IMPACTS OF ONE TIME BIOSOLIDS AND FERTILIZER APPLICATION ON LONG-TERM METAL AND NUTRIENT CONCENTRATIONS ON TWO TAILINGS PONDS IN THE BC SOUTHERN INTERIOR

M.E. Phillips[1]
W.G. Gardner[2]
T.G. Pypker[3]

[1] Thompson River University, MSc Student
[2]Thompson Rivers University, Associate Professor, PhD
[3]Thompson Rivers University, Assistant Professor, PhD

Thompson Rivers University
900 McGill Road
Kamloops, BC, V2C 0C8

ABSTRACT

Previous research has demonstrated that the use of organic amendments, specifically biosolids, can address limitations to initial vegetation establishment on mine tailings. It is less understood how these systems will function in the long term. In 2015, a field study at Teck Highland Valley Copper in the BC Southern Interior was conducted to determine the long term effects of fertilizer and biosolids on nutrients and elemental concentrations in two tailings ponds. Seventeen years prior, biosolids were applied in a randomized complete block design at rates 50, 100, 150, 200, and 250 Mg ha^{-1}. The biosolids treatments continue to demonstrate a very clear increase to the nutrient status of the tailings, while the fertilizer treatment does not statistically differ from the control treatments. There are also still elevated levels of metals within biosolids treated plots, but results vary by metal with many showing a plateau, where additional biosolids do not increase their concertation. With site specific planning, metal concentrations can be controlled below levels of concern, while at the same time promote nutrient cycling. In conclusion, it appears a one-time biosolids application can assist reclamation in a trajectory towards a self-sustaining state. Further research is also being done examining the plant community and soil development.

Key Words:

Mine reclamation, tailings pond, biosolids, nutrients, potentially toxic metals,

INTRODUCTION

Establishing a self-sustaining vegetation community on mine tailings is challenging because mine tailings are nutrient poor, contain trace toxic metals, lack organic matter, lack healthy microbial community, are vulnerable to erosion, have a low water holding capacity, and lack soil structure (Brown et al. 2003; Santibáñez et al. 2007; Gardner et al. 2011; Brown et al. 2014). Biosolids can improve these conditions by adding nutrients and organic matter, thereby improving nutrient cycling, energy cycling, and leading to a self-sustaining vegetation community (Larney and Angers 2012; McCall et al. 2015). The organic matter in biosolids can also bind with many toxic metals, potentially reducing their harmful effects (Brown et al. 2003; Brown et al. 2005).

The Canadian Council of Ministers for the Environment (CCME) defines municipal biosolids as "… municipal sludge which has been treated to meet jurisdictional standards, requirements or guidelines including the reduction of pathogen and vector attractions, where municipal sludge is the mixture of water and solids from sewage systems" (CCME 2012). The *Organic Matter Recycling Regulation of British Columbia* (2002) defines biosolids as stabilized municipal sewage sludge from waste water treatment or septage treatment process, reducing pathogens and vector attractants. The resulting biosolids are used in forestry, mine reclamation, agriculture and generally to improve degraded land (CCME 2012).

Biosolids impact the nutrient and metal parameters of tailings, including plant available fractions (Gardner et al. 2011). The impacts of biosolids application depend on the chemical condition of both the biosolids and tailings. Biosolids significantly increase the concentration of essential macronutrients in mine soils, due to the high concentration in the biosolids (Gardner et al. 2011; Antonelli et al. 2012). Past research demonstrates that biosolids are associated with an increase in ammonium (NH4), nitrate (NO3), phosphorus (P), potassium (K), calcium (Ca) and carbon (C) (Tripathy et al. 2003; Shrestha and Lal 2006; Wallace et al. 2009; Gardner et al. 2011; Brown et al. 2014; McCall et al. 2015). Hence, biosolids can alleviate one of the largest limitations to vegetation development, nutrient availability. Biosolids can also increase concentrations of metals of concern, with most of this documentation occurring on agricultural soils with annual applications (Zhang et al. 2012; Yang et al. 2014). For example, Yang et al. (2014) reported increases in cadmium (Cd), copper (Cu), molybdenum (Mo), lead (Pb), antimony (Sb), tin (Sn), and zinc (Zn). The environmental risk of metals due to mobility and leaching is still not well understood. In field experiments on mine tailings and agricultural fields, Gardner et al. (2011) and Yang et al. (2014) did not identify leaching through the soil profiles, whereas in a bench top experiment Zhang et al. (2012) identified leaching of some metals through mine tailings treated with biosolids. The potential of metals to leach due to biosolids applications depends on interactions with the substrate and site specific characteristics.

The majority of past work on biosolids has reported the short term impacts of biosolid application (e.g. Santibáñez et al. 2008; Boyter et al. 2009; Gardner et al. 2011). The few longer term studies have mainly focused on agricultural crops and multiple applications (e.g. Yang et al. 2014). Long term studies on tailings ponds are necessary to examine the fate the organic matter, and how metal availability may change over time (McBride 1995). It has been hypothesized that the benefit of biosolids may only be short term, and as organic matter decomposes, the site will again become nutrient deficient, compromising its ability to be sustainable in the long run (McBride 1995). In contrast, others hypothesize that biosolids kick start the nutrient cycling process and the growth and decomposition of vegetation continues to sustain nutrient levels (Shrestha and Lal 2006; Antonelli et al. 2012). Because of these long term uncertainties, further research has been needed.

In 1998, two mine tailing ponds received a one-time application of biosolids (Gardner et al. 2011). These sites provide the opportunity to address questions on how reclaimed ecosystems function through time (Larney and Angers 2012). This project provides the opportunity to look at these parameters in two alkaline Cu-Mo tailings ponds, very close in proximity, with different moisture contents and textures, under the same experimental conditions. Hence, the objective of this study is to examine total and available nutrient and chemical concentrations in two tailings ponds 17 years after biosolids were applied and determine if these ponds i) still demonstrate increased nutrients compared to a control with no biosolids or a one-time fertilizer, ii) if total and available metal concentrations are elevated in biosolids treatments above control treatment, iii) and provide insight to potential long term benefits and risks associated with the use of a onetime biosolids application.

METHODS

Site Description

The study site is located at Teck Highland Valley Copper (HVC), approximately 76 km south west of Kamloops, British Columbia, Canada (lat. 50°28’23.22” N, long. 121°01’18.50”W). HVC is located on the Thompson Plateau physiographical subdivision, within the Engelmann Spruce Subalpine Fir very dry cold, Montane Spruce very dry cool, and Interior Douglas Fir dry cool, biogeoclimatic zones of BC (Government of British Columbia 2014). The experiment was conducted on two mine tailings ponds, Trojan and Bethlehem. Trojan pond is classified as sand textured, herein referred to as the sand (S) pond and Bethlehem pond is classified as a silt loam, herein referred to as the silt loam (SiL) pond. The S site is located at 1432 m above sea level, and the SiL at 1480m, with the centre of each pond approximately 1.5 km apart. Both were milled from granodiorite rock containing 60% plagioclase, 10% k-feldspar and 10% quartz with the remaining mineral composition including calcite and other differing minerals (Gardner et al. 2010). Both tailings ponds have been inactive since the early 1980’s. Further site details can be found in Gardner et al. (2010).

Experimental Design

In 1998, a randomized complete block design with seven treatments and eight blocks was established on each pond. Blocking was used to account for an increase in moisture moving towards the centre of the tailings ponds. Treatment plots were originally seven by three meters. In 2015, the size of the plots were was reduced to five by two meters to reduce edge effects and minimize the effects of vegetation and sediment drift that had occurred over time. Treatments were applied in 1998, including a control (C0), a fertilizer treatment (F0), and class B biosolids amendments applied at rates of 50, 100, 150, 200, and 250 Mg ha^{-1} (B50, B100, B150, B200, and B250). Biosolids treatments were incorporated into the top 15cm of the soil surface with a rototiller. Nitrogen (N), phosphorus (P), potassium (K), zinc (Zn) and boron (B) content of the fertilizer matched the concentrations in the B150 treatments. In spring 1999, all treatment plots were hand raked and seeded with an agronomic grass and legume seed mixture at a rate of 36 kg ha^{-1}. The seed mix consisted of 33.2% pubescent wheatgrass (*Agropyron trichophorum* (Link) Richt.), 7.5% orchard grass (*Dactylis glomerata* L.), 4.0% creeping red rescue (*Festuca rubra* L. var. rubra), 14.7% Russian wild rye grass (*Elymus junceus* Fisch.), 34.6% alfalfa (*Meticago sativa* L.) and 5.9% alsike clover (*Trifolium hybridum* L.) by weight.

Tailings Sampling

Tailings samples were collected in September 2015. Within each treatment plot, ten random soil cores were taken from the 0-15 depth. Within each plot, soil cores were mixed together for a given depth to create a single homogenized composite sample. These samples were then dried at 60°C for 24 hours and sieved to 2mm (9 Tyler mesh) to break up aggregates, remove large pebbles and any root or photosynthetic material. Samples were then sent to an external lab for metal and nutrient analysis. The specific parameters and the methodologies are shown in Table 1.

Table 3. Parameters tailings samples were tested and the corresponding methodologies used.

Parameter	Method
Total Ag, As, B, Cd, Cr, Co, Cu, Fe, K, Mg, Mn, Mo, Ni, P, Pb, Sb, Se, Sn and Zn	ICPMS analysis and strong acid digestion
Total Mercury (Hg)	ICP-OES Analysis
Available Cu, Fe, K, Mn, Mo, P, Zn	ICPMS, Ab-DPTA extraction
Available Sulphate (SO_4)	Extraction and ICP-OES
Ammonium (NH_4) and Phosphate (PO_4), Nitrate (NO_3)	Extraction and Colorimeter
Total C and N	Combustion

Statistical Analysis

Results for each parameter were pooled for each treatment across both ponds. Some parameters were log or square root transformed to better meet assumptions of homogeneous variance. A two-way analysis of variance with additional blocking (ANOVA) was conducted to examine potential interactions between treatment and pond, and differences between treatments. If treatments were significant ($p<0.05$), a Tukey multiple comparison of means post hoc testing was conducted. All statistical analyses were conducted in R version 3.2.3.

RESULTS

Nutrient Response to Biosolids

Nutrients significantly increase in concentration with biosolids application compared to the fertilizer and control treatments (Figure 1 and 2). Many nutrients show a plateau effect between the B100 and B200 treatments, where higher application rates did not significantly increase nutrient concentrations (Table 2). Significant interactions occurred with both total C (p=0.020) and P (p=0.007). Concentrations of C and P were similar at both ponds in the control, but the response to biosolids in the SiL pond is larger (Figure 1). Concentrations were generally higher in the SiL pond, with only PO_4 being higher at the S pond (Table 3).

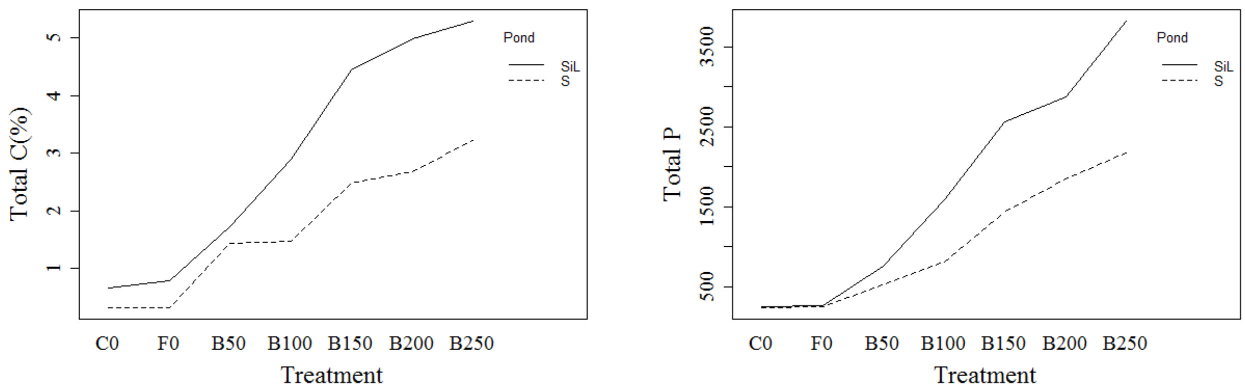

Figure 1. Nutrient concentrations by treatment and pond that displayed a significant interaction effect. Units are in mg kg^{-1} unless otherwise described.

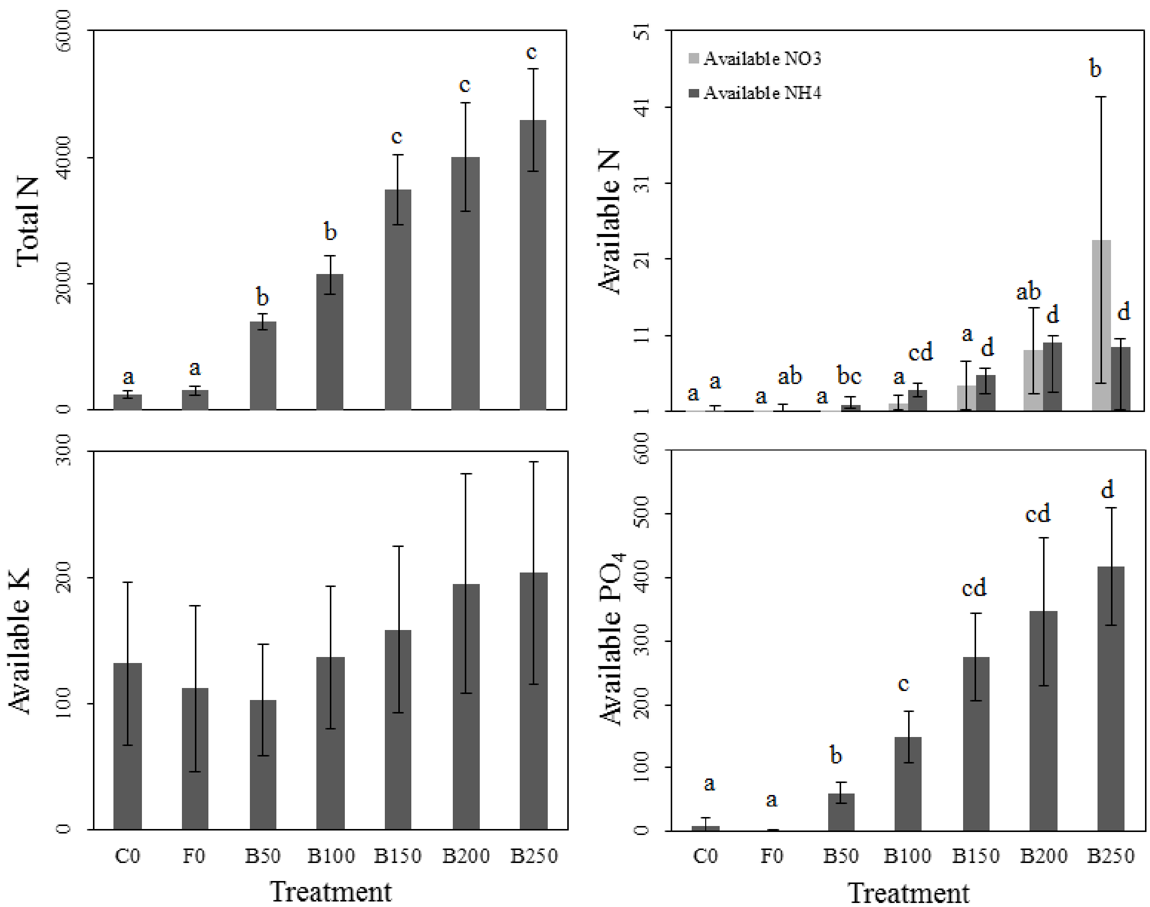

Figure 2. Treatment means for total and available nutrients that demonstrate a significant treatment effect and an insignificant pond interaction. Error bars represent 95% confidence intervals and units are in mg kg^{-1}.

Metal Response to Biosolids

Of the 24 metals examined, 19 showed a significant treatment effect (Table 2). All significant effects resulted in increasing trends where the C0 and F0 treatments were not statistically different for any parameter (p<0.05). Many metals, similar to the nutrients, demonstrated a plateau effect, where higher application rates did not result in higher concentrations (Table 2). In most cases, these parameters were found in higher concentrations in the SiL and lower concentrations in the S pond, with the exception of total Cu (table 3). Total Ag (p=0.91) and Se (p=0.071) were the only metals not significantly affected by pond. Some metals displayed significant interactions between treatment and pond. Significant interactions were seen with total As (p=0.003), Se (p=0.002), and available Mo (p=0.000), SO_4 (p=0.022), and Cu (p=0.000). These parameters are affected differently by the biosolids treatments depending on whether the SiL or S pond is being examined (Figure 3).

Table 4. Nutrients and metals with a significant treatment effect as detected by a two-way ANOVA, and the treatment and respective mean, with the highest concentration as detected by a post hoc Tukey HSD. All significant effects demonstrate an increasing trend with additional biosolids application. All means and standard errors are displayed in mg kg^{-1} unless otherwise specified.

Parameter	P Value	Highest Significant Concentration	Mean[a]	SEM[b]
Total Nutrients				
C (%)	$<2x10^{-16}$	NA[c]	4.31	4.20
P	$< 2.2x10^{-16}$	NA	3049.33	155.65
N	$< 2.2x10^{-16}$	B150	3480.00	282.77
Available Nutrients				
K	$8.89x10^{-4}$	NS [d]	1298.27	170.39
NH4	$< 2.2x10^{-16}$	B100	3.75	0.47
PO4	$< 2.2x10^{-16}$	B150	274.66	35.55
NO3	$1.88x10^{-4}$	B200	8.98	2.88
Total Metals				
Mg	$1.03x10^{-7}$	NS	1250.07	111.21
Fe	$2.91x10^{-7}$	NS	7683.33	540.60
Co	$2.32x10^{-8}$	NS	1.91	0.16
B	$<2.2x10^{-16}$	B100	0.41	0.07
Ag	$<2.2x10^{-16}$	B150	5.05	0.51
Sn	$<2.2x10^{-16}$	B150	5.01	0.51
Sb	$<2.2x10^{-16}$	B200	0.85	0.12
Cd	$<2.2x10^{-16}$	B200	1.02	0.12

Hg	$<2.2x10^{-16}$	B200	1.04	0.12
Ni	$1.06x10^{-13}$	B200	8.77	0.70
Pb	$<2.2x10^{-16}$	B200	22.01	2.86
Se	$<2.2x10^{-16}$	NA	1.13	0.12
As	$<2.2x10^{-16}$	NA	3.36	0.33
Cr	$<2x10^{-16}$	B250	16.27	2.02
Zn	$<2.2x10^{-16}$	B250	203.67	24.34
Available Metals				
Fe	$2.88x10^{-10}$	NS	95.90	16.91
Mo	$9.60x10^{-4}$	NA	2.29	0.64
SO4	0.043	NA	245.13	86.15
Zn	$<2.2x10^{-16}$	B100	20.00	5.81

[a] Mean of the treatment listed in the previous column, or B250 treatment if not specified
[b] Standard error of the mean
[c] Not Applicable, significant interactions, therefore no post hoc testing was conducted
[d] Not Significant, no differences detected with Tukey HSD

Table 5. Nutrients and metals with a significant pond effect as detected with a two-way ANOVA. The pond with the higher concentration is also shown.

Parameter	P Value[a]	Higher Concentration
Total Nutrients		
C	$< 2x10^{-16}$	SiL
K	$< 2.2x10^{-16}$	SiL
N	$6.89x10^{-13}$	SiL
P	$7.26x10^{-11}$	SiL
Available Nutrients		
K	$<2.2x10^{-16}$	SiL
NH4	$2.21x10^{-7}$	SiL
PO4	$2.39x10^{-4}$	S
Total Metals		
As	$1.34x10^{-8}$	SiL
B	$<2.2x10^{-16}$	SiL
Cd	1.51x10-[15]	SiL
Co	$<2.2x10^{-16}$	SiL
Cr	$<2.2x10^{-16}$	SiL
Cu	3.53x10-[12]	S

Fe	$<2.2x10^{-16}$	SiL
Hg	$8.46x10\text{-}^{12}$	SiL
Mg	$<2.2x10^{-16}$	SiL
Mn	$<2.2x10^{-16}$	SiL
Mo	$<2.2x10^{-16}$	SiL
Ni	$3.501x10^{-11}$	SiL
Pb	$1.52x10^{-11}$	SiL
Sb	$2.95x10^{-11}$	SiL
Sn	$6.43x10^{-10}$	SiL
Zn	$1.16x10^{-9}$	SiL
Available Metals		SiL
Cu	$5.38x10^{-7}$	SiL
Fe	$<2.2x10^{-16}$	SiL
Mo	$<2.2x10^{-16}$	SiL
SO4	$1.07x10^{-15}$	SiL
Zn	$3.21x10^{-15}$	SiL

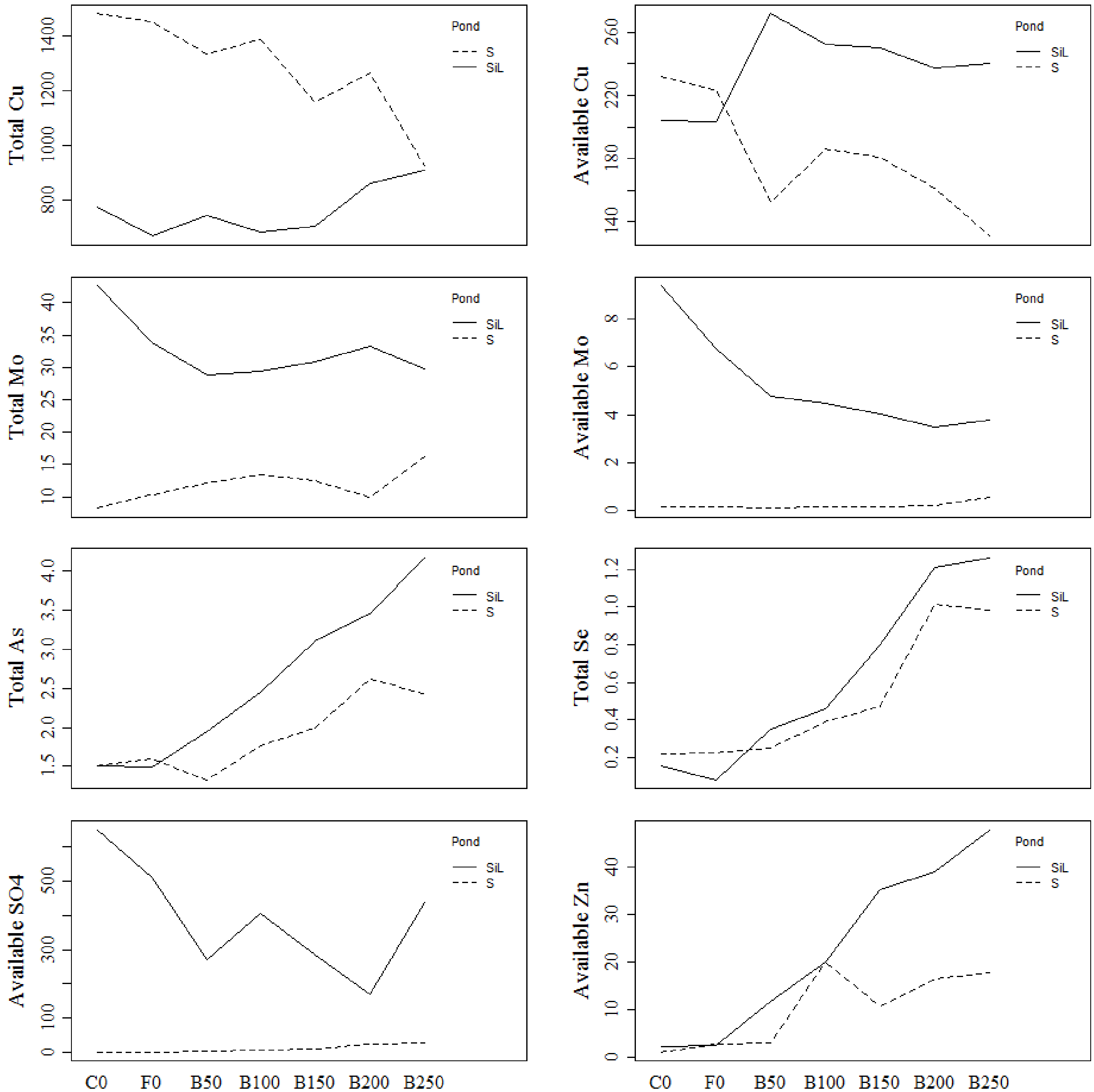

Figure 10 Metals concentrations by treatment and pond that had a significant interaction. Units are in mg kg^{-1}.

DISCUSSION/CONCLUSION

Nutrient Response to Biosolids

Seventeen years post treatment, nutrients were elevated relative to the control and fertilizer treatments, clearly demonstrating that a onetime biosolids application can result in a vegetation community that replenishes nutrients in the soil. The benefit to the nutrient status of a soil with biosolids has been widely observed in both mine wastes and degraded soils, ranging in time frames from 5 months up to 5 years after application (Brown et al. 2003; Walter et al. 2006; Brown et al. 2007; González-Alday et al. 2008; Forján et al. 2014). In these cases, biosolids have provided benefits to the nutrient status of the soil, specifically improving the C:N ratio and N over that of using fertilizers alone. While many studies suggest that there is a longer term benefit, many point out the lack of long term data, which limits the ability to make long term conclusions (Brown et al. 2003; Basta et al. 2004). This study is the longest time frame the authors are aware of that examines the effects of a one-time biosolids application on tailings. Similar to Walter et al. (2006), this study provides evidence that biosolids application can accelerate the trajectory of reclamation and soil development and provide a long term benefit to the nutrient status of the tailings.

The mean concentrations of total carbon in the biosolids applications are similar to those levels found between 1998 and 2001 (Gardner et al. 2011), indicating biosolids promote a self-sustaining trajectory and nutrient cycling within the tailings. In Gardner et al. (2011), the average C concentrations, and their standard error, across all B150 treatments was 36±0.2 g kg^{-1}, and 64±5.8 g kg^{-1} in B250 treatments. In 2015, those averages are 34±3.1 g $kg^{-1}$and 43±4.2 g kg^{-1} in B150 and B250 treatments, respectively. Preliminary analysis of these data suggests that total carbon concentrations do not differ between 2000 and 2015, suggesting the site is maintaining carbon levels through plant growth and nutrient cycling. Other nutrients examined in 2015 also display similar trends to those seen in Gardner et al. (2011), further supporting the hypothesis that biosolids provide a long term supply of nutrients that benefit the reclamation of tailings ponds.

Metal Response to Biosolids

Metals are generally elevated in biosolids treatments, but responses at higher biosolid applications varied from metal to metal. Variations in metal availability across higher applications may be the result of metals competing for binding sites, as those with greater affinities will outcompete those with lower affinities to biosolid constituents (Stietiya and Wang 2011). The elevated metal concentrations in biosolids treatments likely exists because of complexation with organic matter, and/or Al, Mn and Fe oxides, which provide critical binding sites, preventing mobility of many metals (Basta et al. 2004; Stietiya and Wang 2011). Binding to metal oxides is also most effective in high pH's, like those found in the two ponds of this study (Basta et al. 2004; Gardner et al. 2011). Therefore, elevated metals are not surprising, as even if the organic matter has been decomposing, oxides are still effective binding sites (Stietiya and Wang 2011).

Many trends seen with metal concentrations in Gardner et al. (2011) were also seen in 2015. Those that demonstrated no treatment effect in Gardner et al. (2011) but demonstrated an increased trend in 2015 included As, B, Cd, Cr, and K. No analysis has yet been done to confirm if the two years are significantly different, but a visual comparison shows the greatest differences with B and Cr. B concentrations reached a mean and standard error of 28±0.92 mg kg^{-1} in B250 treatments in Gardner et al. (2011), but never exceeded 1 mg kg^{-1} in 2015. Gardner et al. (2011) reported that mean Cr concentrations where 36±2.33 mg kg^{-1}, but 17 years later these values were only 16.28±2.02 mg kg^{-1} in 2015 on the B250 treatments. Lower concentrations are also found in control treatments in 2015 compared to Gardner et al. (2011) (unpublished data). In some cases, it is also possible that a treatment effect with these metals is now apparent because metals in the tailings without biosolids were more readily mobile, whereas the biosolids treatments may assist in preventing some mobility. This effect has been reported under shorter time frames in benchtop experiments (Pond et al. 2005; Kelly et al. 2014), and could further differentiate the treatments over time, although this has not yet been directly examined.

Generally, the SiL demonstrated higher concentrations than the S pond, and some metals demonstrated significant interactions between treatment and pond. Initially, metal concentrations and cation exchange capacity in the finer textured SiL pond was greater (Gardner et al. 2010). The differences in metal concentrations seen between the two ponds in 2015 are likely related differences in these initial characteristics and differences in milling processes and ore bodies when the tailings were first deposited. It has also been shown that some heavy metals, specifically Cu, Pb and Zn, will show greater retention and lower mobility in finer, clay fraction of soils (Brazauskiene et al. 2008; Rinaudo et al. 2009). This may also contribute to the higher concentrations found in the SiL site and the different responses between ponds seen for some metals.

Overall Benefits and Risks

One of the risks associated with the use of biosolids is the potential to elevate metals to levels of concern. While it is well known that total metal concentrations can increase, the vital processes to consider are availability and mobility. When an elemental component is bound to the residual constituents in the soil, such as organic matter, metal oxide, and sometimes carbonates, the mobility of that metal is greatly reduced. When biosolids are added, these constituents are also added, increasing the ability of the soil to immobilize metals (Basta et al. 2004). If biosolids have been decomposing, which reduces the organically bound fraction of metals, Al, Fe and Mn oxides may become more important long term factors that mitigate the risk of increased mobility (Basta et al. 2004). More alkaline soils also tend to show greater retention of metals, with lower risk of leaching (Walter et al. 2006) . This research shows that many elemental levels will increase with biosolids application, but with appropriate, site specifically prescribed application rates, exceedances can be avoided. In this study, a few 'total' metals exceeded Canadian Council of Ministers of the Environment (CCME) guidelines for agricultural soils due to biosolids, but none exceeded guidelines for industrial soils.

This study demonstrates there are long term benefits to soil nutrients when biosolids are used in tailings reclamation. While high nutrient loading can increase the risk of NO_3 and P leaching and runoff, it is suggested that these risks are especially low in arid environments (Walter et al. 2006). The benefits of the added nutrients are also visible in other site parameters, specifically biomass and species composition (unpublished data). This site was also tested from 1994 to 1996 to identify any concerns with metal toxicity to livestock from forage consumption, and no adverse reaction to cattle were reported (Gardner et al. 2003). With the collective data from metals and nutrients in this study, there appears to be a great benefit to the long term sustainability of these sites, with no metals raising critical concern.

Every tailings pond will provide a new set of parameters to consider, including initial metal concentrations, pH, hydrology, climate, and other physical parameters like texture, all which are likely to change the benefits and risks to some degree. Careful planning and appropriate application rates will reduce the risk of metal loadings exceeding guidelines, reduce metal mobility and provide necessary nutrients for plant establishment and long term growth, which may be an indication of a positive and sustainable trajectory for the plant community, overall helping reclamation goals to be achieved.

Future work at this site will expand our understanding of the long term impacts of using biosolids. This work includes examining metal and nutrients in a second depth in the soil, plant nutrient and metal content, plant species composition, a number of soil physical characteristics, as well as statistically comparing all data collected from 1998 to 2000 with data collected in 2015. Collectively this will provide a more in depth examination of the long term effects of using a one-time biosolids application compared to a fertilizer treatment as a strategy for tailings reclamation.

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