

# RESEARCH EXTENSION NOTE No. 14—Feb 2021

## ENHANCING MINE AND ENERGY CROP SOILS TO PROMOTE WILLOW (SALIX MIYABEANA) GROWTH USING ASH AND BIOSOLIDS: A GREENHOUSE STUDY

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# Enhancing Mine and Energy Crop Soils to Promote Willow (*Salix miyabeana*) Growth Using Ash and Biosolids: A Greenhouse Study

#### Abstract

Addition of wood ash to soil can supply plant nutrients and reduce soil acidity. We assessed the growth of willow in two soils amended with wood ash generated at bioenergy facilities. As nitrogen is limiting in ash, we also investigated the addition of biosolids (1% by mass, dry basis), a common residual of municipal wastewater treatment. A sandtextured soil from a bioenergy plantation received greater application of ash(0, 1, 2%)dry weight basis) than the second soil, a loamtextured mine overburden (0, 0.5 and 1%), due to the acidic nature of the former soil (pH 4.7) vs the latter (pH 7.6). Compared to a non -amended control, ash-only treatments did not result in significantly greater willow biomass over the 4-month study. However, the addition of biosolids did result in significantly

greater above-ground and total biomass over treatments that did not include biosolids. The combined application of UNBC ash (1%) and biosolids (1%) to the sand-textured soil produced the greatest total biomass measured in the two trials, but this treatment was not significantly different than other biosolids treatments. The 1% ash application to the mine loam soil created very basic conditions (pH > 9), likely having a detrimental impact on willow growth. Overall, these results suggest that ash composition can have different effects on the growth of willow, and that interaction effects with other amendments are important when optimizing amendment mixtures. Biosolids addition benefited both soils but ash addition only benefited the acidic plantation sand.

## Introduction

Wood wastes from harvesting, milling and manufacturing are increasingly being used as biomass fuel for bioenergy production (Natural Resources Canada 2020). In addition to energy, these high-temperature combustion processes produce ash, typically considered a waste residual. Approximately 1-5% of the dry weight of woody biomass becomes ash, most of which is currently landfilled or stockpiled in Canada (Pitman 2006, Hannam et al. 2018). However, wood ash may prove to be an effective soil amendment on selected sites in the British Columbia (BC) central Interior, as ash is a source of many plant nutrients and may amend soil acidity (Pitman 2006, Adler et al. 2008, Helle et al. 2009, Domes et al. 2018). Land application of bioenergy ash has the potential to have positive economic and ecological benefits, especially in areas impacted by resource extraction.

Little research about the land application of bioenergy ash has been conducted in Canada, especially in the context of western silvicultural applications and short-rotation bioenergy plantations (Pitman 2006, Marron 2015, Domes et al. 2018, Hannam et al. 2018). Chemical properties of ash differ depending on the species of wood burned and whether the biomass originates from stem, bark or foliage (Hakkila 1989, Someshwar 1996, Pitman 2006). Temperature and combustion efficacy also influence the chemical composition of ash (Etiégni and Campbell 1991, Pitman 2006).

One vital nutrient limiting in ash, but essential for plant growth, is nitrogen. Most nitrogen is expelled as gas during biomass combustion, reducing N quantity in ash (Steenari and Lindqvist 1997, Demeyer et al. 2001, Pitman 2006). By combining ash with a supplement relatively rich in nitrogen, such as biosolids, its significance as a fertilizer can be enhanced (Cavaleri et al. 2004, Adler et al. 2008). Biosolids are stabilized municipal sewage sludges and are relatively high in nutrients and organic matter. Application of biosolids to soil can enhance nutrient supply, improve soil physical properties and may stimulate soil microbial activity (Sylvis 2008, CCME 2012). Regulations and guidelines for the land application of wood ash vary throughout Canada (Hannam et al. 2016). In British Columbia, land application of fly ash derived from wood combustion is regulated under the *Code of Practice for Soil A mendments;* land application of biosolids is regulated under the *Organic Matter Recycling Regulations* (Government of BC 2002, 2007).

Soils affected by anthropogenic disturbances and resource management practices, such as mining and forestry, can become inhospitable for plants, due to soil compaction, low soil organic matter content and/or poor nutrient supply (Delong et al. 2012). Compaction, from forestry machinery for instance, can reduce root growth by impeding access to water, air and nutrients in the soil (Weil and Brady 2017). Topsoil removal and the mixing of multiple horizons of soil, which often occurs in mining, may lead to low soil organic carbon (SOC) contents (Shukla et al. 2004). Reclaiming mine soils by improving SOC, decreasing bulk density and improving water-holding capacity may allow plants, including tree species, to establish more readily (Shukla et al. 2004; Delong et al. 2012).

Fast-growing trees, like willow and poplar, are valued for their accelerated growth and are cultivated for use in short-rotation tree plantations (Ceulemans et al. 1996, Marron 2015). These trees are commonly propagated in bioenergy plantations through stem cuttings collected during the dormant season and planted in the spring (Kopp et al. 1997). Generally, after the first year of growth, the cuttings are trimmed to within 2-4 cm above-ground to encourage coppicing in the following growing season (Kopp et al. 1997, Abrahamson et al. 2002). Biomass harvests can occur as soon as 3-5 years after planting (Kopp et al. 1997), although the short rotation of these trees can lead to the relative rapid removal of nutrients from plantation soils (Adegbidi et al. 2001). The application of biomass ash and/or biosolids to bioenergy plantation soils may off-set nutrient removal and soil

acidification processes. Few studies have examined the combined effects of biosolids and ash amendments on the growth of species typically used for short-rotation coppice (Cavaleri et al. 2004, Adler et al. 2008, Marron 2015).

Our main objective was to determine whether the application of waste residuals, in this case, bioenergy wood ash and municipal biosolids, would impact shoot (i.e. new leaves and stems) and root biomass production of willow. Here we report on two concurrent 4-month greenhouse trials differentiated by soil type (sandy bioenergy plantation soil and loam mine soil). Three different ashes were examined (two for each trial). We tested different rates of ash application, with and without biosolids addition. Results should provide insights into the use of these residuals in bioenergy plantation soils and in the reclamation of mine soils.

### **Materials and Methods**

#### **Plant Material**

Willow stem cuttings were harvested on October 3, 2012 from a bioenergy plantation located at the Pacific Regeneration Technologies Inc. (PRT) nursery south of Prince George, in Red Rock, BC under the guidance of the PRT greenhouse manager (O. Bonnefoy). The hybrid willow (SX64 or Salix miyabeana Seemen) is a species native to Japan (Tharakan et al. 2004). Whips, 10 to 15 mm in diameter, were collected from first-year growth because younger stem cuttings are more likely to take root (FAO 1980). These harvested branches were trimmed into 20 cm stakes (Tharakan et al. 2004, Bourret et al. 2009), bundled and submerged into enough water to cover the basal portion. Stake tops were loosely wrapped with plastic wrap to prevent desiccation during 4week cold storage prior to use.

#### **Soil and Amendments**

Soils were selected on the basis of having undergone intensive disturbance (mine overburden) or intensive crop production (bioenergy plantation soil), and were in need of enhancement. The loam overburden soil was collected (October 11, 2012) close to the open pit at the Gibraltar copper-molybdenum mine near McLeese Lake, BC (Taseko Mines Ltd), where Gibraltar staff (J. Evans) facilitated the collection. Sandy soil was collected (October 15. 2012) from a fallow area of a short-rotation plantation at the Pacific Regeneration Technologies (PRT) Nursery, located in Red Rock, BC. Soils were passed through a 4 mm sieve (no. 5 mesh) to remove large fragments; subsamples were passed through a 2 mm (no. 10 mesh) prior to chemical analysis. Three wood ashes were utilized for this study. UNBC ash (mix of bottom and fly ash) was collected (July 13, 2012) from the Nexterra biomass gasifier ash bin (located inside the UNBC Bioenergy plant, Prince George campus). Bottom ash was collected (April 27, 2012) from CPLP (Canfor Pulp Limited Partnership) boiler #2 at the Canfor Pulp and Paper mill (P.G. Pulp) in Prince George, BC. Bottom ash from the PRT Red Rock bioenergy plant was collected (October 15, 2012) from an outdoor stockpile located next to the bioenergy facility. Biosolids (anaerobically digested sewage sludge) were collected (Oct 15, 2012) from the Prince George Lansdowne Wastewater Treatment Plant.

Each soil substrate was homogenized prior to use in this study. The BC Ministry of Environment (MOE) Analytical Laboratory (Victoria, BC) characterized the two soils, three ashes and biosolids (Table 1 and 2). Soil pH measurements were determined using a 1:2 solid-toliquid (g solid: mL deionized water) ratio (Kalra and Maynard 1991). A 1:5 solid-toliquid ratio was used for pure ash and pure biosolids. Gravimetric moisture contents of all starting materials were determined to adjust application rates to an equivalent dry weight basis. Gravimetric moisture contents for soil and ashes were determined by oven drying at 105°C for 24 hours (Kalra and Maynard 1991). Moisture content for the biosolids were determined by heating to 70°C for 48 hours to minimize the loss of volatile organic compounds (Kalra and Maynard 1991).

Table 1. Initial properties of sand and loam soils (n=3) prior to amendment additions (mean $\pm$ standard
deviation). Concentrations are expressed on a dry-weight basis. See Appendix for additional elemental
concentrations.

	Sand	Loam
pH (1:2 in water)	4.7 ± 0.1	7.6 ± 0.2
Total C (%) <sup>1</sup>	1.5 ± 0.1	0.4 ± 0.01
Inorganic C (%) <sup>1</sup>	0.1 ± 0.02	0.1 ± 0.01
Effective CEC (cmol <sub>c</sub> kg <sup>-1</sup> ) <sup>2</sup>	$2.6 \pm 0.4$	10.9 ± 0.4
Electrical Conductivity Sat. Paste (mS cm <sup>-1</sup> )	0.4 ± 0.01	3.1 ± 0.1
Bray Available P (mg kg <sup>-1</sup> )	376 ± 21	$4.9 \pm 0.2$
Sand (%)	92.4 ± 1.3	46.5 ± 0.7
Silt (%)	7.2 ± 0.7	34.6 ± 0.7
Clay (%)	$0.4 \pm 0.7$	19.0 ± 0.0
Macronutrients (%)		
Total Ca <sup>3</sup>	0.4 ± 0.01	1.7 ± 0.03
Total K <sup>3</sup>	$0.3 \pm 0.002$	0.5 ± 0.07
Total Mg <sup>3</sup>	$0.5 \pm 0.005$	0.8 ± 0.01
Total N <sup>1</sup>	0.1 ± 0.003	0.02 ± 0.001

Total S<sup>1</sup>

Total P<sup>3</sup>

<sup>1</sup> determined by dry combustion (Dumas method)

<sup>2</sup> effective CEC determined by BaCl<sub>2</sub> (Kalra and Maynard 1991)

<sup>3</sup> determined by ICP-OES following acid (conc. HNO<sub>3</sub>-HC1) microwave digestion, EPA Method 3051A

 $0.2 \pm 0.002$   $0.1 \pm 0.001$ 

 $0.02 \pm 0.001$   $0.3 \pm 0.003$ 

Table 2. Chemical properties (n=3), including macronutrients, for the ashes and biosolids used in this study (mean  $\pm$  standard deviation). Concentrations are expressed on a dry-weight basis. See Appendix for additional elemental concentrations.

	PRT Ash	UNBC Ash	CPLP Ash	Biosolids
pH (1:5 in water)	9.2 ± 0.1	11.9 ± 0.1	11.1 ± 0.1	8.0 ± 0.1
CaCO <sub>3</sub> Equivalent (%)	13.5 ± 0.5	46.3 ± 1.3	28.3 ± 0.3	not done
Total C (%) <sup>1</sup>	4.9 ± 0.1	6.7 ± 0.5	58.8 ± 2.6	39.5 ± 0.2
Inorganic C (%) <sup>1</sup>	0.5 ± 0.1	1.9 ± 1.0	3.3 ± 0.3	not done
EC (mS/cm) 1:5 (ash) or sat. paste	0.5 ± 0.01	10.1 ± 0.5	5.6 ± 0.1	10.2 ± 0.2
Macronutrients (%)				
Total Ca <sup>2</sup>	6.1 ± 0.1	18.6 ± 1.1	9.8 ± 0.1	2.9 ± 0.02
Total K <sup>2</sup>	$2.7 \pm 0.02$	5.1 ± 0.3	$2.7 \pm 0.03$	0.2 ± 0.01
Total Mg <sup>2</sup>	1.3 ± 0.01	2.7 ± 0.1	1.1 ± 0.01	$0.4 \pm 0.002$
Total N <sup>1</sup>	0.04 ±0.001	$0.04 \pm 0.001$	$0.2 \pm 0.003$	5.5 ± 0.1
Total P <sup>2</sup>	0.5 ± 0.01	0.8 ± 0.1	0.5 ± 0.01	1.8 ± 0.02
Total S <sup>1</sup>	0.04 ± 0.002	0.2 ± 0.01	0.4 ± 0.01	1.3 ± 0.2

<sup>1</sup> determined by dry combustion (Dumas method)

<sup>2</sup> determined by ICP-OES following acid (conc. HNO<sub>3</sub>-HC1) microwave digestion, EPA Method 3051A

Both soils were very low in total carbon and nitrogen. The PRT sand was quite acidic (pH 4.7), while the Gibraltar loam was relatively pH neutral (pH 7.6). Of the three ashes, UNBC ash was the most alkaline (pH 11.9) and exhibited the greatest calcium carbonate equivalence (46.3%). The CPLP ash was rich in total carbon (58.8%) compared to the PRT and UNBC ashes (4.9 and 6.7%, respectively); much of the CPLP ash resembled charcoal. Total nitrogen content in biosolids was 5.5%, which was 27- to 135-times greater than nitrogen in the biomass ashes used in this study. Total sulphur in the loam was high relative to forest soils in northcentral BC (Arocena and Sanborn 1999, Sanborn et al. 2005, Domes et al. 2018) but similar to other BC mine soil (Carson et al. 2014). Available P was relatively high in the sand, likely due to previous fertilization events at the plantation. See Appendix for additional characterization data (Tables A1 and A2).

#### **Greenhouse Trials**

The PRT sand and Gibraltar loam trials were initiated the first week of November 2012 in the I.K. Barber Enhanced Forestry Laboratory (EFL) at the UNBC Prince George campus. Each trial employed a randomized design examining two factors: 3 ash treatments x 2 biosolids treatments x 6 replicates. Ash application rates (sand: 0, 1.0 and 2.0%; loam: 0, 0.5 and 1.0% dry weight basis) differed in the two trials as initial soil pH was 4.7 in sand, and 7.6 in the loam (Table 1). Each trial utilized two kinds of ash: PRT and UNBC ashes in sand trial; CPLP and UNBC ashes in the loam trial. Logistical constraints prevented the use of three ashes in both trials. The biosolids application rate (1%, dry weight basis) was equivalent to a low to medium application rate for silvicultural applications in north-central BC (Sylvis 2008). The growth trial period varied slightly between the sand trial (118-120 days) and loam trial (120-122 days) because of staggered planting and harvesting days (Gilbert 2013).

Amendments were mixed into soils before adding to PVC pots (D40H Deepots -Stuewe and Sons, Inc., Oregon), which were 7 cm in diameter, had a capacity of 656 cm<sup>3</sup> and a depth of 25 cm. The bottom ~7 cm of each pot was filled with inert substrate materials to conserve soil and minimize soil loss through drainage holes. For the loam trial, small stones (~1-2 cm diameter) previously removed from the soil were used. For the sand trial, Hydrotron<sup>Tm</sup> expanded clay pellets (~ 1 cm diameter) were used. Six pots of each amendment treatment were placed into trays with a capacity for 20; pots were 1.5 cm apart. Cuttings were placed 10 to 12 cm into soil with a minimum of two viable buds remaining in the aboveground portion (6-8 cm).

The 4-month growth period employed 16 h supplemental light per day (400W HP sodium lamps; 160 cm height). Temperature was 16°C during non-lighted conditions (i.e., at night), and 22°C during the day. Trays were turned 90 degrees and rotated clockwise along the bench once a week to minimize effects due to uneven lighting or temperature. As stems gained biomass, pots were gradually spread further apart (to 6 pots per tray) to minimize light limitations. Gilbert (2013) provides details of watering scheme. Aphids, thrips and spider mites were observed on willow seedlings and were dealt with using methods detailed in the Appendix. See Figure 1 and 2 for selected images of the greenhouse trial.



Figure 1. Willow biomass in selected containers approximately 2 weeks into the sand trial. Photo taken by N. Gilbert on November 15, 2012.



Figure 2. Willow biomass in selected containers approximately 13 weeks into the sand trial. Photo taken by N. Gilbert on February 4, 2013.

After 4 months, live green shoot biomass was trimmed close to each cutting and placed in paper bags; these were oven-dried at 70°C for 48 hours and subsequently weighed. This biomass was added to dead leaves collected and dried earlier in the growth period. Pot contents were placed in a tray, homogenized and a representative soil sample was collected for subsequent soil pH analysis. Cuttings and roots were gently washed by hand to remove soil. Roots were removed from cuttings, dried at 70°C for 72 hours and weighed. Root and shoot biomass weights were combined to arrive at total biomass.

Soil pH was determined at UNBC for soil samples collected from three pots of each treatment (i.e. n = 3) at the time of experimental set up (Time 0) and at time of harvest using methods described above.

#### **Statistical Analyses**

Willow growth characteristics (dry shoot biomass, root biomass, total biomass, and root to shoot ratio) and soil pH were analyzed using linear 2-way mixed effects models with soil (PRT sand or Gibraltar loam) included as a random factor. Ash was included as a fixed factor and was characterized as "UNBC" or "Other" (PRT or CPLP), and partitioned into "low" or "high" application rates, while the presence or absence of biosolid was included as the second fixed factor. Tukey's HSD tests were performed *a posteriori* to determine differences between treatment means (p < 0.05). Statistical analyses were performed using the nlme (Pinheiro et al. 2020) and agricolae (de Mendiburu 2020) packages in R, a language and environment for statistical computing (version 3.6.0, R Core Team 2019). Each model was fit using log-likelihood maximization.

## Results

Overall, shoot biomass (Fig. 3) and total biomass (Fig. 4) of willow were significantly greater (p < 0.001) in treatments that received biosolids, compared to treatments that did not receive biosolids (Table 3). Biosolids had no significant effect (p > 0.05) on root biomass or root:shoot ratios (Table 3). Ash by itself had no significant effect on willow growth over non-amended controls (Table 3). Although the greatest mean shoot mass and greatest mean total biomass were observed for the combined biosolids-low UNBC ash treatment (Fig 1 and 2), these means were not significantly different from other treatments that utilized biosolids. Overall, willow biomass was greater in the PRT sand trial than in the loam trial (Table A4).

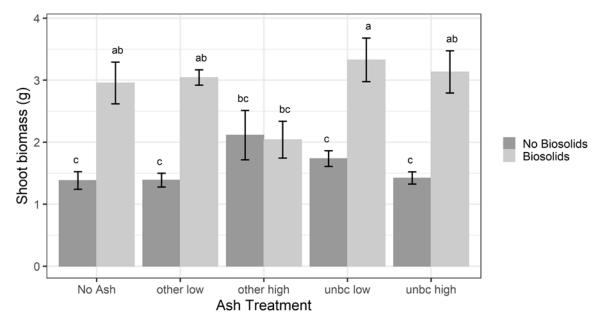


Figure 3. Mean willow shoot biomass ( $\pm$  standard error) for the two trials combined (dry weight basis). Ash is designated as being "UNBC" or "Other", the latter representing PRT ash for the PRT sand trial and CPLP ash for the Gibraltar loam trial. Application rates varied with trial: Low (0.5% for Gibraltar, 1% for PRT trial); High (1% for Gibraltar, 2% for PRT trial). Treatments sharing the same lower case letter are not significantly different from each other (p < 0.05).

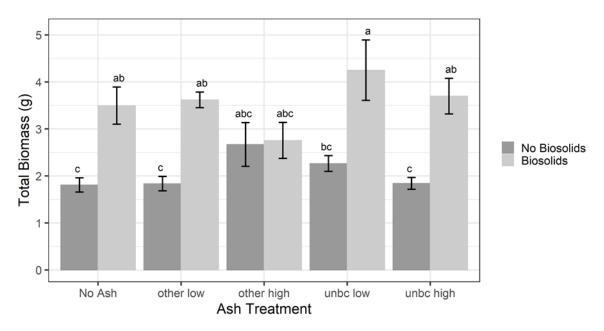


Figure 4. Mean willow total biomass ( $\pm$  standard error) for the two trials combined (dry weight basis). Ash is designated as being "UNBC" or "Other", the latter representing PRT ash for the PRT sand trial and CPLP ash for the Gibraltar loam trial. Application rates varied with trial: Low (0.5% for Gibraltar, 1% for PRT trial); High (1% for Gibraltar, 2% for PRT trial). Treatments sharing the same lower case letter are not significantly different from each other (p < 0.05).

Table 3. Two-way ANOVA results for ash and biosolids effects on seedling biomass components, and soil pH, with trial ("Gibraltar loam" or "PRT sand") included as a random factor. Ash application rates varied with trial: Low (0.5% for Gibraltar, 1% for PRT trial); High (1% for Gibraltar, 2% for PRT trial).

	Asl	h	Bioso	olid	Ash*Bioso acti		Random	Effect
Response varia- ble	F ( <i>4,50</i> )	p-value	F ( <i>1,50</i> )	p-value	F ( <i>4,50</i> )	p-value	Plot StDev.	Residual StDev.
Shoot Biomass	1.463	0.219	82.93	<0.001	5.038	0.001	0.339	0.728
Roots Biomass	1.334	0.262	4.868	0.03	0.4	0.808	0.107	0.434
Total Biomass	1.608	0.178	58.05	<0.001	2.826	0.029	0.455	0.995
Root:Shoot Ratio	1.537	0.197	6.745	0.011	2.455	0.05	<0.001	0.147
Soil pH Time 0	126.7	<0.001	12.38	0.001	0.595	0.668	1.013	0.249
Soil pH Final	14.19	<0.001	0.506	0.48	1.189	0.327	0.611	0.535

Initial soil pH was significantly greater (p < 0.001) in treatments receiving ash or biosolids relative to non-amended controls (Table 3). Although there was a tendency for UNBC ash treatments to have greater soil pH at the beginning (Fig. 3) and end of the trials (Fig. 4), these trends were not significantly (p > 0.05) different from the other ash treatments. pH response of each soil to the various amendments are summarized in Table A3. Addition of UNBC ash to the sand (at both rates, with and without biosolids), contributed to a relatively neutral

soil pH throughout the growing period. While PRT ash addition increased soil pH over the control, the rates of PRT ash application used for this trial were not sufficient to increase the soil pH to a neutral 7. The addition of 1% UNBC ash resulted in a very high pH in the loam soil (> pH 9). Soil pH in the ash-treated loam tended to decrease over the course of the study, with UNBC ash treatments generally exhibiting a greater pH decrease than the CPLP ash treatments.

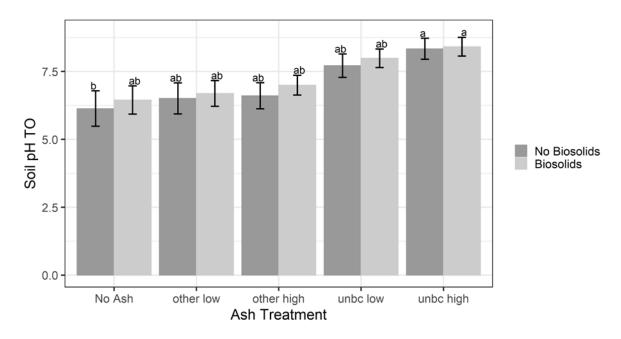


Figure 5. Mean soil pH ( $\pm$  standard error) for the two trials combined at the initiation of the study (time 0). Ash is designated as being "UNBC" or "Other", the latter representing PRT ash for the PRT sand trial and CPLP ash for the Gibraltar loam trial. Application rates varied with trial: Low (0.5% for Gibraltar, 1% for PRT trial); High (1% for Gibraltar, 2% for PRT trial). Treatments sharing the same lower case letter are not significantly different from each other (p < 0.05). See Table A3 for soil pH data separated by trial.

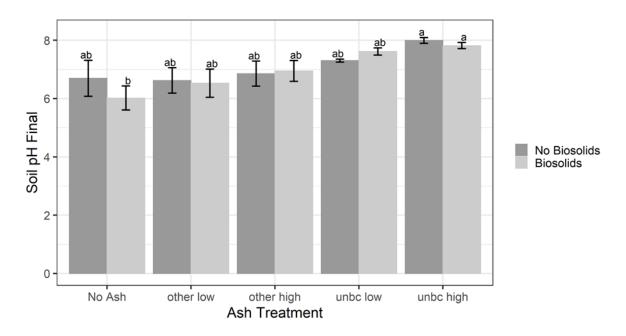


Figure 6. Mean soil pH ( $\pm$  standard error) for the two trials combined at the end of the study (harvest). Ash is designated as being "UNBC" or "Other", the latter representing PRT ash for the PRT sand trial and CPLP ash for the Gibraltar loam trial. Application rates varied with trial: Low (0.5% for Gibraltar, 1% for PRT trial); High (1% for Gibraltar, 2% for PRT trial). Treatments sharing the same lower case letter are not significantly different from each other (p < 0.05). See Table A3 for soil pH data separated by trial.

## Discussion

To determine whether land application of waste residuals would be a suitable valueadded alternative to landfilling or stockpiling, two concurrent 4-month trials utilizing bioenergy ash and biosolids were performed in a greenhouse. Using depleted soils collected locally from a mine site and a bioenergy plantation, we assessed short-term biomass production of willow (SX64 or Salix miyabeana Seemen) and soil pH changes in response to different ash and biosolids additions. In accord with similar studies (Marron 2015), we found that plant growth was enhanced with the biosolids supplement, with or without ash addition. The outcomes of our trials were consistent with published results that ash-only treatments do not always enhance tree growth like those treatments that include N fertilizers or-N rich amendments, such as biosolids (Park et al. 2005, Hannam et al. 2018). Although there was a tendency for treatments with biosolids and UNBC ash to have greatest overall biomass (Figure 3 and 4), these treatments were not statistically (p > 0.05) greater than other treatments utilizing biosolids. This suggests wood ash from bioenergy processes, along with other organic residuals, like biosolids from municipal wastewater treatment facilities, may have potential for improving the biomass of plants in certain areas in central Interior BC.

As resource extraction expands in BC, there will be increased demand to restore plant productivity at locations affected by anthropogenic disturbances. Mine soils similar to the Gibraltar loam provide challenges to reclamation as they often have a reduced capacity to support vegetation growth due to land use history (J. Evans, pers. comm., October 9, 2012). Soil disturbance can lead to lower SOC and nutrient contents in surface layers due to the displacement of organic-rich surface horizons, erosion and/or dilution with subsoil materials. High quality salvaged topsoil is often lacking at mine sites (Delong et al. 2012). Further, the use of heavy equipment at mine sites can result in soil compaction, which can decrease the efficacy of successful plant establishment

(Delong et al. 2012, Weil and Brady 2017). Improving on-site soils can present a challenge, but if the rooting capacity for plants such as willow can be ameliorated, soil physical and chemical properties, like SOC content for instance, can be improved (Delong et al. 2012). Subsequent increases in soil organic matter stimulate soil microbial activity, which supports nutrient cycling for plant life (Ingram et al. 2005, Machulla et al. 2005). Therefore, promoting plant success with soil amendments seems wise, especially when a single application of biosolids has been shown to help sequester SOC on mine tailing for over a decade (Antonelli et al. 2018).

Willow (*Salix* spp.) from the Salicaceae family is commercially cultivated in short rotations for use as bioenergy fuels (Park et al. 2005, Marron 2015). The willow (SX64) used in the current study is a hybrid designed for fast growth in such bioenergy plantations. The ecosystem and bioremedial functions of willow species are numerous. In addition to their ecological value for wildlife, willow species have been used in engineering wastewater filtration systems (Fillion et al. 2010), phytoremediation (Pulford et al. 2002), erosion mitigation (Wilkinson 1999), shelterbelts (Kort and Turnock 1998) and riparian buffers (Bourret et al. 2009).

The loam mine soil used in this study was initially pH neutral and the addition of UNBC ash (0.5% or 1%) elevated the soil pH to very high levels (pH > 9). The lower application rate of ash would be most appropriate for this soil type; there was a trend of decreased biomass production with the 1% UNBC application rate (Table A4). The same application rates of CPLP ash produced lesser increases in soil pH in the loam, likely due to the lower pH and calcium carbonate equivalence of this ash (Table 2). As noted previously, the CPLP ash contained a very high level of unburned carbon  $(\sim 50\%)$  that existed in a form resembling ground charcoal. Although the CPLP ash did not enhance the growth of plants in this study, it is expected that this ash could improve soil physical properties (e.g. reduced bulk density,

greater porosity) not investigated in this shortterm study. Further research is recommended in this area.

The PRT sand in this study was very acidic (pH 4.7) and pH neutralization using ash should have benefits to plantation soils even though we could not definitively document a willow yield increase in our trial. Although not statistically significant, the UNBC ash seemed more effective than the PRT ash for increasing plant growth when combined with biosolids, and for neutralizing soil acidity. This was likely due in part to the much greater pH and calcium carbonate equivalence of the UNBC ash (Table 2) than the PRT ash. Aside from the weathering of PRT ash during storage, different combustion conditions and fuel composition may have contributed to the lower pH, CCE and concentrations of Ca and S in this ash relative to the others (Demeyer et al. 2001, Pitman 2006). Sandy soils tend to be well-draining and lack water-holding capacity. For this reason, the willows planted in the sand trial were more prone to wilting and required more frequent watering than those planted in the loam trial. Improving the water-holding capacity of sandy soils could prove to be beneficial for the sustainable growth of willow. Biosolids have been shown to improve water retention in certain mining soils (Cele and Maboeta 2015) and bioenergy ash is by nature hydrophilic (Etiégni and Campbell 1991). Therefore both materials have potential to improve soil water-holding capacity. Although logistical limitations prevented us from studying the CPLP ash in the PRT sandy soil, we believe the high carbon content of the CPLP ash may help to improve some of the soil physical properties of the PRT sand (e.g. greater waterholding capacity) and recommend that followup investigations consider this line of research.

A limitation to this study was the lack of knowledge around long-term retention and supply of nutrients provided by the ash and biosolids, more specifically nitrogen. Bendfeldt et al. (2001) acknowledged the difficulty of long-term retention of nitrogen in mine soils. However, some studies suggest that high carbon materials, perhaps like a high-carbon ash, can help immobilize nitrogen to provide some long-term benefit to the plants (Haering et al. 2000, Daniels et al. 2001). The decomposability (and hence immobilization potential) of the CPLP high carbon ash also deserves investigation.

Research into long-term benefits of these amendments to willow production and soil quality are worthy of further study (Nissim et al. 2013), including investigation into wood fuel quality, which may be impacted by age, stand structure and soil amendments (Adler et al. 2008). Future studies could examine the longer-term neutralizing effect of ash to determine if repeat applications are needed. Willow is common in natural succession after disturbance. As such, by following natural succession templates, the likelihood of revegetation success on reclamation sites should increase. An initial fertilization or amendment to encourage rooting of these plants in these depleted soils, like the acidic sand and the restrictive loam, could prove to be an advantageous reclamation strategy. Considering the essential nutrients for plant growth contained in both ash and biosolids, along with other benefits such as neutralizing soil acidity (Pitman 2006) and improving SOC content (Antonelli et al. 2018), land application of these waste materials may be a sensible alternative to landfilling or stockpiling.

By furthering our knowledge on the behavior of ash and other residuals generated by society, land application of such materials may become more widely accepted. Sectors such as mining, forestry, bioenergy as well as oil and gas, may benefit from these types of studies, which could facilitate the reclamation and restoration of degraded or nutrient-poor sites.

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## Appendix

#### **Pest Control Measures**

Plant vigor and condition, such as wilting and insect damage, were noted qualitatively at every second watering session. Signs of stress, induced by drought or insect, were treated accordingly. Aphids were treated on November 29<sup>th</sup>, 2012 with Safer's brand insecticidal soap, at a rate of 20 mL soap/L (Woodstream Canada Corporation, Brampton, ON, 2012). Thrips were identified and controlled on December 11<sup>th</sup>, 2012, with spinosad (Success 480 SC: Naturalyte Insect Control Product, Dow AgroSciences, Calgary, AB, 2012), using a 0.5 mL / 10L application rate. Spider mites were identified February 19th, 2013, approximately day 105-110, and were removed by hand.

#### **Additional Data**

Table A1. Concentrations of additional elementals in the three ashes and biosolids. Concentrations in mg kg<sup>-1</sup> dry-weight basis, except when noted otherwise. (Mean  $\pm$  Standard Deviation; n=3). Elements determined by ICP-OES following concentrated HNO<sub>3</sub>/HCl microwave digestion.

	PRT Ash	UNBC Ash	CPLP Ash	Biosolids
As	< 4.0	< 1.0	<1.0	< 4.0
В	69.8 ± 0.7	212 ± 14	145 ± 18	$22.9 \pm 0.7$
Cd	1.2 ± 0.03	2.6 ± 0.1	5.1 ± 0.04	$2.2 \pm 0.04$
Со	38.5 ± 0.3	23.2 ± 3.3	19.7 ± 1.5	$5.3 \pm 0.2$
Cr	50.3 ± 2.8	30.6 ± 1.0	13.2 ± 0.6	53.3 ± 1.3
Cu	67.8 ± 0.3	81.5 ± 3.7	46.4 ± 4.8	2520 ± 39
Hg	< 2.0	2.4 ± 2.0	1.0 ± 0.8	< 2.0
Mn (%)	0.5 ± 0.004	1.1 ± 0.1	0.6 ± 0.01	0.1 ± 0.002
Мо	< 1.0	< 7.0	< 7.0	$26.4 \pm 0.2$
Na (%)	0.5 ± 0.003	0.7 ± 0.04	0.2 ± 0.01	0.1 ± 0.002
Ni	30.8 ± 0.8	55.8 ± 1.5	18.3 ± 0.7	32.0 ± 5.9
Pb	1.9 ± 1.6	< 4.0	< 0.4	55.9 ± 2.5
Se	< 4.0	< 7.0	< 7.0	42.6 ± 1.1
Zn	246 ± 5	470 ± 19	641 ± 16	1190 ± 24

Table A2. Concentrations of additional elements in the two soils used in study. Concentrations in mg kg<sup>-1</sup> dry-weight basis. (Mean  $\pm$  Standard Deviation; n=3). Elements determined by ICP-OES following concentrated HNO3/HCl microwave digestion.

	Loam	Sand
As	< 4.0 ± NA	< 4.0 ± NA
В	$6.42 \pm 0.76$	4.61 ± 0.07
Co	37.3 ± 0.7	19.5 ± 0.2
Cr	43.7 ± 1.3	80.4 ± 22.7
Cu	446 ± 10	18.9 ± 1.7
Hg	< 2.0 ± NA	< 2.0 ± NA
Mn (%)	0.078 ± 0.001	$0.053 \pm 0.000$
Мо	< 1.0 ± NA	< 1.0 ± NA
Na (%)	$0.09 \pm 0.01$	0.057 ± 0.001
Ni	25.4 ± 0.3	33.1 ± 0.5
Pb	1.3 ± 0.3	3.1 ± 1.3
Se	< 4.0 ± NA	< 4.0 ± NA
Zn	107.0 ± 2.1	76.8 ± 2.3

Table A3. Soil pH for ash and biosolids treatments at Time 0, the beginning of the growing period, and at the end of the growing period (n=3).

Sand Trial	Time 0	Time of Har- vest	Loam Trial	Time 0	Time of Har- vest
Control	4.7 ± 0.1	5.3 ± 0.1	Control	7.6 ± 0.2	8.1 ± 0.1
UNBC 1%	$6.8 \pm 0.04$	7.4 ± 0.1	UNBC 0.5%	8.7 ± 0.1	7.2 ± 0.1
UNBC 2%	7.5 ± 0.04	7.8 ± 0.1	UNBC 1%	9.2 ± 0.1	8.2 ± 0.1
PRT 1%	5.2 ± 0.3	5.8 ± 0.1	CPLP 0.5%	7.8 ± 0.1	7.5 ± 0.7
PRT 2%	5.5 ± 0.2	5.9 ± 0.1	CPLP 1%	7.7 ± 0.1	$7.8 \pm 0.3$
0% + B	5.3 ± 0.03	5.1 ± 0.01	0% + B	7.6 ± 0.1	$6.9 \pm 0.2$
UNBC 1% + B	7.2 ± 0.1	7.3 ± 0.1	UNBC 0.5% + B	8.8 ± 0.1	7.9 ± 0.1
UNBC 2% + B	7.7 ± 0.1	8.0 ± 0.1	UNBC 1% + B	9.2 ± 0.3	$7.7 \pm 0.3$
PRT 1% + B	5.6 ± 0.02	5.5 ± 0.4	CPLP 0.5% + B	7.8 ± 0.1	$7.6 \pm 0.2$
PRT 2% + B	$6.2 \pm 0.06$	6.2 ± 0.1	CPLP 1% + B	7.8 ± 0.1	7.7 ± 0.1

Table A4. Mean dry biomass (g) for willow in both the sand and loam trials (n= 6). Leaves and stems were considered above-ground biomass (i.e. "shoots") and roots made up below-ground biomass. For some of the loam trial, sample size did not always equal 6.

_	Mean Total Above Ground Biomass	Mean Total Below Ground Biomass	Mean Total Biomass
Sand			
0% (Control)	$1.6 \pm 0.6$	$0.4 \pm 0.2$	$2.0 \pm 0.6$
PRT 1%	$1.6 \pm 0.4$	$0.6 \pm 0.3$	$2.2 \pm 0.5$
PRT 2%	2.8 ± 1.2	0.7 ± 0.2	3.5 ± 1.3
UNBC 1%	1.9 ± 0.5	$0.6 \pm 0.2$	2.4 ± 0.7
UNBC 2%	1.6 ± 0.3	0.4 ± 0.1	$2.0 \pm 0.4$
PRT 0% + B	3.1 ± 1.4	$0.5 \pm 0.2$	3.7 ± 1.6
PRT 1% + B	$3.0 \pm 0.6$	$0.6 \pm 0.3$	$3.6 \pm 0.7$
PRT 2% + B	$2.3 \pm 0.7$	1.0 ± 0.1	3.3 ± 0.8
UNBC 1% + B	4.2 ± 1.2	1.5 ± 1.4	5.6 ± 2.5
UNBC 2% + B	$3.8 \pm 0.9$	$0.6 \pm 0.4$	4.4 ± 1.3
Loam			
0% (Control)	1.2 ± 0.3	0.4 ± 0.1	1.6 ± 0.5
CPLP 0.5%	$1.2 \pm 0.2$	0.3 ± 0.1	1.5 ± 0.2
CPLP 1%	1.1 ± 0.2	0.3 ± 0.1	1.4 ± 0.2
UNBC 0.5%	1.6 ± 0.1	0.5 ± 0.1	2.1 ± 0.1
UNBC 1%	$1.3 \pm 0.4$	$0.4 \pm 0.2$	1.7 ± 0.5
0% +B	$2.8 \pm 0.8$	$0.5 \pm 0.2$	3.3 ± 1.0
CPLP 0.5% + B	$1.5 \pm 0.4$	$0.4 \pm 0.4$	1.9 ± 0.4
CPLP 1% + B	1.7 ± 0.7	$0.5 \pm 0.2$	2.2 ± 0.9
UNBC 0.5% + B	$2.5 \pm 0.4$	$0.4 \pm 0.4$	$2.9 \pm 0.4$
UNBC 1% + B	2.1 ± 0.4	$0.5 \pm 0.4$	2.6 ±- 0.7