concern for agricultural land application of fly ash to our soils if not leached regularly and managed carefully.

Barium increased 7–42% in fly ash-amended soil (Table 6). As a consequence, the stem and leaf Ba levels combined increased 60–110% (46.3–60.8 mg kg<sup>-1</sup>) compared to control trees (28.9 mg kg<sup>-1</sup>;  $P < .0001$ ; Figure 2). The increase of Ba is not uncommon when fly ash is applied to land [46]. With regards to Ba, it is difficult to assume environmental safety because we did not analyze for potentially toxic forms (barium carbonate, barium chloride, and barium acetate) [48].

As for the composted biosolids material or the composted biosolid/soil mixtures, we did not analyze for the presence of antibiotics and/or personal care products. Recent attention to these persistent organic constituents shows that biosolids, especially when not composted, may impart potential antibiotic resistance in soil micro-organisms or molecules from personal care products may exhibit hormonal effects on aquatic organisms [14, 49]. Although the risk factors associated with antibiotics and personal care products can be mitigated by composting [49, 50], an analysis for these constituents is essential to strengthening our knowledge of the material used in this study and

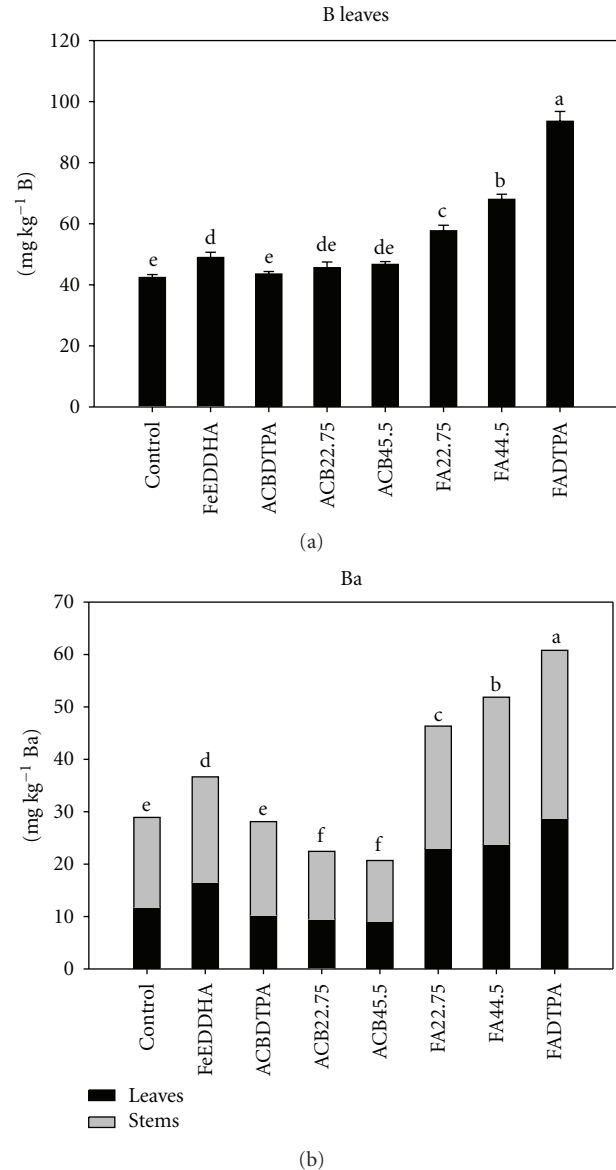


FIGURE 2: Leaf B (a) and leaf+stem Ba (b) of the hybrid poplar clone OP-367 cultivated in soil amended with Fe EDDHA + UAN (urea ammonium nitrate), fly ash + UAN, and composted biosolids. Means with the same letter are not significantly different at  $\alpha = 0.05$  level for combined leaf and stem material. Refer to Experimental Design and Statistical Analysis section for treatment codes.

in developing a future comprehensive environmental risk assessment when composted biosolids might be considered for agricultural land application.

#### 4. Conclusions

The Fe EDDHA + UAN had the highest SPAD values and plant Fe (stem + leaves). This is an expensive synthetic fertilizer input with 6% plant-available iron. In comparison, fly ash plus UAN significantly increased SPAD values and plant Fe in hybrid poplars even though soil Fe remained



statistically similar to the control soil. The trend in SPAD values and plant Fe generally followed fly ash application rate—the higher the rate, the greater the response. Despite the increase in soil pH from fly ash additions, which would imply even lower solubility of micronutrients, and the fact that N from UAN remained constant among the fly ash (and Fe EDDHA treatments), the uptake of Fe in fly ash-amended poplars was likely related to an acidulation of the rhizosphere from UAN applications which made Fe more available just at the root/soil interface. Leaf area was greatest in hybrid poplar trees grown in soil amended with fly ash treatments, but growth was similar to the control for all other biometric parameters. The highest plant B accumulations occurred in the fly ash + UAN treatments, especially at the fly ash rate equivalent to 74 Mg ha<sup>-1</sup>. Increasing leaf B was inversely associated with stem length in these trees. Stem and leaf Ba was also highest in trees grown in fly ash-treated soil. Potentially toxic forms of Ba found in fly ash were not measured, which warrants further investigation. Finally, the fact that NO<sub>3</sub>-N was inversely related to growth raises the need for us to conduct a UAN exclusion study before a definitive recommendation can be made concerning fly ash applications to agricultural lands.

Biosolids significantly increased soil Fe and P in all treatments, and NO<sub>3</sub>-N only at the 44.5 Mg ha<sup>-1</sup> rate. Although lower than the Fe EDDHA treatment, composted biosolid-treated trees had SPAD and plant Fe values significantly increase in proportion to application rate, showing that composted biosolids could supply plant-available Fe to trees growing on an alkaline soil. Likewise, total plant N increased, but only the 44.5 Mg ha<sup>-1</sup> treatment equaled the response of UAN-treated trees. Trees grown under composted biosolids applied at the DTPA (14 Mg ha<sup>-1</sup>) and 22.75 Mg ha<sup>-1</sup> rates generally had the highest growth (stem dry weight, stem length, and leaf dry weight). Saturated paste extracts of the byproducts demonstrated that composted biosolids had the most potential for increasing soil salinity due to their complex mixtures of soluble salts. Soil treated with the 44.5 Mg ha<sup>-1</sup> rate had an EC of 4 dS m<sup>-1</sup> which may explain why the lower biosolids application rates generally had greater tree growth. An inverse relationship between increasing salinity and decreasing root dry weight was shown, but salinity increases were below tolerance levels defined for hybrid poplar, and salts would be flushed from the root zone if leaching of containers was allowed in this study.

Overall, trees grown under biosolids generally exhibited the greatest response in regards to growth, soil, and plant Fe, N, and P increases *without* the need to provide supplemental N nutrition in the form of UAN. Other benefits not reported included an increase in Zn, Cu, and Mn in trees grown with composted biosolid amended soil. Amendment/soil mixtures showed little potential for environmental hazard in terms of heavy metal increases. It appears that a onetime application of the ACB 22.75 Mg ha<sup>-1</sup> rate is sufficient to supply plant-available Fe and growth benefits to hybrid poplar seedlings without the risk of increasing salinity in *unleached* circumstances. The beneficial recycling of nutrients from biosolids to agricultural crops produced on a calcareous soil is feasible.

An analysis of the Albuquerque composted biosolids for antibiotics and personal care products would add value to future field plot studies.

## Acknowledgments

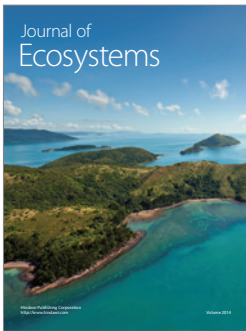
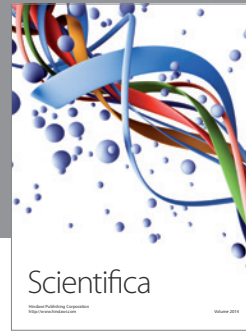
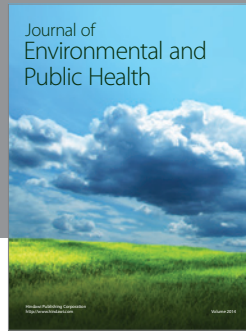
The authors would like to thank the U.S. Department of Energy National Energy Technology Laboratory and the Combustion Byproducts Recycling Consortium for partial funding of this paper. The authors wish to thank Steven Glass, City of Albuquerque, for donating the composted biosolids and Bruce Salisbury, Arizona Public Services, for the fly ash.

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