LONG TERM CHANGES IN COPPER-MOLYBDENUM MINE TAILINGS RECLAIMED WITH A ONE-TIME BIOSOLIDS APPLICATION IN THE BRITISH COLUMBIA, SOUTHERN INTERIOR

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DEDICATION

This thesis is dedicated to the memory of my mother, Elaine Kobzey. She is the one who encouraged me to pursue a Master's degree and gave me the confidence to do so.

Live in each season as it passes;
breathe the air, drink the drink, taste the fruit,
and resign yourself to the influences of each.
- Henry David Thorenu

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ABSTRACT

During mineral mining, ore is removed from the ground, and milled to a fine particle size to extract the desired mineral. After the desired mineral is removed the remaining ground up rock are the tailings. Tailings are commonly stored in tailings storage facilities, covering a designated land in meters of finely ground rock that is intensively managed and monitored. In British Columbia, these tailings storage facilities are required by law to be returned to a productive sustainable use which usually involves revegetation. Tailings present many challenges during revegetation. They are usually nutrient deficient, contain no organic matter, can contain high metal concentrations, are vulnerable to erosion, have poor water holding capacity and lack soil structure. Therefore, before vegetation will establish and persist, these site limitations need to be addressed. The use of organic amendments, such as biosolids, has shown to alleviate these limitations. There is still debate whether biosolids provide a long term benefit, or if these benefits diminish with time as the organic matter decomposes. There are also concerns that biosolids increase metal loadings, potentially posing a risk to the environment through leaching and plant uptake. To test this, a study was conducted on two tailings storage facilities, one a sand and the other a silt loam, in British Columbia's southern interior. Biosolids were applied at rates of 50, 100, 150, 200 and 250 Mg ha^{-1} along with a control and fertilizer treatment in a randomized complete block design field experiment in 1998. These plots were sampled in 2000, 2015 and 2016. Comparisons were made between 2000 and 2015, and between treatments in 2015 for macronutrients, total and available metals, bulk density, and biomass. Other physical characteristics that were compared across treatments in 2016 included aggregate stability, saturated hydraulic conductivity, and water retention curves. Many macronutrients such as carbon did not change from 2000 to 2015, and remained elevated in biosolids treated plots, demonstrating a long-term benefit to the tailings. Many metals still remained elevated, suggesting little movement through the soil profile. Zinc and nickel were the only metals that showed some exceedances above guidelines for agricultural soils. Aggregate stability and hydraulic conductivity improved in biosolids treated plots over the control in 2016. Biomass and litter production was also greater in biosolids treated plots in 2015. This data suggests that biosolids can provide a long-term benefit as an organic amendment to tailings, while proper applications rates can mitigate risks of causing metal exceedances.

Keywords:

Tailings, biosolids, mine reclamation, nutrients, metals, soil, aggregates, hydraulic conductivity, bulk density, biomass

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LIST OF ABBREVIATIONS

AWHC = Available water holding capacity

D_b = Bulk density

FC = Field capacity

K_{sat} = Saturated hydraulic conductivity

MPN = Most probable number

PAW =Plant available water

PPCP = Pharmaceuticals and personal care products

PWP = Permanent Wilting point

S = Sand

SiL = Silt loam

TSF = Tailings storage facility

WRC = Water retention curves

Chapter 1. INTRODUCTION

MINING AND RECLAMATION

Mining is a major contributor to the economy in Canada, directly employing more than 373,000 people and contributing 3.4% of the country's GDP in 2015 (Marshall 2016). In total, the mining industry contributed $56 billion to the 2015 GDP, with mineral extraction making up $24.6 billion (Marshall 2016). Mineral production in British Columbia alone was worth $5.9 billion (Marshall 2016). In 2012, $56 million was spent by the Canadian mining industry reclaiming and decommissioning mine affected areas (Statistics Canada 2012). This resource development is dependent on environmentally responsible practices, including the effective and efficient reclamation of areas no longer part of the active mining stream.

The mining process tends to impact a relatively small area, but those impacts tend to be very significant (Marshall 2016). Through mineral mining, rock is pulled from the ground and either placed as waste rock in stockpiles or ore containing rock is milled to a very small particle size to allow separation of the desired mineral from the rock. These practices result in two waste products that need to be reclaimed, waste rock and tailings. The production of these waste products disturbs and removes native soil and vegetation communities. Replacing and rebuilding these soils and vegetation communities to a self-sustaining state continues to be a challenge for many mines.

In British Columbia, mined lands must be reclaimed to an end land use approved by the chief inspector (Mines Act 2008). This generally involves revegetating the land to a self-sustaining state, requiring the re-establishment of soil and ecosystem functions and services. It is possible that this may happen naturally, but the time scale required may not be acceptable by public or industry, thereby requiring human intervention. Additionally, in some cases, succession may not be able to develop a fully functioning soil because of extreme anthropogenically caused conditions, such as metal toxicity and the removal of native organisms (Larney & Angers 2012; Shrestha & Lal 2006). For example, a tailing site in Colorado had no vegetation established over 70 years until human intervention (Brown et al. 2007). The purpose of human intervention is to address limitations of vegetation community development so progression can occur within an acceptable timeline, then continue without additional human intervention.

TAILINGS LIMITATIONS

Tailings present many limitations to reclamation and vegetation establishment. They tend to be nutrient poor, contain trace toxic metals, lack organic matter, are vulnerable to erosion, have a low water holding capacity, and lack soil structure (Norland and Veith 1995; Brown et al. 2003; Santibáñez et al. 2007; Gardner et al. 2011; Brown et al. 2014). Organic amendments, such as manure, composts, wood chips, and biosolids, have shown to be very successful at alleviating these limitations (Larney and Angers 2012; Gardner et al. 2012; Biao, Yanbing, and Qingrui 2015; Santibáñez et al. 2008) whereas fertilizers alone do not address the physical limitations (Santibáñez et al. 2007; Gardner et al. 2010). The common goal between these amendments is the creation of a self-sustaining vegetation community through the creation of favorable soil conditions. Hence, the goal of creating a self-sustaining vegetation community starts with the goal of establishing favourable soil conditions on these tailings.

An important component of soil and the communities that develop on it is organic matter (Evanylo et al. 2005; Shrestha and Lal 2006). Therefore, amendments that increase organic matter are vital to the development of soil on mine wastes (Larney and Angers 2012; Brown et al. 2014; Evanylo et al. 2005). Organic amendments also change water, nutrient, and metal movement through the substrate which in turn impacts the development of soil through chemical and physical processes (Gardner et al. 2012). Organic amendments can also support the development of microbial communities which support vegetation through symbiotic relationships and nutrient cycling (Gardner et al. 2010; Sheoran et al. 2010). For example, organic amendments have been shown to improve soil conditions for plant growth by reducing bulk density, increasing water holding capacity, increasing infiltration, increasing nutrients, and changing the availability of metals to plants (García-Orenes et al. 2005; Asensio et al. 2013; Tripathy et al.2003; Gardner et al. 2010). All of these parameters interact together to create a more favorable soil substrate for plants to grow. The overall benefit of an amendment and how it will impact the substrate and vegetation community is very site specific, depending on the initial chemical and physical condition of the substrate to be reclaimed (Larney and Angers 2012).

BIOSOLIDS

Biosolids are one of the organic amendment options available for reclamation. 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. Biosolids can be created through anaerobic or aerobic digestion, alkaline stabilization, dewatering and composting. Most production methods use high temperatures over an extended period of time (OMRR 2007). After treatment, biosolids have reduced volatile organic compounds, reduced odour, and decreased or eliminated pathogens (CCME 2012; OMRR 2007). The resulting biosolids are used in forestry, mine reclamation, agriculture, and to degraded land (CCME 2012).

The use of biosolids are controlled mainly through provincial and territory regulation (CCME 2010). In British Columbia, biosolids are regulated under the *Organic Matter Recycling Regulation (OMRR)* through the *Environmental Management Act* and the *Health Act* (CCME 2010). Under OMRR, biosolids can be classified as class A biosolids and class B biosolids depending on the levels of pathogens and heavy metals. The pathogen and metal limits for class A and class B biosolids from the OMRR are listed in Table 1.1.

Table 1.1. The limits for metal (ug g^{-1}) and pathogen (MPN) contaminants in class A and class B biosolids (OMRR 2007).

Parameter	Class A Biosolids	Class B Biosolids
Pathogens (MPN per gram of total dry solids)	< 1 000	< 2 000 000
Arsenic (ug g^{-1})	12	75
Cadmium (ug g^{-1})	3	20
Chromium (ug g^{-1})	100	1060
Cobalt (ug g^{-1})	34	150
Copper (ug g^{-1})	400	2200
Lead (ug g^{-1})	150	500
Mercury (ug g^{-1})	2	15
Molybdenum (ug g^{-1})	5	20
Nickel (ug g^{-1})	62	180
Selenium (ug g^{-1})	2	14
Zinc (ug g^{-1})	500	1850

It has been demonstrated that biosolids can alleviate many of the limitations to vegetation establishment on tailings. 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. 2005b; Brown et al. 2003). Overall, biosolids have can alter the soil chemical and physical properties of a receiving medium, which in turn effects the vegetation community.

Soil Chemical

Biosolids impact the chemical parameters of tailings including pH, cation exchange capacity, electrical conductivity, nutrients and metal availability (Norton et al. 2004; Brown et al. 2005a; Walter et al. 2006; Gardner et al. 2010; Asensio et al. 2014). The impact of biosolids application depends on the chemical condition of both the biosolids and tailings. For example, the risk of metal leaching is very site specific due to the many variables involved, such as pH, carbonates, metal oxides, particle size, and soil moisture to name a few (Evanko et al. 1997).

The pH of the substrate will change depending on the relative pH of the biosolids, thereby altering the availability of cations. For example, tailings with a lower, acidic pH will increase towards a more neutral to alkaline if biosolids tend to be more neutral or alkaline (Brown et al. 2003; Brown et al. 2007; McBride and Evans 2002). If tailings are more alkaline and biosolids are more neutral or acidic, their application will lower the pH of the substrate (Santibáñez et al. 2008). The pH is a very important parameter because it is one of the factors that determine how metals and nutrients will react and move in the soil substrate. Generally, as pH increases cations (cadmium (Cd), copper (Cu), lead (Pb), nickel (Ni), potassium (K), sulphur (S), zinc (Zn)) become less available and anions (molybdenum (Mo) and arsenic (As)) become more available (Richards et al. 2000; Brown et al. 2005a; Pond et al. 2005; Wichard et al. 2009). If biosolids increase the pH of the receiving substrate, making it become more basic, the mobility of cationic metals will decrease (Tripathy et al. 2003). This is the case for most of the literature as mine spoils tend to have a lower pH, being more on the acidic end of the scale. The pH of soil or tailings is one of the factors determining available metals from the total metals.

Cation exchange capacity (CEC) is the ability of the soil to hold onto cations and nutrients that can be exchanged between plant roots and the substrate it is growing in. CEC measures the amount of sites on the substrate that are available for binding to metals (Silveira et al. 2003). The CEC of a soil can be influenced by the application of biosolids (Gardner et al. 2010). Because CEC is strongly influence by organic matter and mine tailings tend to be extremely deficient in organic matter, the addition of biosolids increases CEC (Evanylo et al. 2005; Gardner et al. 2012; Shrestha and Lal 2006; Rate et al. 2004). In contrast, there is also a portion of literature showing that CEC may not be altered by biosolids applications, even when organic carbon is significantly increased (Zebarth et al. 1999; Brown et al. 2005b).

Biosolids significantly increase the concentration of essential macronutrients found in mine soils, due to the high concentration in the biosolids. Applying biosolids has increased nitrogen (N), phosphorus (P), potassium (K), and carbon (C) (Shober et al. 1996; Veeresh et al. 2003; Basta et al. 2004; Shrestha and Lal 2006; Walter et al. 2006; Wallace et al. 2009; Gardner et al. 2011; Brown et al. 2014; McCall et al. 2015). This alleviates one of the main limitations to vegetation development, nutrient availability.

Soil Physical Improvements

Biosolids may contribute to soil development in mine tailings (Brown et al. 2014). Physical development refers to the physical changes of the tailings from a massive uniform material to a complex, heterogeneous material. Changes suggestive of soil development in this trajectory include parameters such as reduced bulk density and increased porosity, improved aggregate stability, improved water infiltration and improved water holding capacity. Biosolids application results in an increase in organic carbon, which can decrease bulk density, increase porosity, increase water holding capacity and increase aggregate stability (Brown et al. 2014; García-Orenes et al. 2005; Tripathy et al. 2003; Gardner et al. 2012; Shrestha and Lal 2006; Wallace et al. 2009). The improvement of these parameters leads to increased plant growth and microbial activity (Gardner et al. 2010).

With an increase in biological activity and organic carbon, stable soil aggregates begin to form (Tripathy et al. 2003; Asensio et al. 2013). Larney and Angers (2012) suggested these aggregate formations may be signs of early pedogenesis. Aggregate formation also contributes to reducing bulk density, increasing porosity and the increased root growth (Asensio et al. 2013). This continues to contribute to the organic carbon helping form more stable aggregates (Asensio et al. 2013). In agricultural soils, increased biosolids applications have shown increased aggregate stability, depending on soil texture (García-Orenes et al. 2005). Increased aggregation found in biosolids amended soils also contributes to increases in water infiltration and increased water holding capacity as more macropores are formed

(Aggelides and Londra 2000; Asada et al. 2012; Larney and Angers 2012; Sun and Lu 2014).

Water retention curves and maximum water holding capacities often increase with biosolids, but this does not necessarily result in an increase in plant available water (Gardner et al. 2010; Tripathy et al. 2003; Shrestha and Lal 2006). Gardner et al. (2010) found that water retention curves shifter to a high water content due to the biosolids, but the plant available water was not greatly altered. This means that while more water is stored in the organic matter of the biosolids amended soil it does not mean that is available for plants to use.

Hydraulic conductivity (K_{sat}) is the ease at which water can move through the soil matrix. K_{sat} tends to increase with coarser textured soils and soil with high macroporosity (Rawls et al. 1998). Therefore an increase in aggregate stability, and increased water retention due to biosolids application can result in small increases in K_{sat} in fine textured soils, over no amendment (Harris and Meghara 2001). In coarse soils with a high K_{sat} the addition or organic matter may slow water movement as the organic matter fills macropores, decreasing K_{sat} (Schneider et al. 2009; Larney and Angers 2012). In both scenarios, K_{sat} is improved.

Vegetation

Biosolids and organic matter application can significantly improve biomass production by addressing site limitations, such as nutrient availability and water availability (Jong et al. 1983; Gardner et al. 2011; Sun and Lu 2014). The magnitude of those impacts is site specific. Vegetation responses differ across sites because biosolids additions influence nutrient and metal uptake and growth in slightly different ways, depending on the physical and chemical state of the soil or tailings (Evanylo et al. 2005). For example, elevated P concentrations can reduce Pb uptake in plants (Brown et al. 2003; Scheckel and Ryan 2004), or increased Zn concentrations can reduce Cd uptake (Basta et al. 2004). Biosolids have shown to increase growth on mudflats in China, in greenhouse experiments with tailings in Chile, and field experiments where no vegetation established before biosolids amendments (Bai et al. 2013; Santibáñez et al. 2008; Gardner et al. 2012; Brown et al. 2007; Brown et al. 2003; Brown et al. 2014). While increases in biomass have been well documented, some studies show over longer term (16 years) biosolids treated overburden does not differ from a control without any amendments (Bendfeldt et al. 2001). This illustrates the need for further research to better understand how biosolids may impact long term vegetation growth on mine tailings.

Risks

There are risks and concerns surrounding the use of biosolids. Items of concern include excess nutrient loading, metal loading, addition of pharmaceuticals and personal care products (PPCP's) and how those parameters transport into ground and surface water, uptake into plants and the subsequent intake by wildlife and humans (Bright and Healey 2003; CEC 2002; Gardner et al. 2003; Zenker et al. 2014). In addition, biosolids may provide more nutrients than desirable plant species require, promoting establishment of weedy species (Paschke et al. 2005). Public opinion of biosolids also limits its use because it can be viewed as an undesirable waste (Larney and Angers 2012).

Biosolids application can potentially increase heavy metals leaching into groundwater sources. Some metals have shown to have greater risk of leaching, including Cd, Ni and Zn (Richards et al. 2000; Gardner et al. 2011; Yang et al. 2014; McCall et al. 2015). The level of risk generated by specific metals is also dependent on the site specific characteristics (Zhang et al. 2012). In an agricultural setting with annual biosolids applications Yang et al. (2014) reported Cr was found throughout the soil depth, possibly indicating leaching. Gardner et al. (2011) reported Zn leaching on copper-molybdenum tailings treated with biosolids. Zhang et al. (2012) found the leaching of Cd, Ni, and Cr from mine wastes mixed with biosolids and fly ash exceeded European drinking water standards. Some metals have found to increase with biosolids application, but do not increase at depth suggesting a low leaching risk. Yang et al. (2014) reported this with Cd, Cu, Mo, Pb, Sb, Sn and Zn and Gardner et al. (2011) reported this with B, Pb and Cr. There is the potential that metals can form precipitates or bind with anions, preventing the leaching ability of a given metal, such as Pb stabilization using high phosphorus amendments (Yang et al. 2014; Kumpiene et al. 2008). While increases to metals is reported with biosolids application some studies conclude that metal movement in amended soils is negligible and poses little risk to the environment (McCall et al. 2015).

Concerns have been raised about metal accumulation in plants transferring to wildlife or humans. This risk varies by parameter, some metals cause phytotoxicity before zootoxicity, therefore the elevated concentrations may not be transferred to the next trophic level (Evanylo et al. 2005). Evanylo et al. (2005) used Cu, Ni, and Zn as example of metals that are readily taken up by plants and reach phytotoxic levels before reaching levels potentially causing harm to wildlife that may consume them. Santibáñez et al. (2008) also reported concentrations of Cu, Ni, and Zn with biosolids application that reached levels of concern for plant survival, but remained far below levels detrimental to animals. This is referred to as the “soil-plant-barrier” (Basta et al. 2004; Evanylo et al. 2005). This occurs when elevated metals cannot be taken up into plants due to chemical process in the soil, or if taken up into a plant causes phytotoxicity before reaching levels that would cause zootoxicity preventing its transfer higher into the food chain (Basta et al. 2004). Many plant species accumulate metals in tissues not commonly foraged, such as in the roots of some ryegrass species and willows, preventing zootoxicity (Santibáñez et al. 2008; Boyter et al. 2009). The organic matter in the biosolids can also reduce metal uptake in plants. Cationic metals tend to bind with the negatively charged organic matter, making them less available to plants (Kumpiene et al. 2008; Evanylo et al. 2005). In this case increased biosolids may decrease uptake of some metals (Evanylo et al. 2005).

While heavy metal accumulation due to biosolids is well studied in agriculture setting with vegetable crops and livestock, the transfer to other organisms such as wild ungulates past the soil-plant barrier, is not as well documented. Heavy metals of particular concern include Pb, Hg, Cd, and Cr (CEC 2002). Generally speaking, Pb and Cr form complexes in terrestrial systems binding with particles and reducing their bioavailability. Contrastingly, Hg and Cd are relatively more bioavailable (CEC 2002). In two cattle experiments, one on smelter wastes with elevated Pb and Cd another on mine tailings with elevated Mo, elevated levels of metals in the feed and forage did not result in adverse impacts on the livestock, even though guidelines for metal concentrations in feed were exceeded (Stuczynski et al. 2005; Gardner et al. 1996). Bourioug et al. (2015) examined the impact of biosolids on metal concentrations in snails with two biosolids applications of 3 t ha^{-1} in a tree plantation. After 28 days of exposure, snails showed signs of accumulation of Cd and Cu due to increased concentrations in litter consumed by the snails but, mortality remained below 1%. While metal transfer has been documented in a few studies, the overall effect on wildlife is not well understood.

Nutrients that tend to be of specific concern and research interest include nitrate (NO_3) and phosphate (PO_4) due to their high mobility (Elliot et al. 2002; Pond et al. 2005; Esteller et al. 2009; Marofi et al. 2015), and potential to contaminate water (Shober et al. 1996; Elliot et al. 2002; Santibáñez et al. 2007; Cogger et al. 2013). The cation, ammonium (NH_4), does not leach as readily as the anion NO_3 because NH_4 is tightly bound to negatively charged surfaces common on organic matter (Santibáñez et al. 2007), and therefore would be less of a leaching risk with the use of biosolids. In contrast, negatively charged NO_3 is a much more mobile form of N (McCall et al. 2015), and more easily leached through biosolids amended substrate (Larney and Angers 2012). Phosphorus' mobility tends to be more complex, as it can adsorb with soil particles (Marofi et al. 2015). From 41 biosolids originating from different facilities, Brandt's et al. (2004) results showed biosolids had higher total P levels than manure, but water extractable P decreased. Lower extractable P may be attributed to the dewatering process of biosolids manufacturing that may remove soluble forms (Brandt et al. 2004). Plant establishment and applying biosolids in low precipitation conditions may also reduce the risk of NO_3 and PO_4 leaching (Santibáñez et al. 2008; Santibáñez et al. 2007; Larney and Angers 2012).

Biosolids may contain pharmaceuticals and personal care products, collectively known as PPCP's, which may pose a risk to environment. PPCP's in biosolids can include antimicrobials used in soaps in lotions (i.e. triclocarbon and triclosan), antihistamines (i.e. diphenhydramine), hormones (i.e. testosterone), antibiotics (i.e. ciprofloxacin and norfloxin), and many others (Prosser and Sibley 2015). These compounds generally target biological systems and can adversely impact the physiology and behavior of organisms (Murdoch 2015). Hence, concerns exist about their accumulation in plants, the potential bioaccumulation then into the next trophic levels of organisms, their leaching potential into water bodies and impact on aquatic systems, and the potential to adversely impact soil microbial populations. Although, some research shows the benefit of biosolids additions can mitigate these risks (Wu et al. 2012; Morais et al. 2013; Park et al. 2013; Prosser and Sibley 2015). The examination of PPCP's is beyond the scope of the current thesis and isn't further discussed.

The final potential risk associated with the use of biosolids is the encouragement of invasive plant species. Invasive or weedy species are more competitive and better adapted to take advantage of the high nutrient soils created with biosolids compared to native species which evolved in lower nutrient environments (Brown et al. 2007). Paschke et al. (2005) attributes increased invasion of weedy species to increased N, which also results in a decrease in plant diversity and a decrease in perennial grass species. Contrastingly, Evanylo et al. (2005) showed insignificant weed encroachment on seeded biosolids amended sites, but invasion did occur where less vigorous species where seeded. Larney and Agner's (2012) review assessed the relationship between biosolid application and increased invasives. They report that biosolids application can lead increased invasives, but that outcome is not universal.

While there are real risks that need to be mitigated, proper management can limit adverse effects on the environment. For example, continuous applications of biosolids are more at risk for accumulation of metals and PPCP's in plants and soil compared to one-time applications (Yang et al. 2014). Certain management practices can be used to control the potential of leaching and contamination, such as avoiding application in adverse weather conditions and establishing vegetation

(Santibáñez et al. 2008; Santibáñez et al. 2007; Larney and Angers 2012). When using biosolids, the benefits must outweigh the costs of biosolids use (Bright and Healey 2003). To determine if the benefit is greater, more research needs to be done in varying climates and soil conditions (Larney and Angers 2012).

KNOWLEDGE GAPS

Past work on biosolids has focused on the short term (<10 years) impacts on mine wastes, longer term (>10 years) studies on biosolids mixed with other amendments such as lime or wood chips, and impacts of multiple applications in agriculture (e.g. Santibáñez et al. 2008; Boyter et al. 2009; Gardner et al. 2011; Yang et al. 2014; Basta et al. 2016). Where there has been longer term studies, they have mainly focused on agricultural crops or forms of mine wastes other than tailings (e.g. Bendfeldt et al. 2001; Yang et al. 2014; Sidhu et al. 2016). In many cases additions of lime or marble were added to increase the low pH of the mine substrate. There is less information on the long term impacts of a one-time biosolids application on alkaline mine tailings at different rates, such as the current study. This study becomes even more valuable as the two tailings ponds have different physical parameters but the same experimental conditions are examined.

There are theories on what happens to metals and nutrients over a longer time frame but there is insufficient research on the topic to clearly understand the potential changes. Long term studies on tailings ponds are necessary to examine the fate the organic matter, and how metal availability will change; potentially increasing phytotoxicity in plants (McBride 1995). One theory is the benefit of biosolids may only be short term. As organic matter decomposes the site will again become nutrient deficient, compromising its ability to be sustainable (McBride 1995). The other hypothesis is the increased vegetation will supply sufficient biomass to continue the nutrient cycling process without the need for additional amendments. Because of these long term uncertainties, further research is needed.

One of the biggest questions in reclamation is: how will reclaimed ecosystems function through time and what are the mechanisms of soil genesis (Larney and

Angers 2012)? Physical improvements in agriculture soils and short term improvements in tailings after biosolids amendments have been seen (Gardner et al. 2010; Zanuzzi et al. 2009; Tripathy et al.2003; Wallace et al. 2009), but the continuing improvement of tailings through time remain less understood because of the complexity of the mechanisms leading to soil genesis and a lack of long term examination. The current study provides a long term time frame for soil development on a tailings pond after a one-time biosolids application where nutrients, metals and biomass are examined 17 years after biosolids application, and physical soil development is examined 18 year post application.

RESEARCH GOALS

While there is a lot of information on biosolids and information related to the proposed study, none have fully combined all the aspects that will be examined over a relatively long term. This project provides the opportunity to look at chemical and physical changes in two alkaline Cu-Mo tailings ponds, very close in proximity, with different moisture contents and textures, under the same experimental conditions.

Examining soil chemistry and physical parameters will provide a broad picture of how the tailings are developing over time due to biosolids application. The objectives of this study were to examine the effects of a one-time biosolids application in 1998, at rates between 0-250 Mg ha^{-1}, on a sand and silt loam tailings storage facility, on:

1. Metals and nutrients 17 years after application. This was done by examining the total and available nutrients and metal concentrations between 2000 and 2015, as well as across different application rates, from 0 to 250 Mg ha^{-1}, in 2015.

2. Physical parameters 18 years after application. This was done by examining changes between treatments and tailings texture in 2016. The specific parameters examined included biomass, litter, aggregate stability, water retention, saturated hydraulic conductivity and bulk density. Biomass and bulk density were also examined between 2000 and 2016.

The hypothesis is that 17 years after biosolids application, increasing application rates will demonstrate higher concentrations of macronutrients, higher total metal concentrations, and potentially decreased available fractions. Compared to concentrations in the year 2000, it is expected that some metals and nutrients may have reduced in concentration as the sites reached equilibrium after an influx through biosolids addition. If the site is sustaining itself, if would be expected that carbon and nitrogen concentrations will be similar or will have increased from 2000 to 2015. It is also expected biomass, litter, aggregate stability, volumetric water content, saturated hydraulic conductivity will increase in higher applications, and bulk density will decrease in higher applications. If this hypothesis is true than this study may provide evidence that biosolids can improve soil parameters on alkaline tailings ponds, and promote a self-sustaining trajectory.

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Chapter 2. INFLUENCE OF A ONE-TIME BIOSOLIDS APPLICATION ON ELEMENTAL AND NUTRIENT CONCENTRATIONS ON TAILINGS AND VEGETATION

INTRODUCTION

Reclamation and revegetation after hard rock mining presents many large scale challenges not faced in other fields of ecosystem restoration. Mining creates waste products such as waste rock dumps and tailings that create new topography and hydrology compared to the surrounding undisturbed area (Shrestha and Lal 2006). These materials are also almost entirely made from rock, essentially creating a new geological time zero that needs to be productive on an operational time scale. Mine waste products such as tailings also create site specific challenges. These can include poor soil structure, poor hydrological function, high metal concentrations, no nutrients, lack of soil microbes, and a complete lack of organic matter (Brown et al. 2003; Gardner et al. 2011). In many cases these limitations need to be address before vegetation will establish on these sites (Gardner et al. 2011).

The overarching goal of reclamation activities is to create a productive, self-perpetuating and self-sustaining ecosystem which requires site limitations to be addressed. Solutions such as fertilizers do not address all the site limitations, and usually require annual inputs to sustain vegetation (Gardner et al. 2011). Organic amendments, such as biosolids, can provide nutrients and improve soil structure, bulk density, aggregate stability, hydrological function, soil microbial status and add organic matter (Aggelides and Londra 2000; Wallace et al. 2009; Gardner et al. 2010; Gardner et al. 2011). This has led to the successful revegetation of many mining waste products not previously sustaining vegetation (Santibáñez et al. 2007; Gardner et al. 2011).

The use of biosolids in mine revegetation is also a benefit to the waste water treatment stream. Biosolids are treated municipal sewage sludge, which provides a continuous, relatively low cost source of organic matter and nutrients. The land application also diverts biosolids from being disposed of in the ocean or landfills, wasting nutrients and energy (Bright and Healey 2003; Fytili and Zabaniotou 2008).

Biosolids have also been associated with trace metal loading and nutrient leaching. This concern is associated with ground water contamination and high metals in forage (Boyter et al. 2009; Gardner et al. 2011). Although many studies have found that with proper management, the risk of nutrient leaching and metal loadings are not ecologically significant, and pose minute risk (Rate et al. 2004; McCall et al. 2015; Basta et al. 2016). Additionally, mine reclamation usually involves one-time applications opposed to multiple applications over multiple years seen in agricultural uses.

While these benefits and risks associated with biosolids have been examined in different contexts – laboratory (Santibáñez et al. 2007; Zhang et al. 2012; Marofi et al. 2015; Sidhu et al. 2016), agricultural (Shober et al. 1996; Esteller et al. 2009; Haney et al. 2015), overburden and other mine related wastes (Akala and Lal 2001; Bendfeldt et al. 2001; Evanylo et al. 2005; Stuczynski et al. 2005), or short term tailings studies (<5 years) (e.g. Brown et al. 2007; Santibáñez et al. 2007; Wallace et al. 2009; Zanuzzi et al. 2009; Gardner et al. 2011) – fewer studies have examined the effect of a one-time biosolids application on the long term response of metals and nutrients in tailings reclamation. It is the object of this study to examine the effects of a one-time biosolids application in 1998, at different loading rates, on metals and nutrients 17 years after application on two texturally different tailings storage facilities (TSF). This was done by examining the total and available nutrients and metal concentrations between 2000 and 2015, as well as across different application rates, from 0 to 250 Mg ha^{-1}, on each a sand (S) and a silt loam (SiL) tailings storage facility(TSF).

METHODS

Study Site

The study sites were located at Teck Highland Valley Copper (HVC), an open pit copper mine located in British Columbia, Canada, on the Thompson Plateau physiographical subdivision at 50°28’23.22”N, and 121°01’18.50W”. The mine is located on the granite rock of the Guichon Creek Batholith containing porphyry copper and copper-molybdenum, calc-alkaline deposits with ore grades approximately 0.40 to 0.45% copper (Bergey 2009).

Field experiments were conducted on two tailings storage facilities (TSF), Trojan and Bethlehem tailings. Trojan tailings are located at 1442 m above sea level and are a sand texture (S). Bethlehem tailings are located at 1481 meters above sea level and are a silt loam texture (SiL). These TSFs are directly adjacent to each other and both consist of the waste material of milling granite rock containing 60% plagioclase, 10% potassium feldspar and 10% quartz (Gardner et al. 2010). The remaining 20% at the sand TSF is biotite, calcite, gypsum and other minerals, and at the silt loam TSF is hornblende and other minerals (Gardner et al. 2010). Both tailings TSFs are considered alkaline. The sand TSF had a mean pH of 8.33 and the silt loam TSF had a mean pH of 8.09, on unamended tailings in 2015.

On average these TSF receive 346 mm of precipitation annually and have a daily average temperature of -6°C (Figure 2.1). Between May and September, the 2015 daily temperature was 20°C, exceeding the historical average of 12°C. Total precipitation in the same time period was 155 mm, similar to the average of 159 mm. Overall, the year sampling took place for this study was warmer than average climate normals (Figure 2.1).

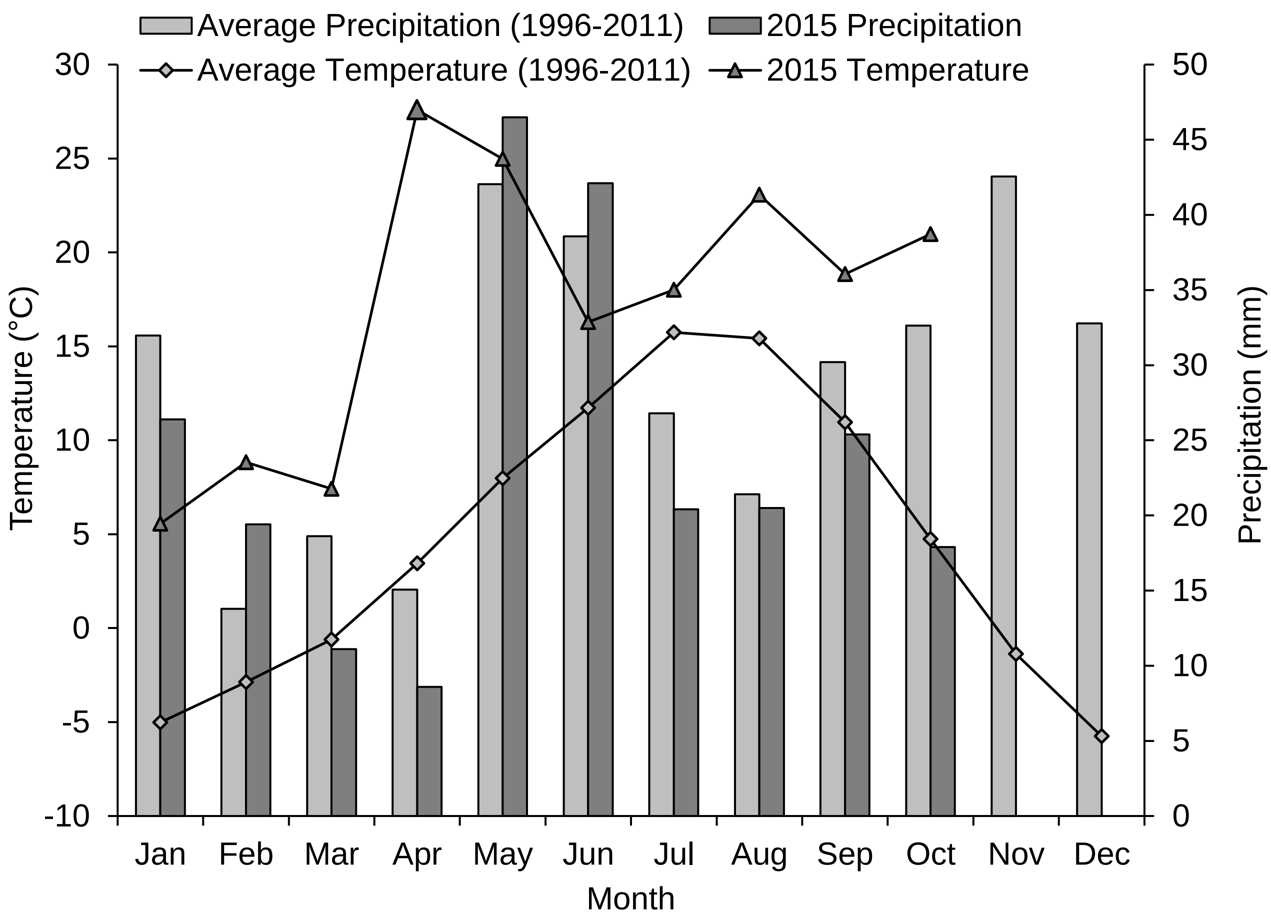

Figure 2.1. Climograph displaying average total precipitation (mm) and temperature (°C) from 1996-2011. Total precipitation and average monthly temperatures are also displayed for 2015. No data for November and December 2015 was available.

Experimental Design

Experimental treatment plots were established on both TSF's July 1998 in a randomized complete block design and are described in Gardner et al. (2010) and Gardner et al. (2011). Treatments consisted of one time applications of biosolids at 50, 100, 150, 200 and 250 Mg ha^{-2} (B50, B100, B150, B200 and B250), a one-time fertilizer treatment (F0) and a control treatment (C0). Each treatment from was applied in a 7x3 meter plot. Each block consisted of a row of randomized treatments, separated by a 0.5 meter buffer, and rows were separated by a 1 meter buffer. This created 8 treatment replicates on each TSF (Figure 2.2). In 2015, treatment plots were reduced to 5x2 m plots to reduce edge effects and vegetation drift along the perimeter of each plot.

Trojan Tailings North (Sand TSF, site A)

Block 1	B200	Control	B50	B250	B150	B100	Fertilizer
Block 2	Fertilizer	B250	B150	B200	B50	Control	B100
Block 3	B50	Fertilizer	B100	B200	B0	B250	B150
Block 4	B250	Control	B150	Fertilizer	B100	B200	B50

Trojan Tailings South (Sand TSF, site B)

Block 5	B250	B50	B150	B200	Control	Fertilizer	B100
Block 6	Control	B100	Fertilizer	B150	B250	B200	B50
Block 7	B50	B250	B100	B0	B150	Fertilizer	B200
Block 8	Fertilizer	B200	B150	Control	B50	B100	B250

Bethlehem Main Tailings South (Silt Loam TSF, site C)

Block 1	Control	B200	B250	Fertilizer	B50	B150	B100
Block 2	B100	B50	Control	B150	B250	B200	Fertilizer
Block 3	B250	B150	B200	B0	Fertilizer	B100	B50
Block 4	B50	Fertilizer	B100	B150	B200	Control	B250

Bethlehem Main Tailings (Silt Loam TSF, site D)

Block 5	B100	Fertilizer	B200	Control	B250	B50	B150
Block 6	Control	B150	B250	B200	B100	Fertilizer	B50
Block 7	B250	B200	B50	Fertilizer	B150	B100	Control
Block 8	Fertilizer	B50	B150	B250	B200	Control	B100

Figure 2.2. Overview of complete block design on the two tailings storage facilities. In the original 1998 design, treatment plots were 7x3 m, blocks were originally separated by 1 m buffer strips and treatment plots by 0.5 m strips.

Biosolids treatments consisted of class B biosolids (OMRR 2002) sourced from Metro Vancouver. Application rates applications were based on dry weight per volume determined before application (Gardner et al. 2010). These treatments were applied in August 1998 with the use of all-terrain vehicle, shovels and rakes, and two weeks later rototilled into the top 15 cm.

In June 1999 inorganic fertilizer treatments were manually broadcasted, but not incorporated. Application rates were based on total nitrogen, phosphorus, potassium, zinc and boron concentrations found in B150 treatments the previous September. The resulting fertilizer amendment was 87 kg ha^{-1} ammonium nitrate (34.5-0-0), 111 kg ha^{-1} triple superphosphate (0-45-0), 83 kg ha^{-1}potassium chloride (0-0-60) and a mineral mix containing 0.5 kg ha^{-1} zinc chloride (99.9%) and 21 kg ha^{-1} granular B (14%) (Gardner et al. 2010). Concurrent to the fertilizer treatments, all treatments were seeded with an agronomic seed mix. This mix consisted of 33.2% pubescent wheatgrass (*Agropyron trichophorum* (Link) Richt.), 7.5% orchard grass (*Dactylis glomerata* L.), 4.0% creeping red fescue (*Festuca rubra* L. var. rubra), 14.7% Russian wild rye grass (*Elymus junceus* Fisch.), 34.6% alfalfa (*Medicago sativa* L.) and 5.9% alsike clover (*Trifolium hybridum* L.) (Gardner et al. 2010).

Data Collection

Soil sampling in 2015 reflected the methods found in Gardner et al. (2010). The plot sizes were reduced from 7x3 meters to 5x2 to reduce edge effects. Vegetation encroachment and blown in sediments were identified on the edges of many plots. Soil sampling then took place September 2015.

A composite soil sample, consisting of 10 random subsamples, was taken from each treatment plot. Subsamples were collected using a soil probe down to a depth of 30cm. These subsamples were then split into 0-15 cm samples (depth 1) and 16-30 cm samples (depth 2). Once all subsamples in a treatment plot were collected, they were homogenized, air dried and sieved to 2 mm.

Laboratory Analysis

For details on laboratory analysis on historical soil samples see Gardner et al. (2010). Samples collected in 2015 were analysed using the same methodologies by an accredited laboratory (The Standards Council of Canada, The Canadian Association for Laboratory Accreditation and SAI Global). Metals and nutrients that were examined, and the methodology are listed in Table 2.1. These metals included total boron (B), chromium (Cr), copper (Cu), iron (Fe), molybdenum (Mo), manganese (Mn), nickel (Ni), lead (Pb), zinc (Zn) and available Cu, Fe, Mo, Mn, and Zn. Nutrients included total carbon (C), potassium (K), magnesium (Mg), nitrogen (N), phosphorus (P) and available K, ammonium (NH_4), nitrate (NO_3), and P. Plant available fractions were determined using diethylenetriaminepentaacetic acid digestion (DTPA) methods.

Table 2.1. Metal and nutrient parameters and their respective analytical techniques.

Category	Parameter	Analytical Technique	Reference
Total Metals	Fe, Mn	ICP/OES[1]	(Austin 2015a) and EPA 6010C
	Cr, Cu, Pb, Mo, Ni, Zn	ICP-MS[2]	(Austin 2015a) and EPA 6020A
	B	ICP/OES	(Austin 2015a) and EPA 6010C
Available Metals	Cu, Fe, Mn, Mo Zn	ICP-OES	(Jones 2001)
Total Nutrients	C	Combustion	(ASTM 2013)
	N	Combustion	(ASTM 2011)
	K, Mg, P	ICP/OES	(Austin 2015a) and EPA 6010C
Available Nutrients	P, K*	Discrete analyzer, *ICP/OES	(Austin 2015a) and EPA 6010C
	NH_4	Discrete analyzer	(Stieg et al. 1997)
	NO_3	Discrete analyzer	(Alberta Agriculture 1988)
Other	pH 1:2	pH meter	(Austin 2015b)

[1] inductively coupled plasma optical emission spectrometry
[2] Inductively coupled plasma mass spectrometry

Statistical Analysis

A 4-way ANOVA was constructed with treatment, year, TSF, and depth. For most parameters, the variables TSF and depth were significant ($p<0.05$), with the exception of total B, Cr and available Mo and NO_3. These results justified a 2-way ANOVA for each TSF at each depth. Therefore, for each parameter a 2-way ANOVA for the S TSF at depth 1 and 2, and the SiL TSF at depth1 and 2 were conducted, testing for treatment, year, and treatment-year interactions.

Statistical assumptions of homogeneity of variance and normality were tested using the Fligner-Killeen test and Shapiro-Wilks tests. Some parameters were transformed using a square root transformation or a logarithmic transformation to meet these assumptions (Table 2.2). In some cases data did not meet the assumption of normality, but did meet homogeneity of variance; but normality QQ plots did not appear to deviate far from normality. These parameters were still tested with the 2-way ANOVA. Assumptions test results and associated transformations used for each parameter, as well as QQ plots and residual versus fitted plots, can be found in Appendix 1.

Table 2.2. Parameters that were transformed to meet assumptions of the ANOVA, or failed assumptions with and without transformations.

	Sand TSF		**Silt Loam TSF**	
Transformation	**Depth 1**	**Depth 2**	**Depth 1**	**Depth 2**
Log	Total Fe, P, Zn; Available K, Mn, NH_4, Zn	Available Fe, Zn	Total C, N, Ni, P; Available Cu, Fe, K, Mn, NO_3, NH_4,Zn	Total N, P, Zn; Available K, Mn, Mo, NH_4
Square Root	Total Pb	Available Mn	Total Mo, Available Mo	
Failed assumptions	pH, total C, N; Available NO_3	pH, Total C; Available P	pH, Total Cr, Zn, Available P	pH, Total C, Available NO_3, Zn

To further examine treatment effects, post hoc testing was conducted with a Bonferroni adjustment. This conservative post hoc test was done to account for a high risk of type I error associated with conducting a high number of statistical tests. All statistical analysis was done on R version 3.2.3 “Wooden Christmas-Tree” or version 3.3.3 “Another Canoe”.

RESULTS

Change across Treatment

Fifteen years after biosolids application, there are still clear differences between nutrients and metals examined between the control and biosolids treatments. The detailed results of all ANOVA’s for all parameters can be found in Appendix 2. Most responses to biosolids treatments resulted in increasing trends, with some differences between the two TSFs. Some of the parameters did not change in response to biosolids treatments and can be found in Appendix 2. At both TSF’s and both depths this included total B and K; at the S at both depths it included total Cu and available Mo, in only depth 1 total Mo and Mn, and in only depth 2 total Cr, Fe, Ni, N and available Cu, and Mn; in the SiL at both depths it included total Mn and available Cu, and in only depth 2 total Cr, Cu, Fe, Ni, Pb, Mg and available Mo, and K (Appendix 2). These treatment comparisons are not further discussed as they did result in a significant response to biosolids. The following results across treatments represent means combined from both 2000 and 2015.

Biosolids reduced the available fraction of some metals and reduced pH. In depth 1, available Cu decreased 34% at the S (Table 2.3) and available Mo decreased 55% at the SiL TSF depth 1 (Table 2.4). Mean available Cu concentrations in the S TSF was lower in all biosolids treated plots compared to the control, but hoc testing did not differentiate between any treatment comparisons ($p>0.1$) (Table 2.3). In the S TSF pH was lowest in the B250, depth 1 at 6.48, and highest in depth 2 of the fertilizer treatment at 8.21. At the SiL pH was lowest in depth 1 of the B250 6.84, and highest in depth 2 of the fertilizer treatment at 8.05. A comparison of only 2015 to CCME guidelines for agricultural soils can be found in Appendix 433

Table 2.3. Mean elemental concentrations (ug g^{-1}) (±standard error) in the sand TSF in the control, 50, 150, and 250 Mg ha^{-1} biosolids treatment, and associated significant ($p<0.05$) trend determined from Bonferroni post hoc comparisons. Means are only provided for significant two-way ANOVA results and both 2000 and 2015 combined (n=16, 8 replicates, 2 years).

	0 - 15 cm				16 – 30 cm			
Parameter	Control	Biosolids 50 Mg ha^{-1}	Biosolids 150 Mg ha^{-1}	Biosolids 250 Mg ha^{-1}	Control	Biosolids 50 Mg ha^{-1}	Biosolids 150 Mg ha^{-1}	Biosolids 250 Mg ha^{-1}
Increasing Trend								
Total C	0.29 ± 0.01*	1.1 ± 0.12*	2.2 ± 0.22	3.07 ± 0.26	0.27 ± 0.01*	0.35 ± 0.02	0.56 ± 0.08	0.65 ± 0.12
Total Fe	0.54 ± 0.01*	0.53 ± 0.01*	0.57 ± 0.01*	0.63 ± 0.01	-	-	-	-
Total Mg	0.05 ± 0*	0.06 ± 0*	0.07 ± 0*	0.08 ± 0	0.06 ± 0	0.06 ± 0 *	0.06 ± 0	0.07 ± 0
Total N	0.01 ± 0*	0.1 ± 0.01*	0.23 ± 0.03*	0.33 ± 0.03	-	-	-	-
Total Ni	3.1 ± 0.26*	3.7 ± 0.44*	4.3 ± 0.34	6.1 ± 0.6	-	-	-	-
Total P	249 ±18*	686 ± 79*	1682 ± 177*	2654 ± 219	257 ± 17*	325 ± 11*	496 ± 62	550 ± 62
Total Zn	19 ± 1.4*	33 ± 3.7*	80 ± 5.3*	142 ± 15	14 ± 1.6	15 ± 0.86	29 ± 5	28 ± 5.8
Available K	12 ± 0.92*	18 ± 1.2*	27 ± 2.8	55 ± 21	-	-	-	-
Available Mn	2.5 ± 0.1*	2.4 ± 0.14*	4.5 ± 0.32*	6.4 ± 0.52				
Available NH4	2.1 ± 0.61*	3.2 ± 1.3*	9.5 ± 2.2	9.4 ± 2.1	-	-	-	-
Available NO3	0.58 ± 0.19*	7.2 ± 4.7*	15 ± 4.1	52 ± 4.2	-	-	-	-
Available Zn	1.0 ± 0.12*	4.5 ± 0.69*	13 ± 1.2	21 ± 2.4	0.81 ± 0.13*	1.1 ± 0.19	2.0 ± 0.48	2.6 ± 0.55
Decreasing Trend								
Available Cu	222 ± 31	172 ± 17	180 ± 18	147 ± 15	-	-	-	-
Variable trends					-	-	-	-
Total Cr	18 ± 4.4	15 ± 3.3	20 ± 4.0	25 ± 3.7	-	-	-	-
Available Fe	26 ± 1.5*	21 ± 1.7*	30 ± 1.7	41 ± 5.7	26 ± 1.6	21 ± 1.2	20 ± 0.78	21 ± 0.85
Total Mn	-	-	-	-	296 ± 6.1	294 ± 60.	285 ± 10	297 ± 7.0
Total Mo	-	-	-	-	13 ± 1.3†	21 ± 4.6*†	17 ± 1.7†	12 ± 1.1†

*Post hoc Bonferroni pairwise comparisons show a significant difference from 250 Mg ha^{-1} treatment
†Exceeds CCME guidelines for agricultural soil.

Table 2.4. Mean elemental concentrations (ug g^{-1}) (±standard error) in the silt loam TSF in the control, 50, 150, and 250 Mg ha^{-1} biosolids treatment, and associated significant ($p<0.05$) trend determined from Bonferroni post hoc comparisons. Means are only provided for significant two-way ANOVA results and both 2000 and 2015 combined (n=16, 8 replicates, 2 years

	0 - 15 cm				16 - 30 cm			
Parameter	Control	Biosolids 50 Mg ha^{-1}	Biosolids 150 Mg ha^{-1}	Biosolids 250 Mg ha^{-1}	Control	Biosolids 50 Mg ha^{-1}	Biosolids 150 Mg ha^{-1}	Biosolids 250 Mg ha^{-1}
Increasing Trend								
Total C	0.07 ± 0.04*	1.9 ± 0.17*	4.1 ± 0.25	5.0 ± 0.26	0.57 ± 0.02	0.65 ± 0.02	0.9 ± 0.13	0.89 ± 0.08
Total Cu	773 ± 37†	770 ± 45†	754 ± 40†	876 ± 56†	-	-	-	-
Total Fe	0.74 ± 0.02 *	0.74 ± 0.03*	0.84 ± 0.02	0.89 ±0.04	-	-	-	-
Total Mg	0.13 ± 0.01	0.12 ± 0.01	0.15 ± 0	0.16 ± 0.01	-	-	-	-
Total N	0.03 ± 0*	0.15 ± 0.02*	0.39 ± 0.02	0.51 ± 0.03	0.04 ± 0.01	0.04 ± 0.01	0.05 ± 0.01	0.09 ± 0.03
Total Ni	6.2 ± 0.22*	6.3 ± 0.2*	8.1 ± 0.31	9.7 ± 0.45	-	-	-	-
Total P	354 ± 39*	1160 ± 161*	2811 ± 185	3941 ± 346	309 ± 24	345 ± 19	599 ± 119	567 ± 55
Total Pb	5.9 ± 1.2*	6.6 ± 1.3*	18 ± 1.1	26 ± 2.6	-	-	-	-
Available K	181 ± 23	138 ± 16	202 ± 24	210 ± 36	-	-	-	-
Available Mn	2.6 ± 0.27*	4.6 ± 0.43*	8.0 ± 1.2	12 ± 2.3	2.5 ± 0.22	3.0 ± 0.26	2.7 ± 0.28	3.4 ± 0.21
Available NH4	3.8 ± 4.4*	5.5 ± 1.3	13 ± 2.6	18 ± 4.3	-	-	-	-
Available NO3	2.0 ± 0.75*	11 ± 8.1*	53. ± 18	116 ± 30	-	-	-	-
Available P	16 ± 6.4*	40 ± 6.3*	129 ± 28	189 ± 47	11 ± 2.8	11 ± 2.5	15 ± 1.8	23 ± 6.4
Available Zn	2.4 ± 0.27*	12 ± 1.6*	32 ± 2.6	40 ± 4.7	1.2 ± 0.1	2.3 ± 0.27	3.2 ± 0.39	4.5 ± 0.81
Decreasing Trend								
Available Mo	8.1 ± 0.72*	4.7 ± 0.72	4.5 ± 0.58	3.6 ± 0.47	-	-	-	-
Variable trends								
Total Mo	40 ± 2.3†	30 ± 2.3†	31 ± 2.9†	28 ± 2.1†	18 ± 1.1†	19 ± 1.5†	22 ± 1.3†	24 ± 1.0†
Available Fe	78 ± 5.2	82 ± 6.1	98 ± 6.8	108 ± 13	100 ± 3.8	100 ± 5.2	66 ± 6.0*	90 ±4.6

* Post hoc Bonferroni pairwise comparisons show a significant difference from 250 Mg ha^{-1} treatment

†Exceeds CCME guidelines for agricultural soil.

With increased biosolids applications, both TSF had increases in nutrient concentrations, specifically in depth 1. The increase in concentration from the control to B250 biosolids treatment for the S and SiL in 2015 is given in brackets. In depth 1, total C (959%; 49%), N (3200%; 1600%), P (851%; 1013%) and available NH_4 (358%; 436%), NO_3 (8833%; 5604%) and P (1906%; 1053%) all increased with increased biosolids application (Table 2.3, Table 2.4). Available K also increased significantly at the S (379%) and the SiL (16%), but post hoc testing was not significant for the SiL. Figure 2.3 and 2.4 demonstrate how total C, N and P increase in response to biosolids in the S and SiL TSF in depth 1 (combination of 2000 and 2015). There you can see significant increases in these nutrient concentrations do not significantly increase above B200 in the S and B150 in the SiL TSF. Available NH_4, NO_3, K and P are displayed in Figure 2.5 and 2.6, and demonstrate no significant increases above treatment B200 at both TSF's. These increases represent those nutrients that did not result in a significant year-treatment interaction. Similar increasing trends in depth 2 for total C, P, and available P, were found for both ponds, and total N in SiL but these also displayed significant treatment-year interactions (Appendix 4).

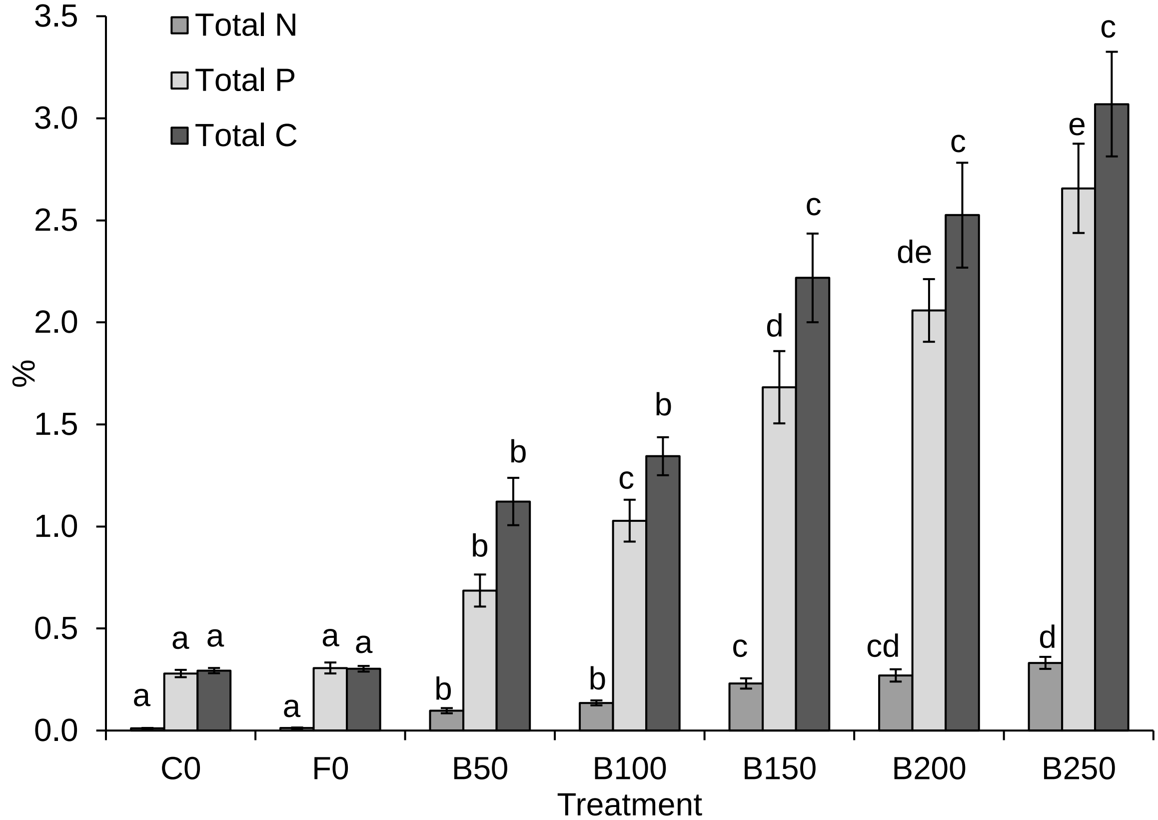

Figure 2.3. Total C, N and P (%) concentration at the sand TSF, in the depth 1. Bars represent means across 2000 and 2015 combined, and error bars represent the standard error of the mean. Letters represent significant differences between treatments for each parameter.

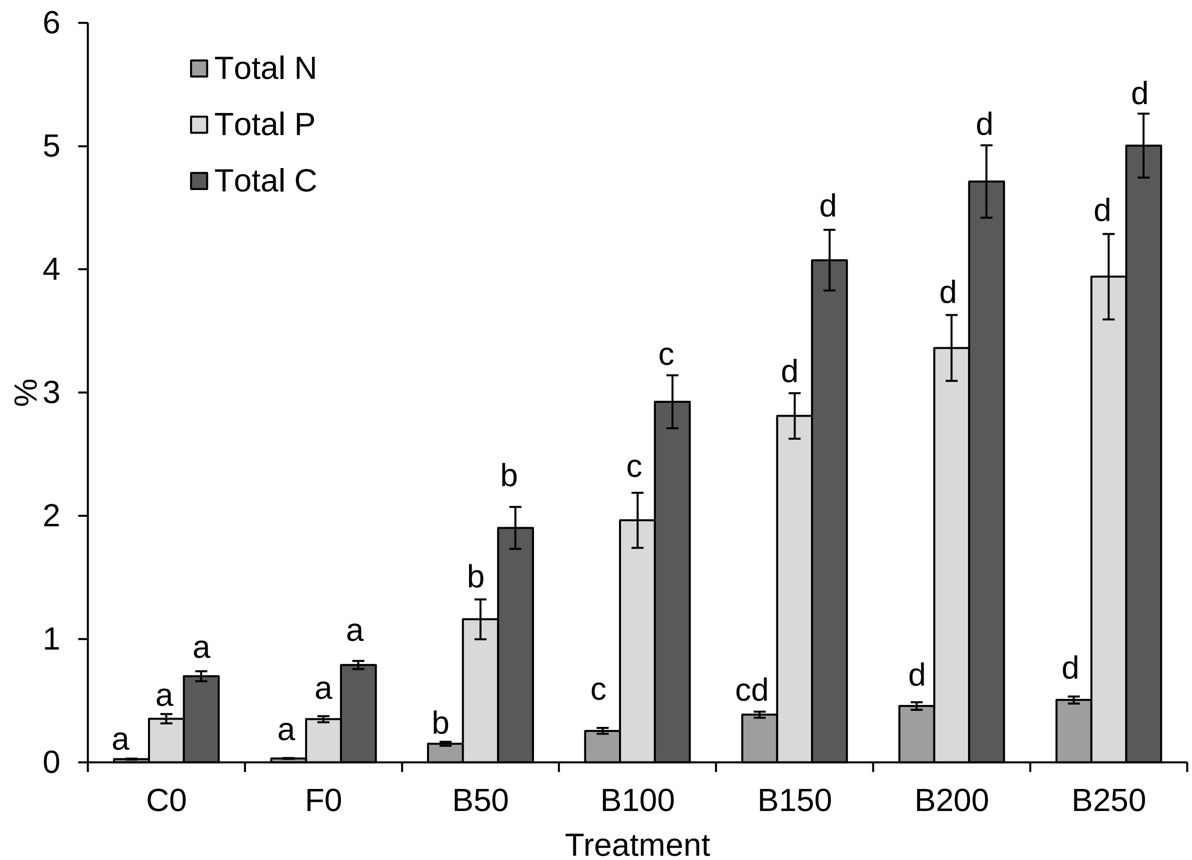

Figure 2.4. Total C, N and P (%) concentration at the silt loam TSF, in depth 1. Bars represent means across 2000 and 2015 combined, and error bars represent the standard error of the mean. Letters represent significant differences between treatments for each parameter

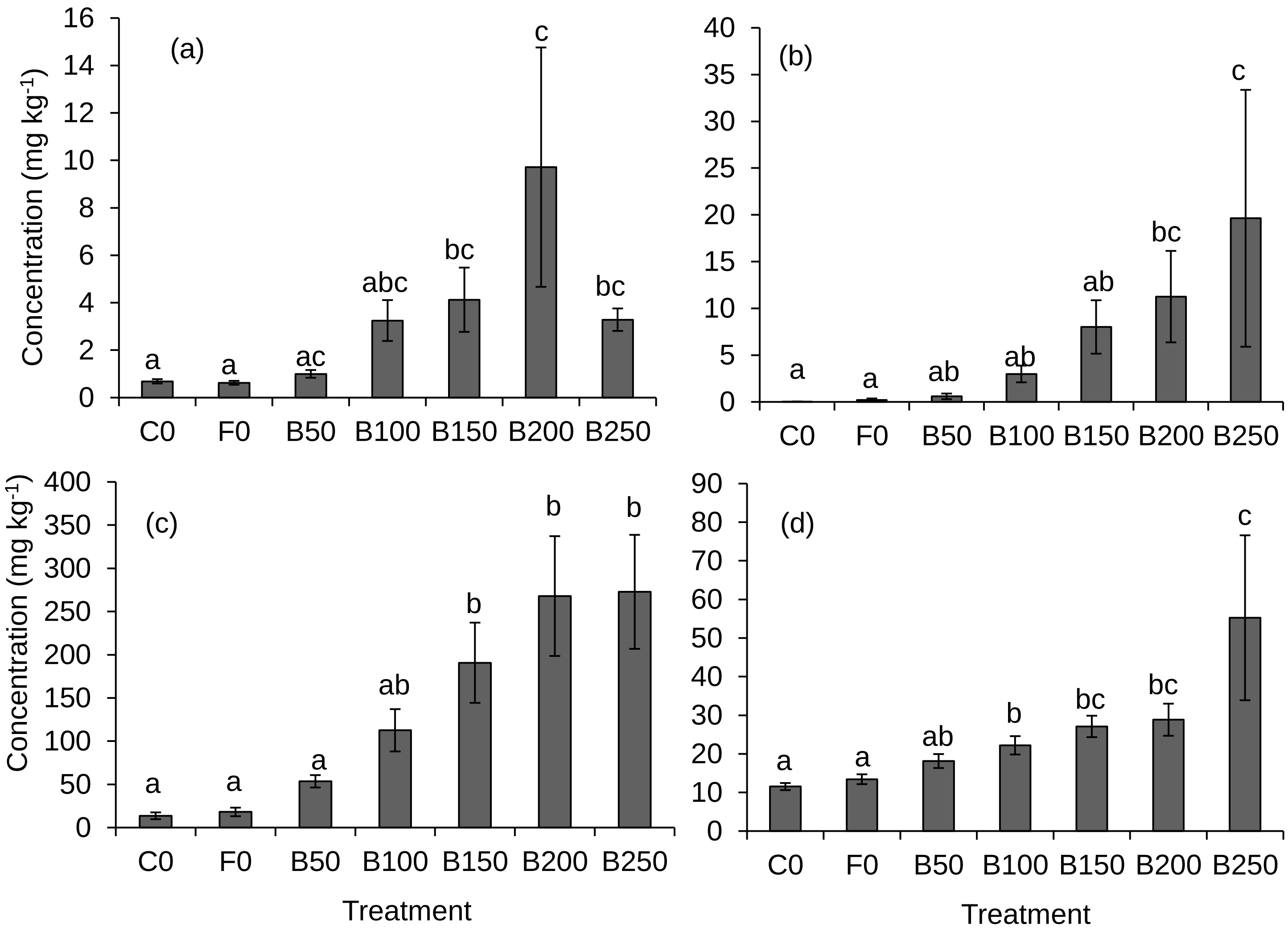

Figure 2.5. (a) Available NH_4, (b) NO_3, (c) P and (d) K concentrations (mg kg^{-1}) in the S TSF, in depth 1. Bars represent means across 2000 and 2015 combined, and error bars represent the standard error of the mean. Letters represent significant differences between treatments for each parameter.

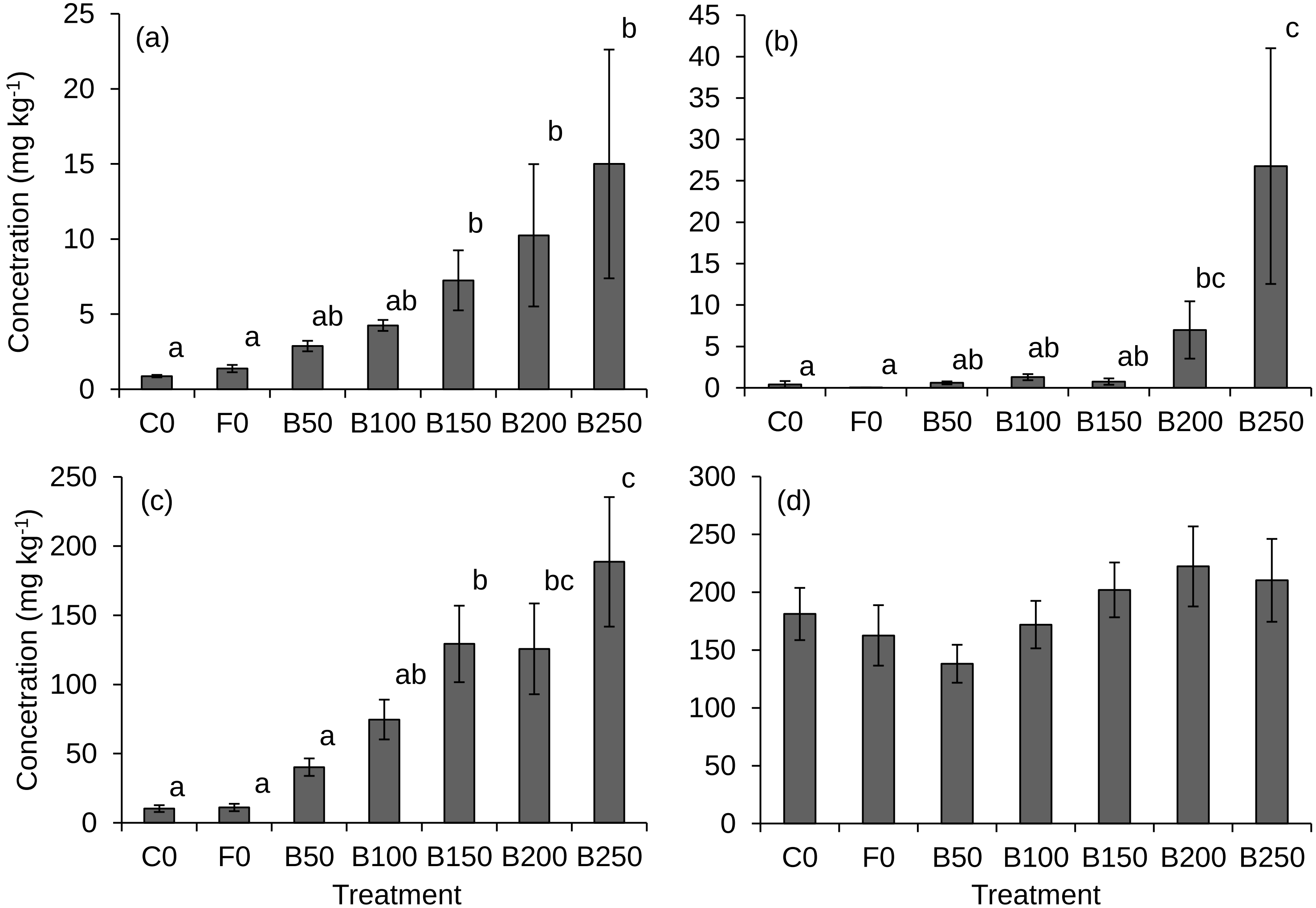

Figure 2.6. (a) Available NH_4, (b) NO_3, (c) P and (d) K concentrations (mg kg^{-1}) in the SiL, depth 1. Bars represent means across 2000 and 2015 combined, and error bars represent the standard error of the mean.

Metals that demonstrated an increase in concentration due to biosolids application at both the S and SiL TSF included total Fe (15%; 20%), Mg (60%; 23%), Ni (98%; 55%), and Zn (1927%; 1596%) in depth 1 (Table 2.5 and 2.6). In addition to these parameters total Zn (92%) increased significantly at the S in depth 1 (Table 2.5). In the SiL, total Cu (13%), Pb (336%) and available Mn (356%) also show an increasing trend in depth 1, but Cu did not result in significant comparisons during post hoc testing (Table 2.5). In depth 2 of the S, total Mg (17%), Zn (92%) and available Zn (216%) increased, but post hoc testing on total Zn was insignificant (Table 2.5). In depth 2 of the SiL, available Mn (356%) and Zn (1596%) increased, but post hoc testing was not significant (Table 2.6). These metals did not display significant treatment year interactions. Additional metals did display a significant treatment effect but also had significant year x treatment interactions. These included total Pb (386%) and available Mn (154%) in the S depth 1; in depth 2 this included total Zn (92%). At the SiL depth 1, total Zn (821%) and Pb (336%); in depth 2 these included total Mo (29%) and Zn (82%) and available Mn (38%) and Zn (149%). Interaction plots can be found in Appendix 4.

Some parameters at the SiL TSF showed variable responses to biosolids, depending on depth or biosolids treatment level (Table 2.4 and 2.5). Available Fe had increasing trend (although post hoc had no significant comparisons), and in depth 2, concentrations in the B150 treatment where significantly lower (post hoc $p<0.05$) then all other treatments including the control. Total Mo in the SiL decreased (post hoc comparisons not significant) in depth 1, and in depth 2 increased (post hoc comparisons not significant) (Table 2.5).

Few means across treatments exceeded CCME guidelines for agricultural soil. Exceedances in 2000 are previously published in Gardner et al. (2011). In both TSF, both depths, and all treatments including the control, total Cu and Mo exceeded guidelines of 36 mg kg^{-1} and 5 mg kg^{-1}, respectively due to high levels in the tailings. In the SiL TSF, in depth 1 total Zn did exceed CCME guidelines for agricultural soils

(200 mg kg^{-1}) in the B200 (μ=231 mg kg^{-1}) and B250 (μ=241 mg kg^{-1}) treatments (Appendix 3). It is worth noting that the agricultural guidelines are the most conservative guidelines, followed by commercial and industrial use guidelines which are set at 360 mg kg^{-1}. Total Zn did not exceed commercial and industrial use CCME guidelines.

Change Over Time

Parameters that showed the following three criteria: 1) increasing or decreasing trends for both year and treatment, 2) insignificant treatment-year interaction, and 3) significant post hoc comparisons, provide a stringent test to show which parameters increase with biosolids application over time. At the S TSF, those include the metals total Cr, Fe, Mg, Ni, C, N, P and available K, NH_4 and NO_3 in depth 1, and total Mo, Mn, Mg and Pb in depth 2. At the SiL TSF, those metals included total Mg, P, available Fe, Mn, Zn, K, NH_4, and NO_3; with no simultaneous year and treatment effect found in depth 2 for any parameter. These are the constituents that changed over the 15 year gap between sampling due to biosolids treatments.

Some nutrients and metals showed an increase between 2000 and 2015 due to biosolids application. These included available K at both TSF; total C (46%), N (500%), and Ni (45%) at the S TSF; and available Mn (102%) and Zn (317%) at the SiL TSF (Table 2.5). No constituents that demonstrated a response to biosolids treatments and had significant year effects, increased in depth 2 from 2000 to 2015 and no metals exceeded CCME guidelines for agricultural soils. These constituent represent increases in concentration over the 17 year period, and did not have a significant treatment effect (Table 2.5)

Some nutrients and metals showed a decrease between 2000 and 2015 due to biosolids application. Those constituents included total Cr, Fe, Mn, Mo, P, Pb and available NO_3 and NH_4 (Table 2.5). Total Cr (-85%) and Fe (-5%) decreased at the S TSF depth 1; in depth 2 total Mn (-8%), Mo (-32%) and Pb (-61%) decreased. In the SiL depth 1, total Mn decreased by 5% (Table 2.5). Even though total Mo demonstrated a decrease over time at the S, this constituent remained above CCME guidelines for agricultural soils due to elevated levels in the tailings. The nutrients total P (S=-30%, SiL=-23%), NO_3 (S=-77% SiL=-94%) and NH_4 (S=-65%, SiL=-52%) decreased over time at both TSFs. These constituents represent decreases in concentration over the 17 year period, and did not have a significant treatment effect (Table 2.5).

In some cases available constituents showed a different trend across time compared to their respective total fractions. This included available Mn which increased in the SiL depth 1, but total concentrations decreased. Total N increased in the S depth 1, but then total NO_3 and NH_4 decreased. These constituents demonstrate a different change over time when either total or available fractions are considered.

Table 2.5. Mean elemental concentrations in tailings in 2000 and 2015 and their mean difference for the sand and silt loam tailings storage facility at two depths (n=16, 8 replicates, 2 years) for those parameters with both a significant year effect and treatment effect, and non-significant interaction. Constituent means that exceed CCME guidelines for agricultural soils are also noted.

	Sand 0-15 cm			Sand 16-30 cm			Silt Loam 0-15		
Parameter	2000 Mean	2015 Mean	Δ Mean	2000 Mean	2015 Mean	Δ Mean	2000 Mean	2015 Mean	Δ Mean
Significant Increasing Trend									
Total C (%)	1.1	1.7	0.51						
Available Mn (mg kg^{-1})							4.1	8.2	4.2
Total N (%)	0.01	0.08	0.05						
Total Ni (mg kg^{-1})	3.1	4.6	1.4						
Available K (mg kg^{-1})	17	33	17				111	258	150
Available Zn (mg kg^{-1})							18	23	57
Significant decreasing Trend									
Total Cr (mg kg^{-1})	34	5.0	-29						
Total Fe (%)	0.58	0.55	-0.03						
Total Mn (mg kg^{-1})				299	274	-25	431	410	-21
Total Mo (mg kg^{-1})				17†	12†	-5.5			
Total P (mg kg^{-1})	1432	1004	-428				2251	1732	-519
Total Pb (mg kg^{-1})				5.6	2.2	-3.4			
Available NO3 (mg kg^{-1})	26	5.8	-20				80	5.3	-75
Available NH4 (mg kg^{-1})	8.9	3.1	-5.8				13	6.0	-6.8
Variable trends									
Total Mg (%)	0.06	0.07	0.01				0.14	0.13	-0.01

†Exceeds CCME guidelines for agricultural soils for total metal concentrations

Those parameters that displayed a significant year and treatment effect, but also had significant interactions are also shown in Appendix 2. In this case, the response to time depends on the treatment considered. At the S TSF, parameters that significantly increased from 2000 to 2015 included available P in depth 1 (354%) and depth 2 (208%), and total C (107%), P (20%), Zn (40%), available Fe (21%) and Zn (312%) in depth 2. At the SiL depth 1 and 2, parameters that significantly increased included total N (17%; 350%) and Zn (30%; 45%);available P (372%) only in depth 1, and total C (33%), P (1%), available Cu (18%) and Zn (158%) in depth 2. Total Pb (S=-28%; SiL=-22%) significantly decreased at both TSF in depth 1. At the SiL TSF total Cr (-47%) in depth 1 also decreased, and total Mo (-19%) and available Mn (-16%) in depth 2. No metal concentrations exceeded CCME guidelines. Available Fe displayed a significant interaction with year ($p<0.05$), likely due to the slightly lowered concentration in the 2015 B100 treatment (S) and B150 (SiL), where all other treatments had higher 2015 concentrations compared to 2000. Year interactions were significant with total Mo because concentrations were higher in 2015 B200 and B250 treatments, and lower in in other biosolids treatments relative to 2000.

Lastly, there were some parameters that show no change with time and no change with treatment. At the S TSF, those included total Cu, Mo tot K and available Mo in depth 1, and total Cu and available Cu, Mo and Mn in depth 2. At the SiL, these were total Fe, K, Mg, Ni, and available Mo in depth 2. These constituents have not change through the experiment, and show no changes in response to biosolid treatments.

DISCUSSION

Overall, the SiL site had higher nutrient and metal concentrations than the S TSF in 2015 biosolids treated plots. Metal concentrations were also greater in the SiL TSF before and after biosolids were applied in 1998, which was attributed to finer texture, greater cation exchange capacity, different milling processes and differences in the ore bodies between the two TSF’s (Gardner et al. 2011). Generally sandy soils are assumed to have a greater potential for leaching, while soils with higher clays and silts have a greater potential and affinity to hold onto components in the soil profile, preventing their downward movement (Carter et al. 2003). Richards et al. (2000) also found higher Cd, Ni, and Zn concentrations in the leachate of a sandy loam soil over a finer textured, silty loam soils treated with biosolids. This may help explain why larger concentrations are found in the SiL TSF over the S, even 15 years after biosolids application.

Most responses over time and across treatments were within the first depth. This has been found in many other studies as well, where the effect of biosolids only exists in the soil surface, or the layer biosolids have been incorporated, with little significant effects at depth (Shober et al. 1996; Albaladejo et al. 2008; Formentini et al. 2015). Other organic amendments tend to show significant effects on the top layer of the soil as well (Yang et al. 2014). Depth 1 had more parameters show significant responses to time and to biosolids treatments than depth 2.

Nutrients

Seventeen years after a one time biosolids application, nutrients were still higher than the control and fertilizer treatments. The control and fertilizer treatments continue to have little to no vegetation cover as seen in previous research on these sites (Gardner et al. 2011), and have very low nutrient concentrations compared to biosolids treatments. Nutrients also show an increase with the increasing biosolids application rates in 2015, but those benefits generally stop increasing at the B150 and B200 treatment. Many short term studies (<10 years) also report that biosolids improve the nutrient status of tailings (Gardner et al. 2010; Forján et al. 2014; Sidhu et al. 2016).

This study provides evidence that biosolids can be used in tailings reclamation to create a self-sustaining, functioning ecosystem. Increases in the total C and N concentrations at both TSF's over time, provides evidence that the sites are not deteriorating but are functioning in a self-sustaining manner that promotes nutrient cycling. The increased carbon from 2000 to 2015 in biosolids treated plots demonstrates that biosolids treated plots may be sequestering more carbon, most likely through increased biomass and root formation (Fornara and Tilman 2008). The ratio of NH_4 to NO_3 may also provide insight into nutrient cycling. If the ratio of NH_4 to NO_3 is greater than 1, this suggests that the ability of microbes to cycle nutrients may be compromised (Brown et al. 2005). In this study, the B250 treatments at both TSF had greater mean NO_3 compared to NH_4, suggesting functional nutrient cycling has been restored (Brown et al. 2005). Available NO_3 and P decreased from 2000 to 2015, but remains higher in biosolids treated plots over the control and fertilizer treatments. This data provides evidence that these two TSF`s are self-sustaining, and have remained productive while the control and fertilizer treatments have not improved from 2000 to 2015.

Results of the current study differ from other studies examining the long term effects of a one-time biosolids application. Bendfeldt et al. (2001) found that 16 years after biosolids were applied to overburden, soil organic matter and total N in biosolids treated plots did not differ from the control. Avery et al. (2017) reported no significant improvement to soil carbon 15 years after biosolids application on degraded rangelands. Ouimet et al. (2015) also reported no statistically significant increase in N and organic C in a forest plantation treated with biosolids 16-19 years prior. These studies did not specifically examine tailings material, which originally had no organic carbon, plant available nutrients and is essentially ground rock. Six et al. (2002) suggested that individual soils inherently have a finite amount of carbon they could store, “soil C saturation”, and if they are close to their C saturation, the accumulation of C will be slowed. Conversely if C is much lower than the potential ability of a soil to retain it, C accumulation may be fast. This may be why the tailings show a continued increase in C 17 years after biosolids application compared to other studies that also report improvements to biomass production (Avery et al. 2017). In contrast, Trlica (2010), reviewed carbon storage at 5 mine sites and found in studies ranging from 1-20 years, biosolids treatments resulted in higher carbon than reclamation activities with fertilizer alone.

The risk of P and NO_3 leaching should be considered with biosolids use as excess loading can impact water bodies. P may have moved downward into the soil profile because total P significantly decreased from 2000 to 2015. Although, no significant changes were found in depth 2 therefore if this was the case enrichment would have happened at deeper in the tailings. While P leaching can be a concern for ground water contamination, some studies suggest that P leaching is negligible and lower than fertilizer or manure amendments (Shober et al. 1996; Richards et al. 2000; Elliot et al. 2002; Sidhu et al. 2016). Incorporating biosolids may reduce leaching and erosion of P (Garcia-Albacete et al. 2016). NO_3 in depth 2 was not collected in 2000, so the same comparisons were not made, but this constituent is known to be water soluble and mobile (Pond et al. 2005; Marofi et al. 2015). NO_3 did decrease in depth 1 from 2000 to 2015, but remains higher in biosolids treated plots. The decline suggests possible downward movement (Pond et al. 2005); plant uptake or volatilization. The tailings are very deep, which may limit the connectivity of surface to ground water, lowering the risk of nutrient contamination. As is important with any organic amendment, site specific planning that takes into consideration the nutrient need of site and proximity to surface or ground water can limit the risk of leaching and contamination of surface and ground water.

Metals

Metals both increased or decreased after biosolids application in this study. Zn was the only parameter to exceed CCME guidelines in 2015 for agricultural soils in the SiL TSF, and only in treatments B200 and B250, but remained below industrial guidelines. Total Zn and Ni demonstrated an increasing trend with biosolids treatments at both the S and SiL TSF from 2000 to 2015. Literature suggests that organic amendments can increase strong organic and residual bonds (Mn and Fe oxides) with Ni and Zn (Shen et al. 2016). Richards et al. (2000) found that the biosolids processing impacted Ni and Zn leaching, with composed biosolids have lower Ni and Zn leaching, and dewatered biosolids having high Ni and Zn leaching. Therefore Ni and Zn may be metals to monitor in the TSFs, but the depth of the tailings may act as a barrier to their leaching into the groundwater.

Total Mo and Cu are the only elements that exceed CCME guidelines on average across both TSF in 2015, but not due to biosolids treatments, but residual levels from the mined rock in the tailings. Cu and Mo are both in very high concentrations and options to remove these metals are not practical, so the management of these materials should focus on immobilization (Park et al. 2011). The addition of organic matter can also increase the cation exchange capacity, creating more binding sites for metals, reducing their mobility. Available Mo did significantly decrease with increased biosolid application in the SiL, and available Cu at the S, in depth 1. Further evidence of immobilization is that total Mo was higher in control and fertilizer treatments in 2000 compared to 2015. Conversely, B200 and B250 treatments in 2015 had very similar levels of total Mo to those found in 2000. This suggests that Mo moved down the profile over time in treatments without biosolids, but remained constant in plots receiving biosolids. This may allow us to cautiously suggest there is a possibility that biosolids could reduce the mobility and bioavailability of these metals.

Interactions show that there is a change in the response to a one time biosolids application over time. Significant interactions between treatment and year (with significant treatment effect) were seen for many parameters, overall demonstrating a stronger response to biosolids application in 2015 compared to 2000. This was seen in the S TSF for total Cr, Pb, available Mn, and P in depth 1, and total C, P Zn, available P, and in depth 2. The concentrations of these elements in 2015 tend to be lower in the control, fertilizer, and sometimes low biosolids applications compared to 2000. Then concentrations tend to be higher in higher biosolids applications compared to 2000. Overall, the response to biosolids applications is more pronounced with a steeper slope, in 2015 compared to 2000 (Appendix 4).These observations are similar at the SiL for total Pb, Zn, in depth 1 and total Mo, Zn, C, P, N, available Mn, and P in depth 2. It is possible there was an increase in mobility of constituents in the control and lower biosolids applications, such as total Mo described above. With lower organic matter, there were fewer opportunities for strong binding between the soil and metals. At higher biosolids applications the higher amount of organic matter increases the available binding sites for these metals, and with additional biomass growth with biosolids this organic matter is maintained. Therefore, as the years have gone by, the control and low biosolids applications have had more leaching, where higher biosolids have a higher capacity to immobilize constituents, making the comparisons between treatments more pronounced (Appendix 4).

pH responded in the opposite fashion than the metals. Compared to 2000, the pH in 2015 was higher (>8) in the control and fertilizer and then lowered at higher biosolids applications to similar, or lower levels than those found in 2000, with the S TSF depth 1 having the lowest pH of 6.46. Normally when pH lowers, metals can become more mobile in soil with the increased H^+ competing for binding sites, but this may be offset by an increase in CEC (not measured). The results of this study do not show high increases in available metals compared to the control and low biosolids applications, as shown with the insignificant post hoc results, except for available P which does significantly increase in 2015 compared to 2000, specifically in higher biosolids applications. Overall the decrease in pH does not appear to be increasing metal leaching, and the benefit of additional binding sites supplied by the organic matter likely counterbalances this drop in pH.

CONCLUSION

The results of this study highlight the long term benefit of increased nutrients with minimal additional metal loading and leaching risk with a one-time biosolids application. Overall Nutrients generally improve with biosolids. There is a decline in NO_3 and total P from 2000 to 2015, but there are higher concentrations as biosolids increase. Metals may increase or decrease, but generally stay below CCME guidelines for agricultural soils. These are the most stringent guidelines and represent levels acceptable to cultivation food for human consumption. The only exceptions are Mo and Cu due to the tailings and Zn in the SiL B200 and B250 treatments. This study shows an application of 150-200 Mg ha^{-1}, similar to Santibáñez's et al. (2008) 200 Mg ha^{-1} recommendation, results in long term benefits to nutrients, while mitigating the risk of metals exceeding CCME guidelines. This is also supported by the fact there is little to no vegetation growth on control and fertilizer treatments. This may be the first long term study on biosolids, strictly focused on their long term impacts on tailings reclamation, with no additional amendment. This study provides evidence that under site specific planning biosolids greatly contribute to the long term sustainability of revegetation and reclamation in mining.

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Chapter 3. INFLUENCE OF BIOSOLIDS ON VEGETATION AND SOIL PHYSICAL CHARACTERISTICS IN TAILINGS 17 YEARS AFTER A ONE TIME APPLICATION

INTRODUCTION

Tailings storage facilities (TSF) are structures that contain the end by-product of hard rock mining, mostly consisting of finely ground rock, water and chemical constituents used during mineral separation. After TSF are no longer in use they are most commonly revegetated, but due to their composition and structure they provide a poor growing medium compared to surrounding undisturbed soils (Shrestha and Lal 2006). These physical limitations can include poor hydrological function, high bulk density, and a lack of organic matter (Brown et al. 2003; Gardner et al. 2011). In many cases these limitations need to be addressed before vegetation will establish on these sites (Gardner et al. 2011). Organic amendments, such as biosolids, can improve soil structure, decrease bulk density, improve aggregate stability, hydrological function, and add organic matter (Aggelides and Londra 2000; Wallace et al. 2009; Gardner et al. 2010; Gardner et al. 2011). This can lead to the successful revegetation of TSF's not previously sustaining vegetation (Santibáñez et al. 2007; Gardner et al. 2011).

Currently, there seems to be little consensus on the long term (>10 years) benefits in terms of physical improvements to mine materials that have received biosolids. Bendfeldt et al. (2001) reported some differences between control and one time biosolids application 16 years prior to aggregate stability and bulk density, but overall concluded the differences didn't justify the use of biosolids. This may have been to the much higher annual precipitation (1150 mm) on the Bendfeldt et al. (2001) site. Avery et al. (2017) reported improved structure, and water holding capacity in a degraded rangeland treated with biosolids 15 years prior in a more similar semi-arid environment, although the medium was not tailings but degraded rangeland soil.

The goal of the current study was to examine physical changes from 2000 to 2016 in 2 texturally different tailings 18 years after a one time biosolids application, as well as differences between treatments in 2016 across treatments and tailings texture. The present study differs from Bendfeldt et al. (2001) and Avery et al. (2017) who examined overburden and a degraded rangeland, respectively, whereas the current study examines mine tailings. Parameters examined across years from 2000 to 2016 included plant biomass, and bulk density. Parameters examined in 2016 across treatments included biomass, litter, aggregate stability, water retention, and saturated hydraulic conductivity.

METHODS

Study Site

The study sites were located at Teck Highland Valley Copper (HVC), an open pit copper mine. It is located in British Columbia, Canada on the Thompson Plateau physiographical subdivision at 50°28’23.22”N, and 121°01’18.50W”. The mine is located on the granite rock of the Guichon Creek Batholith containing porphyry copper and copper-molybdenum, calc-alkaline deposits with ore grades approximately 0.40 to 0.45% copper (Bergey 2009).

Field experiments were conducted on two tailings storage facilities (TSF), Trojan and Bethlehem tailings. Trojan tailings are located at 1442 m above sea level and are a sand texture (S). Bethlehem tailings are located at 1481 meters above sea level and are a silt loam texture (SiL). The center of each pond is approximately 1.5 km a part, with the closest edges being about 300m apart. Both TSF’s are the waste material of milling granite rock containing 60% plagioclase, 10% potassium feldspar and 10% quartz (Gardner et al. 2010). The remaining 20% at the sand TSF is biotite, calcite, gypsum and other minerals, and at the silt loam TSF is hornblende and other minerals (Gardner et al. 2010). Both tailings ponds are considered alkaline. The amended tailings in 2015 had a mean pH of 8.33 and 8.09 at the sand and silt loam TSF, respectively.

On average these TSF receive 346 mm of precipitation and have a daily average temperature of -6°C (Figure 2.1). Between May and September, the 2016 daily temperature was 11.5°C, very close to the historical average of 12°C. Total precipitation in the same time period was 195 mm, which was higher than the 159 mm average. Overall, the year sampling took place for this study temperature was close to the historical average, but precipitation was above average (Figure 3.1).

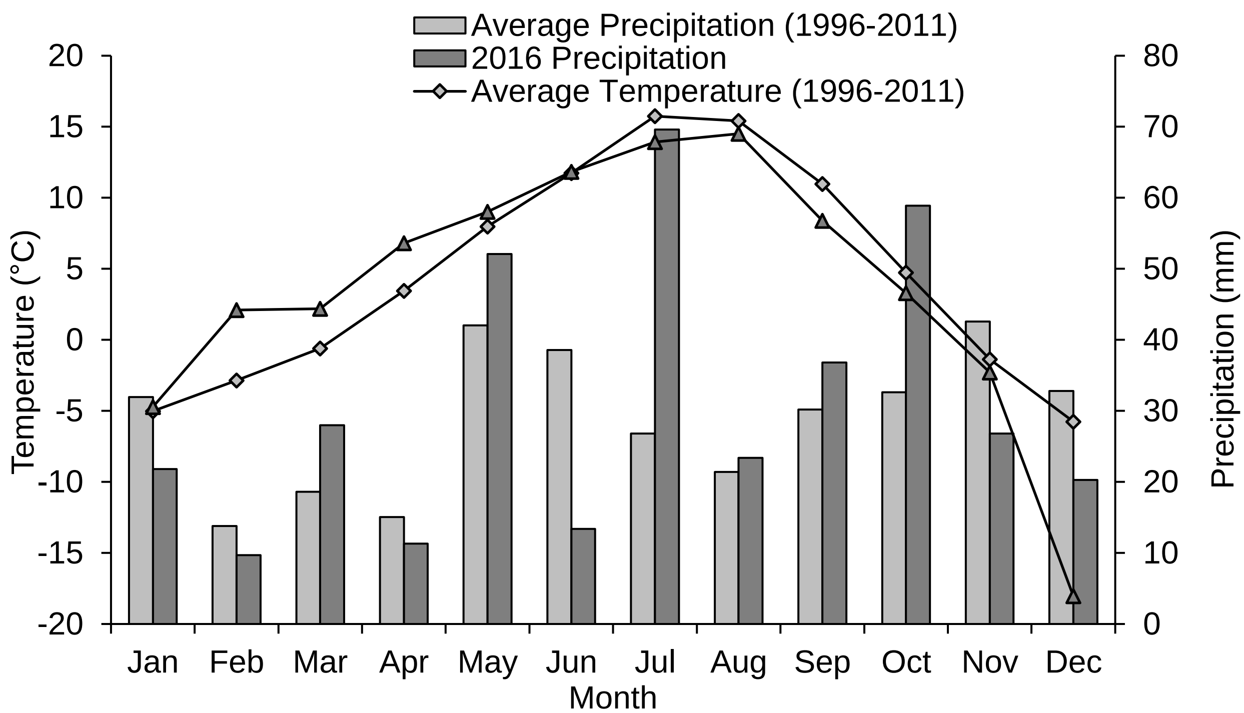

Figure 3.1. Climograph displaying average total precipitation (mm) and temperature (°C) from 1996-2011. Total precipitation and average monthly temperatures are also displayed for 2016.

Experimental Design

Experimental treatment plots were established on both TSF's in July 1998 in a randomized complete block design and are described in Gardner et al. (2010) and Gardner et al. (2011). Treatments consisted of one time applications of biosolids at 50, 100, 150, 200 and 250 Mg ha^{-2} (B50, B100, B150, B200 and B250), a one-time fertilizer treatment and a control treatment. Each treatment from was applied in a 7x3 meter plot. Each block consisted of a row of randomized treatments, separated by a 0.5 meter buffer, and rows were separated by a 1 meter buffer. This created 8 treatment replicates on each TSF (Figure 3.2). In 2015, treatment plots were reduced to 5x2 m plots to reduce edge effects and vegetation drift along the perimeter of each plot.

Biosolids treatments consisted of class B biosolids (OMRR 2002) sourced from Metro Vancouver. Application rates applications were based on dry weight per volume determined before application (Gardner et al. 2010). These treatments were applied in August 1998 with the use of all-terrain vehicle, shovels and rakes, and the two weeks later rototilled into the top 15 cm.

In June 1999 inorganic fertilizer treatments were manually broadcasted, but not incorporated. Application rates were based on total nitrogen, phosphorus, potassium, zinc and boron concentrations found in B150 treatments the previous September. The resulting fertilizer amendment was 87 kg ha^{-1} ammonium nitrate (34.5-0-0), 111 kg ha^{-1} triple superphosphate (0-45-0), 83 kg ha^{-1}potassium chloride (0-0-60) and a mineral mix containing 0.5 kg ha^{-1} zinc chloride (99.9%) and 21 kg ha^{-1} granular B (14%) (Gardner et al. 2010). Concurrent to the fertilizer treatments, all treatments were seeded with an agronomic seed mix. This mix consisted of 33.2% pubescent wheatgrass (*Agropyron trichophorum* (Link) Richt.), 7.5% orchard grass (*Dactylis glomerata* L.), 4.0% creeping red fescue (*Festuca rubra* L. var. rubra), 14.7% Russian wild rye grass (*Elymus junceus* Fisch.), 34.6% alfalfa (*Medicago sativa* L.) and 5.9% alsike clover (*Trifolium hybridum* L.) (Gardner et al. 2010).

Trojan Tailings North (Sand TSF, site A)

Block 1	B200	Control	B50	B250	B150	B100	Fertilizer
Block 2	Fertilizer	B250	B150	B200	B50	Control	B100
Block 3	B50	Fertilizer	B100	B200	B0	B250	B150
Block 4	B250	Control	B150	Fertilizer	B100	B200	B50

Trojan Tailings South (Sand TSF, site B)

Block 5	B250	B50	B150	B200	Control	Fertilizer	B100
Block 6	Control	B100	Fertilizer	B150	B250	B200	B50
Block 7	B50	B250	B100	B0	B150	Fertilizer	B200
Block 8	Fertilizer	B200	B150	Control	B50	B100	B250

Bethlehem Main Tailings South (Silt Loam TSF, site C)

Block 1	Control	B200	B250	Fertilizer	B50	B150	B100
Block 2	B100	B50	Control	B150	B250	B200	Fertilizer
Block 3	B250	B150	B200	B0	Fertilizer	B100	B50
Block 4	B50	Fertilizer	B100	B150	B200	Control	B250

Bethlehem Main Tailings (Silt Loam TSF, site D)

Block 5	B100	Fertilizer	B200	Control	B250	B50	B150
Block 6	Control	B150	B250	B200	B100	Fertilizer	B50
Block 7	B250	B200	B50	Fertilizer	B150	B100	Control
Block 8	Fertilizer	B50	B150	B250	B200	Control	B100

Figure 3.2. Overview of randomized complete block design on the two tailings storage facilities. In the original 1998 design, treatment plots were 7x3 m, blocks were originally separated by 1 m buffer strips and treatment plots by 0.5 m strips.

Data Collection

Biomass and litter samples were collected in 2015 by clipping 10 randomly placed Daubenmire frames within each treatment plot. Biomass clippings were taken as close to the ground as possible, including vegetation overhanging into the frame, and excluding vegetation hanging outside of the frame. Effort was made to only include the current year's growth and exclude standing litter. Litter was collected in the same manner, clipping and collecting all litter within the frame, until the soil surface was exposed. Litter and biomass were dried for 24 hours at 65 °C, and averaged across each plot to determined dry biomass and litter in kg ha^{-2}. This reflected the methods for biomass collection in 2000 described in Gardner et al. (2010).

Aggregate stability was determined using the Jordana Soil Stability Kit to determined stability class (Herrick et al. 2001; Herrick et al. 2009). Sampling methods were modified from Herrick et al. (2009). Nine randomly placed replications in each treatment plot were used to determine the stability class for each plot. Samples were taken from the soil surface, and a subsurface sample and 2-3 cm below the surface. If samples were damp, they were left to air dry for 0.5 – 1 hour to promote uniform moisture content between aggregates (Herrick et al. 2001). Aggregate samples were submerged in tap water and after five minutes were dipped five times then assigned a soil stability class between 1 and 6 (Table 3.1).

One 5.08cm x 15.24cm" bulk density (D_b) sample was taken in the center of each plot using a core sampler with a slide hammer. This sample was dried at 65°C for 24 hours to remove all soil moisture. Cores were taken in the 0-15 cm depth. D_b was also collected in 2000 (Gardner et al. 2010).

Table 3.1. Soil stability class scores and criteria from Herrick et al. (2009).

Stability Class	Criterial for stability class
1	50% of structural integrity lost (melts) within 5 seconds of immersion in water, OR soil too unsTable to sample (falls through sieve).
2	50% of structural integrity lost (melts) 5-30 seconds after immersion.
3	50% of structural integrity lost (melts) 30-300 seconds after immersion, OR < 10% of soil remains on the sieve after five dipping cycles.
4	10–25% of soil remains on the sieve after five dipping cycles.
5	25–75% of soil remains on the sieve after five dipping cycles
6	75–100 % of soil remains on the sieve after five dipping cycles.

Water retention curves (WRC) were determined for disturbed samples using the pressure plate method using a 1600 5 Bar Pressure Plate Extractor (SoilMoisture Equipment Corp.) and 1500F1 15 Bar Pressure Plate Extractor (SoilMoisture Equipment Corp.), at the BC Ministry of Environment, Analytical Laboratory in Victoria BC. Two WRC were created for each TSF. On each pond one soil sample, made up of 10 randomly collected subsamples, was taken from blocks 1-4 and 5-8(Figure 3.2) from the 0-15 cm depth. Water pressure points of 5, 10, 33, 100, 300 and 1500 J kg^{-1}, packed to the measured bulk density for a given treatment, and were used to create the curves of percent volumetric water for each treatment. Available water holding capacity (AWHC) was determined by subtracting the volumetric water content at 1500 J kg^{-1} from field capacity (FC). At the S TSF, FC was the percent volumetric water content at 10 J kg^{-1}, and at the SiL TSF was 33 J kg^{-1}. These methods were also used in Gardner et al. (2010).

A double ring infiltrometer (Turf-Tec, Tallahassee, FL., USA) was used to calculate hydraulic conductivity on control, fertilizer, B50 and B250 treatments. The infiltrometer had an inner ring diameter of 6.03 cm, and the outer ring had a diameter of 10.76 cm, and a water level scale that measured up to 10 cm of head pressure. The infiltrometer was placed approximately in the center of each treatment plot, avoiding area were D_b was taken. The vegetation and litter were removed from the soil surface and care was taken not to disturb the soil surface. The infiltrometer was inserted into the soil surface to a depth of 5.5 cm and water was poured into both rings to a height of 10cm. The drop in water level was then timed from the 9 cm head to 1 cm head reading. Readings were not taken from 10 to 9 cm and 1 to 0 cm to avoid variation in timing at the start, and obstacles or surface texture on the soil that prevented the scale from properly reaching zero. The infiltration rate was repeatedly measured until the readings equilibrated. At the SiL TSF, and some instances at the S TSF, the infiltration rate was very slow (>3.7 min cm^{-1} at the S TSF; >1 hr cm^{-1} at the SiL TSF). This limited the change in head depth that was permitted, due to time constraints. In these cases, typically at the SiL, the infiltrometer was allowed to soak from 30 minutes to over an hour, before one reading was taken rather than multiple runs. In these cases, the infiltrometer was run only once, and that reading was used to determine infiltration rate.

The infiltration readings were used to calculate saturated hydraulic conductivity (K_{sat}) using the following formula from Nimmo et al. (2009):

$$K_{sat} = \frac{L_G}{t} \ln\left(\frac{L_G + \lambda + D_0}{L_G + \lambda + D}\right)$$

Where L_G is the ring-installation scaling length (meters), t is the change in time (seconds), λ is the macroscopic capillary length (meters), D_o is the initial depth of ponding (meters), and D is the final depth at time t (Nimmo et al. 2009). Elrick et al. (1989) and Nimmo et al. (2009) suggested most soils fit within λ=0.08 for coarser textured soils, and λ=0.25m for fine textured soils without macropores, so these values were used for the S and SiL TSF's, respectively.

Statistical Analysis

All data was examined using a two-way ANOVA with blocking, examining year and treatment interactions, with the exception of aggregate stability and K_{sat} which examined depth and treatment. If data did not meet the assumption of normality and homogeneity it was log or square root transformed. In many cases the assumption of normality was not met, but homogeneity of variance was. Many QQ plots of non-normal data did not visually deviate far from a normal distribution. Therefore, the ANOVA was still conducted.

In all scenarios, if treatment was significant, Bonferroni post hoc comparisons were made. A Bonferroni adjustment was chosen over other post hocs because of the high number of ANOVA's during analysis. With multiple ANOVA's there is an increased risk of a type 1 error, but the Bonferroni adjustment is considered very conservative and has a low likelihood of resulting in a type 1 error. All statistical analysis was conducted using R (version 3.3.3 "Another Canoe" or version 3.2.3 "Wooden Christmas Tree").

Water retention curves could not be statistically analysed due to a lack of repetition. This data was graphed and observations based on the Figures are reported.

RESULTS

Biomass significantly decreased from 2000 (μ=200.5 kg ha^{-1}, SE=26.3), to 2015 (μ=144.7 kg ha^{-1}, SE=13.9) at the S, and significantly decreased from 2000 (μ=1499.5 kg ha^{-1}, SE=171.6) to 2015 at the SiL TSF (μ=337.3 kg ha^{-1}, SE=32.1). At the S TSF, biosolids significantly increased biomass production over the control in the B100 treatment and above the B50 treatment in the SiL TSF ($p<0.01$) (Figure 3.3). These results show that a one-time biosolids application improves biomass production up to 17 years after application, with increases evident with increasing biosolids application up to B150 at the S TSF and B100 at the SiL TSF.

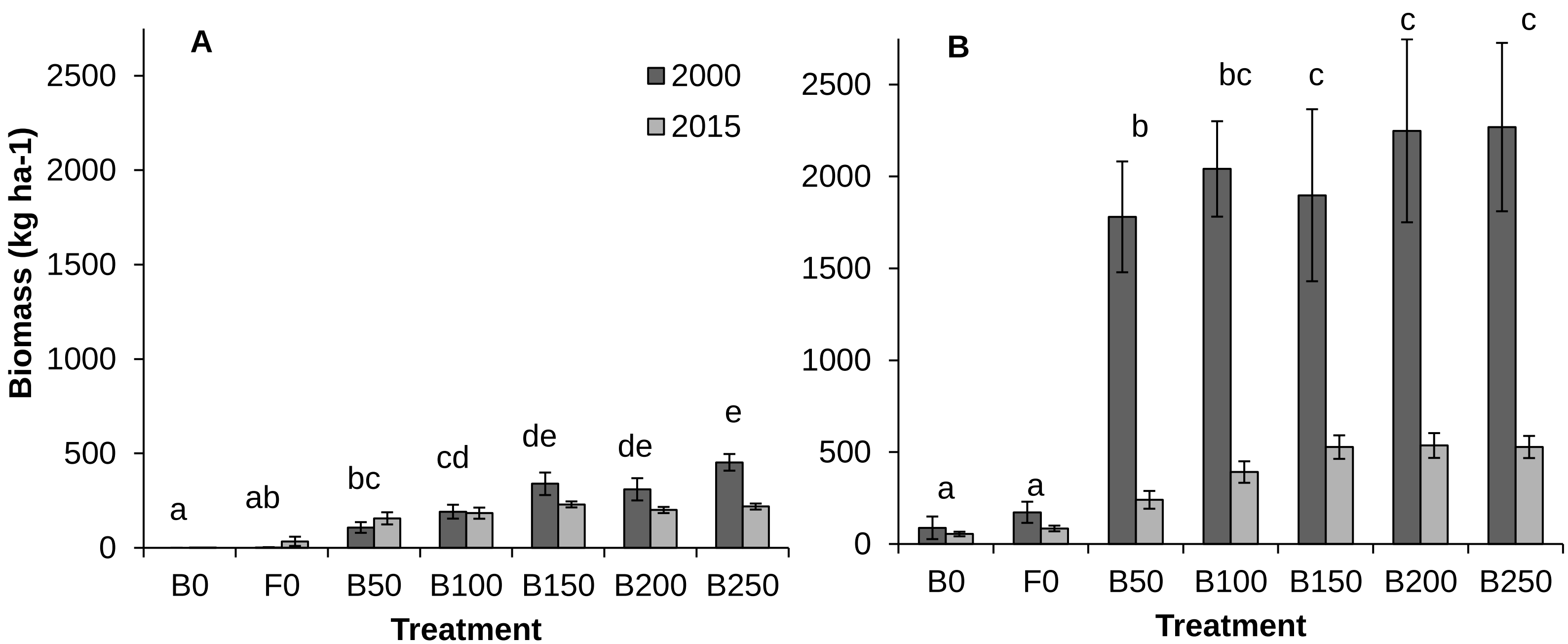

Figure 3.3. Biomass (kg ha^{-1}) at the (a) S and (b) SiL TSF in 2000 and 2015. Error bars represent SE, letters signify significant differences between treatments based on post hoc testing of pooled 2000 and 2015 means

There was a significantly higher amount of litter on biosolids treated plots at both the S TSF ($p<0.001$) and SiL ($p<0.001$) TSF in 2015 (Figure 3.4). None of the biosolids treatments at the S TSF were found to be significantly different from each other, whereas at the SiL TSF, B200 was greater than the B50 treatment (Figure 3.4).

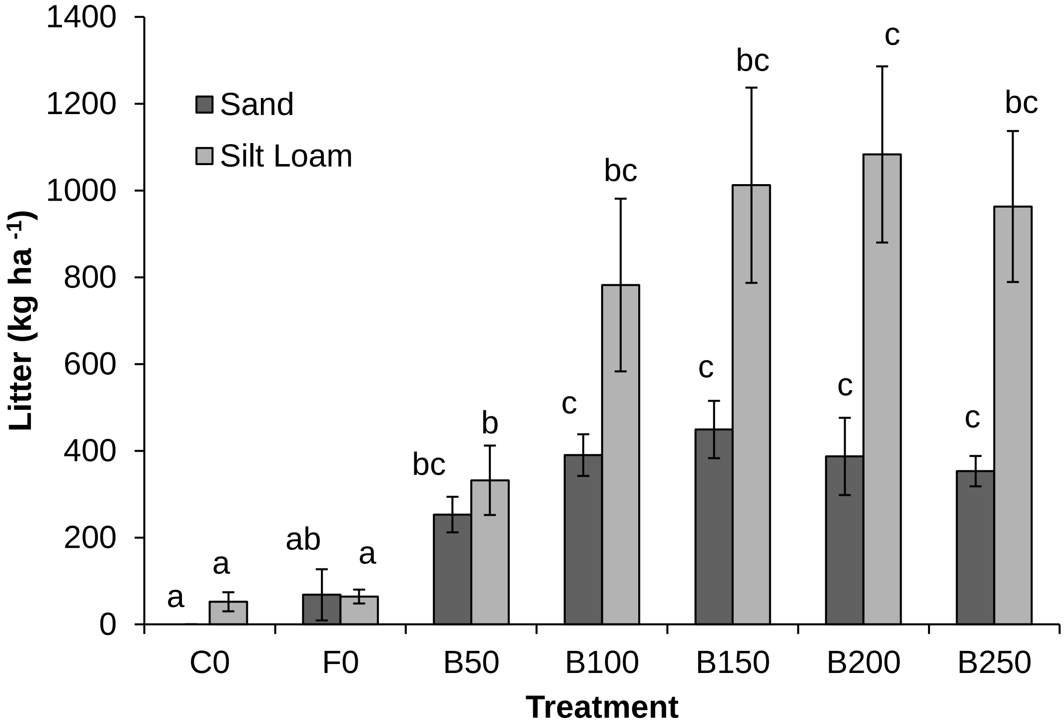

Figure 3.4. Litter (kg ha^{-1}) in 2015 at the S and SiL TSF. Error bars represent SE, and letter represent significant differences between treatments based on Bonferroni post hoc comparisons, for each TSF.

Aggregate class scores were significantly impacted by treatment and depth ($p<0.001$), but post hoc testing could not differentiate between treatments at both TSF's. Although post hoc comparisons were not significant, visual observations suggest that stability class increases with biosolids application at the S TSF and decreased at the SiL TSF (Figure 3.5). Aggregate scores below the surface were different from the surface, and were lower on average (Figure 3.5).

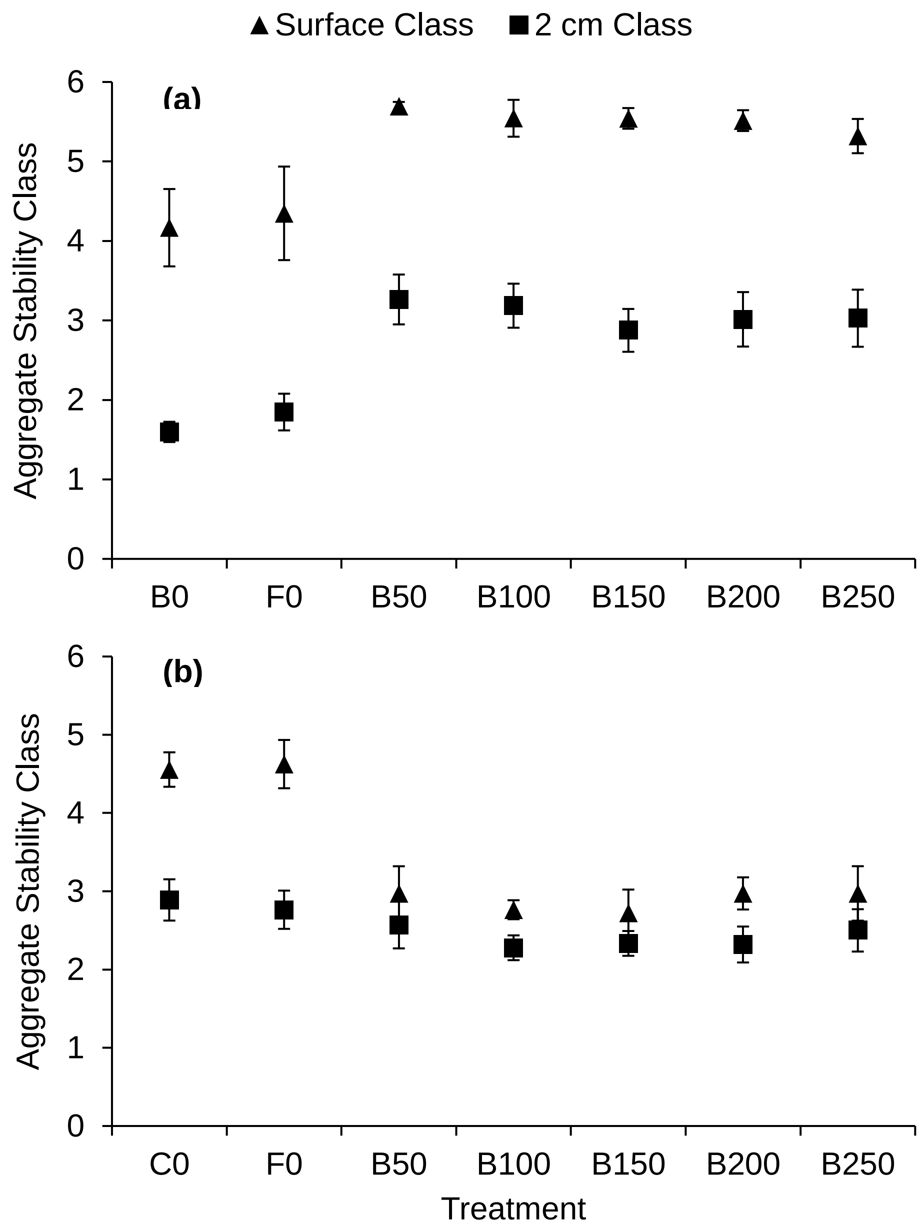

Figure 3.5. Aggregate stability class for the S TSF (a), and SiL TSF (b). Error bars represent standard error. The surface class was taken from the soil suraces, the 2 cm class is the samples taken at a depth between 2-3cm.

No statistical inference can be made on the WRC's, but some visual observations are apparent. At the S TSF, B200 and B250 treatments have the highest volumetric water content across all pressures tested and the highest porosity (Figure 3.6). The control and fertilizer treatments show the lowest volumetric water content at all pressures. This suggests that biosolids on the S TSF can increase water holding capacity of the soil, though this does not mean there is an increase in available water for plant uptake. Observations of the SiL TSF curves are not as simple (Figure 3.7).

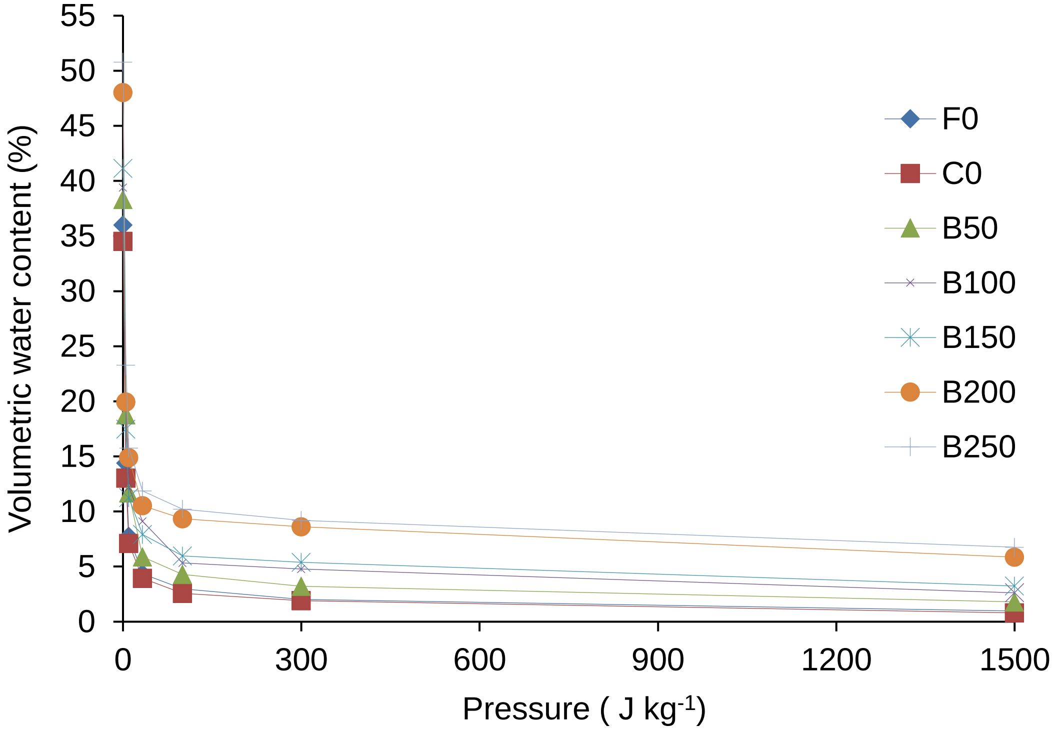

Figure 3.6. Water retention curves for the S TSF. Permanent wilting point is at 1500 J kg^{-1}, and field capacity at 10 J kg^{-1}.

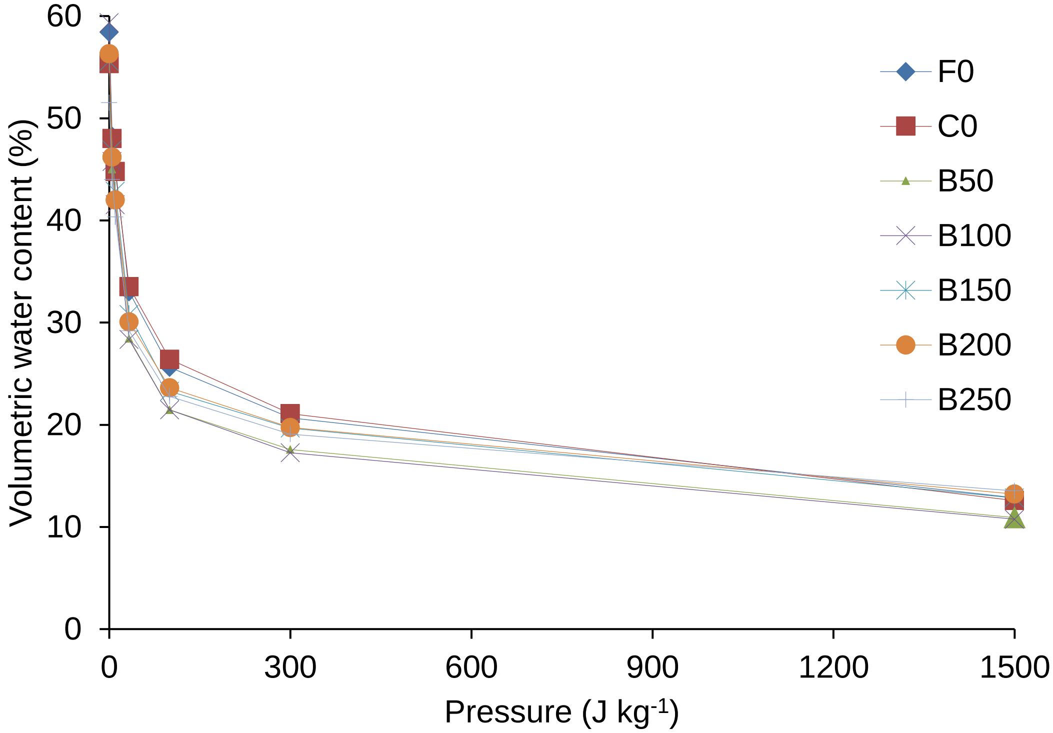

Figure 3.7. Water retention curves for the SiL TSF. Permanent wilting point is at 1500 J kg^{-1}, and field capacity at 33 J kg^{-1}

Saturated hydraulic conductivity was significantly different between the 2 TSF's and increased with a higher biosolids application rate at both TSF's. Post hoc testing could not differentiate between treatments, likely because the Bonferroni is a very conservative test and there was a relatively small sample size (n=8). Regardless, the results clearly show that K_{sat} increased with biosolids application in the B250 treatment in the S TSF, and in the B50 treatment in the SiL TSF over the control and fertilizer treatments (Figure 3.8).

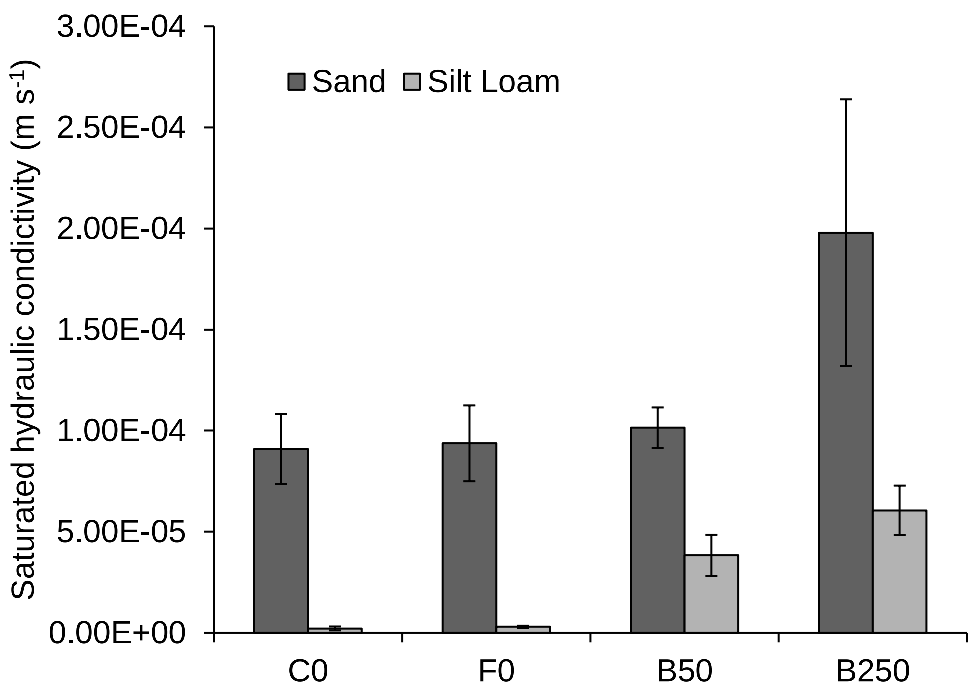

Figure 3.8. Saturated hydraulic conductivity (m s^{-1}) for the S and SiL TSF in 2015. Error bars represent standard error.

Increased biosolids application rates decreased D_b in 2015, at both the S and SiL TSFs ($p<0.001$) (Figure 3.9). At the S TSF, D_b did not change from 2000 to 2015 ($p=0.0817$) but decreased over time at the SiL TSF ($p<0.001$) decreasing from 0.99 to 0.85 g cm^2. The S TSF did have a significant interaction between year and treatment ($p<0.05$) because 2015 values were below 2000 values in fertilizer, control, and B50-B150 values, then increased above 2000 values in B200 and B250 treatments. Differences between treatments were still evident 15 years after a one-time biosolids application, and the SiL TSF D_b continued to decrease through time.

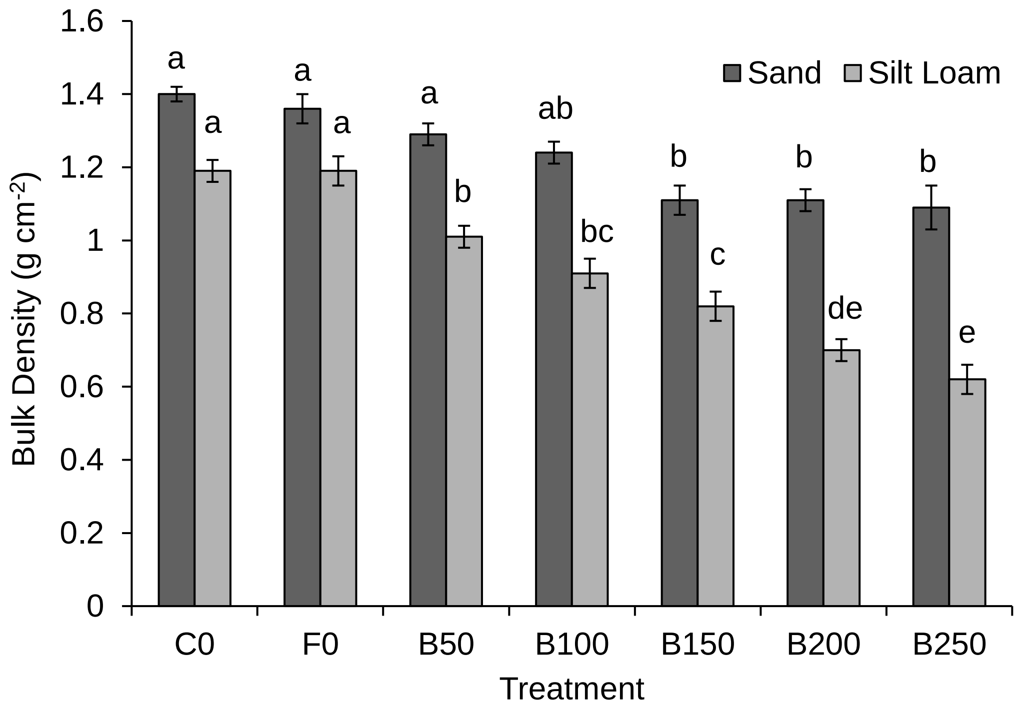

Figure 3.9. Bulk Density (g cm^{-2}) for the S and SiL TSF across treatments in 2015. Error bar represent standard error, and letter represent significant differences across treatments within each TSF.

DISCUSSION

On both TSF biomass increases with biosolids application rate in 2015, as reported in the year 1999 and 2000 (Gardner et al. 2011), but has decreased over time. Many studies demonstrate that biosolids increase plant biomass over no amendments (Andrés et al. 2007; Gardner et al. 2011; Brown et al. 2014). This has been attributed to improvements to nutrients and soil physical and biological characteristics (Andrés et al. 2007; Basta et al. 2016). Other studies have also shown the ability of a one-time biosolids application to increase the biomass of desirable species, under the correct moisture and plant species conditions (Newman et al. 2014; Basta et al. 2016). The decrease we see over time may be attributed to nutrients in the soil equilibrating. After receiving a nutrient pulse in 1998, resulting in a large increase in biomass growth, the biomass may drop to a new equilibrium. Despite the decline in biomass, the results indicate that a one-time application of biosolids allowed for the establishment of a self-sustaining vegetation community. Similar results were found in Brown et al. (2014), where long term (11 years) data showed sustained vegetation growth, over the controls which continued to have no vegetation growth on tailings and overburden.

Aggregates increase at the S TSF, representing an increase in biological activity. Stability on the surface of biosolids treated plots likely increased due to the increased organic matter and biological activity (Wallace et al. 2009; Asensio et al. 2013). This is supported by the increase in litter and biomass found in biosolids treated plots which would increase root exudates and fungal hyphae. Increase in aggregates stability in biosolids treated plots have also been reported in other studies (García-Orenes et al. 2005; Wallace et al. 2009; Asensio et al. 2013).

On the SiL TSF aggregate stability class decreased in biosolids treated plots. This is because control plots had a hard crust that formed on the surface. The higher fraction of fine particle sizes, lack of organic matter and vegetation cover makes this soil surface susceptible to the effect of raindrop splash leading to the formation of surface crusts (Tarchitzky et al. 1984; Mills and Fey 2003; Singer and Shainberg 2004). This crusting is separate from what is considered a biological crust and may be considered a structural or sedimentary crust (Bissonnais 1996). Conversely, biosolids treated plots had much more vegetation growth and biological activity that prevented the same surface crusting. This concept is also supported with the decrease in D_b found in biosolid treated plots. At the SiL TSF, bulk density decreases with biosolids application, which can be an effect of the increased vegetation growth, and prevention of crust formation.

Visual observations of the WRC’s show some response at the S TSF, but not at the SiL TSF where the finer texture may have a greater influence on water retention. At the S TSF, WRC curves have shifted upwards but this does not necessarily lead to increased available water, as the distance between wilting point and field capacity must increase. The WRC for the SiL TSF soils are very tight, suggesting that the impact of the SiL TSF texture has a greater influence over the WRC than the addition of biosolids 18 years prior. It is well known that the addition of organic amendments can increase the water retention capability of the soil but there is no consensus within the literature on whether there is an increase in plant available water. For example, Zebarth et al. (1999), Aggelides and Londra (2000), Gardner et al. (2010); Asada et al. (2012), and Sun and Lu (2014) all report an increase in water retention, but did not a report increase in plant available water. Other studies have shown a biosolids and fly ash amendment applied to a land fill increased plant available water over the fly ash alone 11 years post application (Weber et al. 2015). Other organic amendments such as compost can increase plant available water on degraded soils (Foley and Cooperband 2002; Curtis and Claassen 2005). The increase in volumetric water content across all pressures is likely do to an increase in pore space and aggregate formation both which increases with organic amendments (Aggelides and Londra 2000; Asada et al. 2012; Sun and Lu 2014).

K_{sat} was significantly different between the two textures, but increased at both TSF's in the biosolid treated plots. It is expected that K_{sat} would be greater at the S TSF, because the larger particle size lowers the amount of surface area that acts as an adsorption surface, and creates larger pore spaces between particles allowing water to move more freely through the soil. Because the S TSF already has a high K_{sat}, increases as a result of biosolid applications are not apparent without higher application rates (Figure 3.8). SiL TSF had a lower K_{sat} and biosolids greatly increased and improved the K_{sat} compared to the control and fertilizer treatments. This is likely due to reduced surface crusting that would otherwise block pores preventing downward water movement (Mills and Fey 2003).

D_b decreases at both textures with biosolids treatments. At the SiL TSF D_b also decreased from 2000 to 2015, possibly because it started with the higher D_b, giving it a greater potential for improvement. Decreases in D_b are widely reported with biosolids use and other organic amendments such asbiochar (Aggelides and Londra

2000; Andrés et al. 2007; Wallace et al. 2009; Gardner et al. 2010; Mingorance et al. 2014). This is mainly attributed to the increase in porosity in soils receiving organic matter.

My results differ from the conclusions in Bendfeldt et al. (2001), who concluded sewage sludge amendments on a mine soils improve physical properties in the first 5 years, but after 16 years were not significantly improved from control plots. In Bendfeldt et al. (2001), soil organic matter was not statistically different between the control and biosolids treatments, as the control treatments increased in organic matter over the time period. Bendfeldt et al. (2001) also concluded that biosolids did not greatly improve aggregate stability, bulk density and porosity from that of the control, but the 112 Mg ha^{-1} treatment did have statistically improved aggregate stability, lower bulk density, and increased porosity. The caveat to this was lower applications were not statistically different from control, and the different application rates were not statistically different from each other. Conversely, the TSF at HVC continue to have little to no vegetation growth on control plots, and biosolids treated plots show significant improvements to the tailings in terms of soil quality. This could be attributed to the initial state of the TSF, where there was no vegetation and very poor physical and chemical soil condition with a lot of potential for improvement. While not a lot of detail is given on the initial state of the soil conditions in the Bendfeldt et al. (2001), they are described as a Typic Udorthents soil, with a 1 m cap of overburden, suggesting there was some remnants of native soil.

CONCLUSION

While many short term improvements seen to biomass, litter and soil physical parameters are well supported in the literature, this study shows the long lasting benefits of a one-time biosolids application on mine tailings. Both sites demonstrated a significant improvement in biomass with the application of biosolids. The benefits to other parameters slightly differ between the S and the SiL TSF.

At the SiL TSF, the litter and biomass production likely shielded the soil surface from the effects of rain drop splash which would clog soil pores causing surface crusting (Bissonnais 1996), increasing aggregate stability at the surface (Tarchitzky et al. 1984; Mills and Fey 2003; Singer and Shainberg 2004). The reduction in surface crusting with biosolids at the SiL TSF improved K_{sat} on biosolids treated plots, as water could more easily penetrate the surface. At the S TSF biosolids addition increased aggregate stability at the surface, as the sand particles start to cement together likely due to increased root exudates and increased fungal activity (Tisdall and Oades 1982). The increase in organic matter also increased water holding capacity, decreasing K_{sat} as the water filled the soil pores. The results between these two TSF's were opposite, but both were improvements to the soil condition. While biosolids did decrease bulk density in both TSF's, the same trend of increase porosity with biosolids at the S TSF is not apparent at the SiL TSF because the WRC's are much tighter. This suggests that the finer soil texture has a greater overall influence on soil hydrological function compared the S texture. Overall, this study supports the concept that a one-time biosolids application can set a tailings site, with otherwise no productivity, on a positive trajectory in terms of biomass growth and soil physical improvement and development.

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Chapter 4. RESEARCH CONCLUSIONS, MANAGEMENT IMPLICATIONS, AND FUTURE RESEARCH

RESEARCH CONCLUSIONS

The overarching goal of reclamation activities is to create a productive, self-perpetuating and self-sustaining ecosystem which requires site limitations to be addressed. Solutions such as fertilizers do not address all the site limitations, and usually require annual inputs to sustain vegetation (Gardner et al. 2011). Organic matter amendments, such as biosolids, can provide nutrients and improve soil structure, bulk density, aggregate stability, hydrological function, soil microbial status and add organic matter (Aggelides and Londra 2000; Wallace et al. 2009; Gardner et al. 2010; Gardner et al. 2011). This can lead to the successful revegetation of many mining by-products not previously sustaining vegetation (Santibáñez et al. 2007; Gardner et al. 2011).

While there is a lot of information on biosolids and information related to the proposed study, none have fully combined all the aspects that will be examined over a relatively long term (>10 years). This project provided the opportunity to look at chemical and physical changes in two alkaline Cu-Mo tailings ponds, very close in proximity, with different moisture contents and textures, under the same experimental conditions.

A field experiment was conducted on a sand (S) and a (SiL) tailings storage facility (TSF). The objectives of this study were to examine the effects of a one-time biosolids application in 1998, at rates between 0-250 Mg ha^{-1}, on a sand and silt loam tailings storage facility, on:

1. Metals and nutrients 17 years after application. This was done by examining the total and available nutrients and metal concentrations across two different soil textures, between 2000 and 2015, as well as across different application rates, from 0 to 250 Mg ha^{-1}, in 2015.

2. Physical parameters 18 years after application. This was done by examining changes between treatments and tailings texture in 2016. The specific parameters examined included biomass, litter, aggregate stability, water retention, saturated hydraulic conductivity and bulk density. Plant biomass and bulk density were also examined between 2000 and 2016.

Experimental treatment plots were established on both TSF's July 1998 in a randomized complete block design and are described in Gardner et al. (2010) and Gardner et al. (2011). Treatments consisted of one time applications of biosolids at 50, 100, 150, 200 and 250 Mg ha^{-2} (B50, B100, B150, B200 and B250), a one-time fertilizer treatment and a control treatment. Each treatment from was applied in a 7x3 meter plot. Each block consisted of a row of randomized treatments, separated by a 0.5 meter buffer, and rows were separated by a 1 meter buffer. This created 8 treatment replicates on each TSF. In 2015, treatment plots were reduced to 5x2 m plots to reduce edge effects and vegetation drift along the perimeter of each plot.

Benefits

This research demonstrates that biosolids use in tailings reclamation can promote long term soil development on different textures. The one time biosolids application has improved physical soil structure demonstrated by improved aggregates and hydrological properties over an 18 year period (Brown et al. 2014; Avery et al. 2017). Biosolids treated plots also still demonstrate high macronutrient concentrations compared to control and fertilizer plots, accompanied by higher biomass production.

Nutrients in biosolids treated plots still showed clear improvements 17 years after biosolids were applied. Biosolids treated plots had significantly higher total C, N, and P and available NH_4, NO_3, and P concentrations in biosolids treated plots. These nutrients were higher in the SiL TSF compared to the S TSF. This may be an effect of the soil texture, as finer texture can hold constituents more effectively. Soil surface area is very closely correlated to increasing cation exchange capacity (Ersahin et al. 2006), therefore finer textured soils tend to have greater capacity to retain soil constituents due to greater surface area for complexes to form (Yang et al. 2014). The S TSF also appeared more susceptible to wind erosion (visual observations), which can also be a significant source of nutrient losses (Li et al. 2007). Crusting and biomass cover at the SiL TSF may have preventing this eolian erosion (Singer and Shainberg 2004). There is also greater biomass and litter on the SiL, potentially providing more organic matter to form complexes with. The SiL TSF was also less moisture limited, as this TSF was managed as a wetland, therefore tended to have more moisture along its banks were the treatment plots were established. Collectively, the finer texture may have led to increased nutrient retention, along with higher soil moisture, and increased biomass and litter production, which in turn increased nutrient cycling, and retaining higher nutrient levels on the SiL TSF.

Physical parameters where also in better condition in biosolids treated plots, over that of the control and fertilizer treatments. Eighteen years after a one time biosolids application, physical and hydrological properties of both the S TSF and the SiL TSF had improved compared the control and fertilizer treatments. The S and the SiL TSF had different results in aggregate stability, increasing in the S and decreasing in the SiL. At both TSF’s these responses improved hydrological conditions in the soils. At the S TSF, the increase in aggregate stability is likely a function of increased biological function increased root exudates, fugal hyphae and polysaccharides all which contribute to increased aggregate stability and formation (Tisdall and Oades 1982). At the SiL TSF the higher aggregate stability on the control is due to the fine particle size which is more at risk to the negative effects of raindrop splash that causes surface crusting which increases aggregate stability on the surface (Tarchitzky et al. 1984; Bissonnais 1996). The formation of crusts in the SiL TSF controls results in decreased surface porosity in finely textured soils which helps explain the extremely low K_{sat} on the SiL TSF control plots (Tarchitzky et al. 1984). Biosolids on the SiL added organic matter and promoted vegetation growth which prevented surface crusting, resulting in a decrease relative to the control (Pagliai et al. 2004).

The additional organic matter on the biosolids treated plots improved hydrological function. Biosolids treated plots had increased K_{sat} relative to the control on both TSF's. Lado et al. (2004) also observed that K_{sat} increased in soils with higher organic matter. This may be attributed to stronger soil structure in biosolids treated plots over the control, preventing the slaking and dispersal of fine particles, and swelling that would otherwise block soil pores, slowing water movement (Lado et al. 2004). Additional vegetation and litter cover can also promote increased water retention (Meeuwig 1970), which were the results observed in the S TSF water retention curves. The same trend was not so apparent in the SiL TSF, as the finer texture had a greater influence on hydrological function than the S TSF, represented by the tighter configuration of the WRC. Through modelling, Jong et al. (1983) found that texture did have a greater influence on water retention curves than organic matter. In their modeled water retention curves, the high and low clay soil curves were much closer together and the sand curves were further spread, specifically at higher suction (permanent wilting point). Though not tested in Jong et al. (1983) this may suggest that WRC of sandy soils are more readily altered by organic matter, than finer textured soils. The tighter configuration can also be due to errors in the methodology. It has been reported that using the pressure plate method on fine textured soil can introduce more errors (Solone et al. 2012). This demonstrates the importance of soil texture when trying to determine the effect biosolids will have on a soil medium.

State and transition models suggest that an ecosystem changes along a successional gradient within a state, but if the ecosystem can be forced over a threshold, it can be pushed into a different state with different structure and function. A threshold can be defined as a change in structure or function that alters ecosystem processes (Briske et al. 2005). In this study, biosolids at a certain application rate may have pushed a treatment plot over a threshold that changes the function of that treatment (e.g. hydrological function, as measured by aggregate stability and K_{sat}). The fertilizer treatment was not enough of a change to push those treatments over a threshold into another state; 17 and 18 years later the fertilizer treatments are not any different from the control treatments. Biosolids changed parameters of the soil (i.e. nutrient status, hydrological function) enough to push the biosolids treated plots over a threshold into a new state. Some visual observations suggest there may be different states between treatments as well. For example, in B250 treatments in the SiL TSF, thistles (*Cirsium* spp.) had established, but were not present in other biosolids treatments, although there is no evidence of functional changes. Paschke et al. (2005) also found that 24 years after biosolids addition on a degraded sagebrush steppe habitat soils had a decreased C:N ratio, increased nutrients, and increased biomass relative to control plots. This research suggests that biosolids can set the tailings on a different vegetation trajectory depending on the initial application rate due to changes in ecosystem functioning.

Long term effects of a one-time biosolids addition have shown conflicting results on mine wastes. Pichtel et al. (1994) found biosolids positive increases in biomass and pH on mine acidic bank spoil, 10 years after application, to the same degree as a top soil amendment. Other mine waste products like over burden have also been examined, but did not show positive long term (16 years) improvements over control, but the lack of significant results may have been due to low replication (n=4)(Bendfeldt et al. 2001). These degraded mine soils contain some native soil components compared to tailings which only contain ground rock, essentially creating a geological time zero. Compared to overburden, tailings have no natural soil properties, which likely contributes to the different results between this study and Bendfeldt et al. (2001). The author of this thesis is unaware of any long term trials on mine tailings, focusing strictly on a biosolids amendment. The results presented in this thesis conclude that a one-time biosolids application does have an overall positive effect on mine tailings.

The use of biosolids in reclamation is not only beneficial to mine reclamation, but also to the waste stream itself. There is a finite source of nutrients and energy, and reusing sewage waste in a productive manner prevents the loss of nutrients and energy in an unproductive and potentially destructive way. Other options for municipal sewage waste disposal, including most commonly land filling and incineration (Kelessidis and Stasinakis 2012). This wastes energy and nutrient resources (Peccia and Westerhoff 2015), and contributes to greater greenhouse gas emissions over land application (Miller-Robbie et al. 2015). In a world mismanaged of nutrients, fertilizer manufacturing, and excess crop fertilization, land managers need to seek out effective and ethical ways to ensure effective use of land and nutrients in a way that benefits society and the natural surrounding environment. Considering the use of biosolids and site specific planning of their use may be one of these solutions.

Limitations/Concerns

This research demonstrated that one time biosolid application to mine tailings provided many positive results. The risks examined herein focused on potential metals leaching and plant available metals, but there are guidelines for total fractions (Canadian Council of Ministers of the Environment Guidelines). For the 16 metals measured (not including nutrients), only total Zn exceeded guidelines for agricultural land due to biosolids application in B200 and B250 treatments in the SiL, but did not exceed industrial guidelines. Total Mo and Cu also exceeded CCME guidelines for agricultural soils, but this was due to the source rock of the tailings and not the biosolids application. Other studies examining leaching of metals after biosolids use have also concluded that biosolids do increase metal concentrations over no amendment, but metal mobility is well below predicted amounts or those amounts are negligible (Shober et al. 1996; McBride and Evans 2002; Basta et al. 2016). Some metals did significantly increase over the years, and with treatment at the SiL TSF. These included available Mn (increased 4.2 mg kg^{-1}) and Zn (increased (57 mg kg^{-1}). Total fractions of Mn remained well below guidelines for agricultural soils therefore available fractions are not likely to reach levels of concern.

MANAGEMENT IMPLICATIONS

This research supports the use of biosolids in tailings reclamation to produce a functioning ecosystem with improved hydrology, soil structure and sustained levels of most nutrients. These improvements address many of the limitations to tailings reclamation and revegetation. By addressing these limitations vegetation can establish and maintain itself, supporting the overarching goal of reclamation of creating a self-sustaining and self-perpetuating ecosystem.

Site specific management is necessary to mitigate the risks associated with the use of biosolids. This research has shown that increased levels of biosolids application will increase some metals relative to the application rate, demonstrating a need for site specific planning. Careful consideration should be put into examining the nutrient needs of the vegetation to be established as to not apply at a higher application rate than necessary (CCME 2012). This will help alleviate the risk of nutrient leaching and can prevent the addition of metals above recommended soil guidelines. Understanding soil texture and hydrological function of the receiving medium is also important. For example, in this study in B200 and B250 at the SiL TSF Zn exceeded agricultural guidelines, had the highest NO_3 concentrations (high leaching capability) and in some cases had high cover of thistles (data not presented). Additionally, total nitrogen and biomass did not significantly increase in the B250, suggesting the higher application did not result in greater improvements to the soil and vegetation community over the 150 Mg ha^{-1} and 200 Mg ha^{-1} applications.

FUTURE RESEARCH

This research demonstrates that biosolids can be used effectively for the long term sustainability of reclamation activities, and risks associated with nutrient and metal leaching can be mitigated with site specific planning, considering nutrient needs, soil texture, and moisture regime. This experiment has shown successful results with the use of biosolids that are likely amplified by the tailings starting condition. In order to gain a broader and more complete understanding on how biosolids use has impacted these ponds other factors should also be considered, including soil microbial community, vegetation community composition, and metal and nutrient uptake in vegetation. The further examination on how biosolids could impact higher trophic levels should also be considered, examining potential effects on insects, birds, reptiles, amphibians and mammals, similar to in Brown et al. (2014) and Bourioug et al. (2015).

Biomass and litter were examined but a closer look at species, and metals and nutrients would provide more value. This would provide a further understanding of how plants are taking up, or not taking up metals, and on the potential exposure of these metals to other organisms. Species diversity, richness and types of plants (i.e. competitors, stress tolerators, and ruderals) would help to further decipher how the biosolids may be affecting the plant community. This is important information to examine if the biosolids treated sites push the vegetation community into a desired state and if the sites are meeting reclamation objectives.

Part of a healthy soil is a healthy soil microbial community. Microbes include bacterial and fungal species, which aid in nutrient cycling and plant uptake of nutrients (Sheoran et al. 2010). They also aid in the formation of aggregates through exuding polysaccharides, improving soil structure and protecting against organic carbon loss (Jastrow 1996; Wallace et al. 2009). Examining the microbial community on these plots will help further understand how the ecosystem is functioning and how the improvements reported in this research are occurring in terms of nutrient cycling and improved soil structure. This could also complement previous work by Gardner et al. (2010).

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APPENDICES

Appendix 1. Homogeneity of variance and normality testing, and associated transformations used for ANOVA. A pass is p≥0.05 and a fail p<0.05. Fitted versus residual plots are also included to show a visual representation of homogeneity of variance, and normal Q-Q plots to visually demonstrate normality of select elemental parameters.

Sand, depth 1

Parameter	Transformation	Shapiro Wilks Normality Test	Fligner-Killeen Test of Homogeneity
pH	None	Fail	Fail
Total B	None	Fail	Pass
Total C	None	Fail	Fail
Total Cr	None	Fail	Pass
Total Cu	None	Fail	Pass
Total Fe	Log	Pass	Pass
Total K	None	Fail	Pass
Total Mg	None	Fail	Pass
Total Mn	None	Fail	Pass
Total Mo	None	Fail	Pass
Total N	None	Fail	Fail
Total Ni	None	Fail	Pass
Total P	Log	Fail	Pass
Total Pb	Square Root	Fail	Pass
Total Zn	Log	Fail	Pass
Available Cu	None	Fail	Pass
Available Fe	None	Fail	Pass
Available K	Log	Fail	Pass
Available Mn	Log	Fail	Pass
Available Mo	None	Fail	Pass
Available NO_3	Non	Fail	Fail
Available NH_4	Log	Fail	Pass
Available P	None	Fail	Fail
Available Zn	Log	Fail	Pass

Sand, depth 2

Parameter	Transformation	Shapiro Wilks Normality Test	Fligner-Killeen Test of Homogeneity
pH	None	Fail	Fail
Total As	None	Fail	Pass
Total B	None	Fail	Pass
Total C	None	Fail	Fail
Total Cr	None	Fail	Pass

Total Cu	None	Fail	Pass
Total Fe	None	Fail	Pass
Total K	None	Fail	Pass
Total Mg	None	Fail	Pass
Total Mn	None	Pass	Pass
Total Mo	None	Fail	Pass
Total N	None	Fail	Pass
Total Ni	None	Fail	Pass
Total P	None	Fail	Fail
Total Pb	None	Fail	Pass
Total Zn	None	Fail	Pass
Available Cu	None	Fail	Pass
Available Fe	Log	Pass	Pass
Available K	None	Fail	Pass
Available Mn	Square Root	Pass	Pass
Available Mo	None	Fail	Pass
Available P	None	Fail	Fail
Available Zn	Log	Fail	Pass

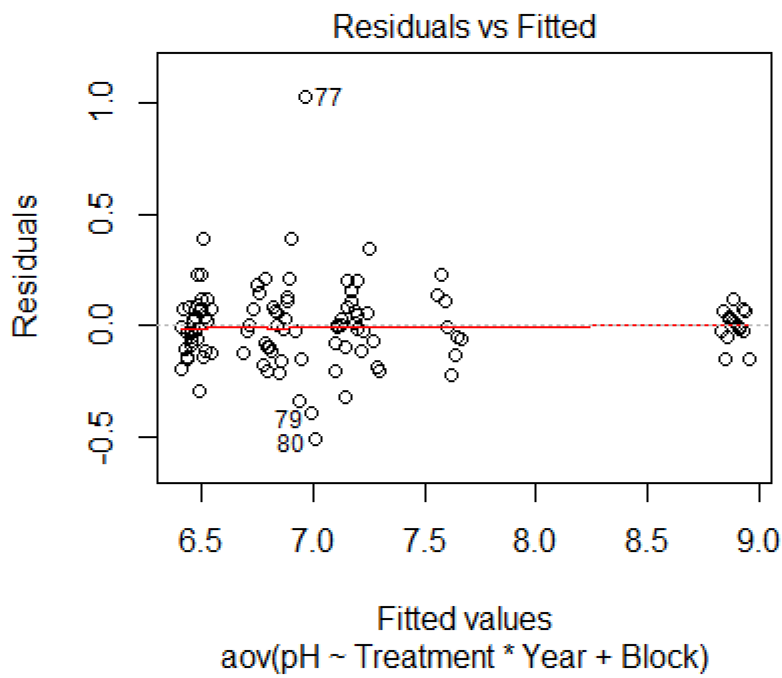
Silt Loam, depth 1

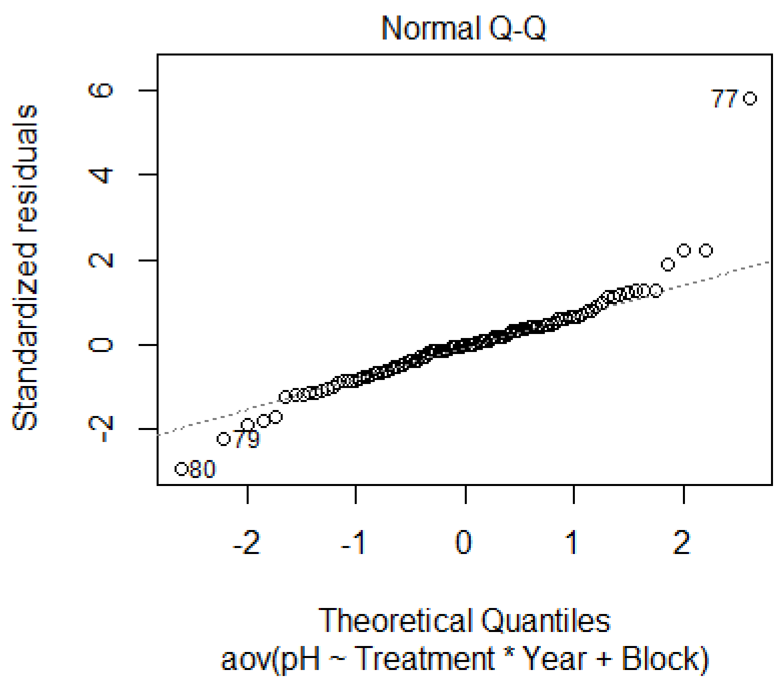
Parameter	Transformation	Shapiro Wilks Normality Test	Fligner-Killeen Test of Homogeneity
pH	None	Fail	Fail
Total B	None	Fail	Pass
Total C	Log	Fail	Pass
Total Cr	Fail	Fail	Fail
Total Cu	None	Fail	Pass
Total Fe	None	Fail	Pass
Total K	None	Fail	Pass
Total Mg	None	Fail	Pass
Total Mn	None	Fail	Pass
Total Mo	Square Root	Pass	Pass
Total N	Log	Fail	Pass
Total Ni	Log	Pass	Pass
Total P	Log	Fail	Pass
Total Pb	None	Fail	Pass
Total Zn	None	Fail	Pass
Available Cu	Log	Pass	Pass
Available Fe	Log	Pass	Pass
Available K	Log	Pass	Pass
Available Mn	Log	Fail	Pass
Available Mo	Square Root	Pass	Pass
Available NO3	Log	Fail	Pass
Available NH4	Log	Fail	Pass
Available P	None	Fail	Fail
Available Zn	Log	Fail	Pass

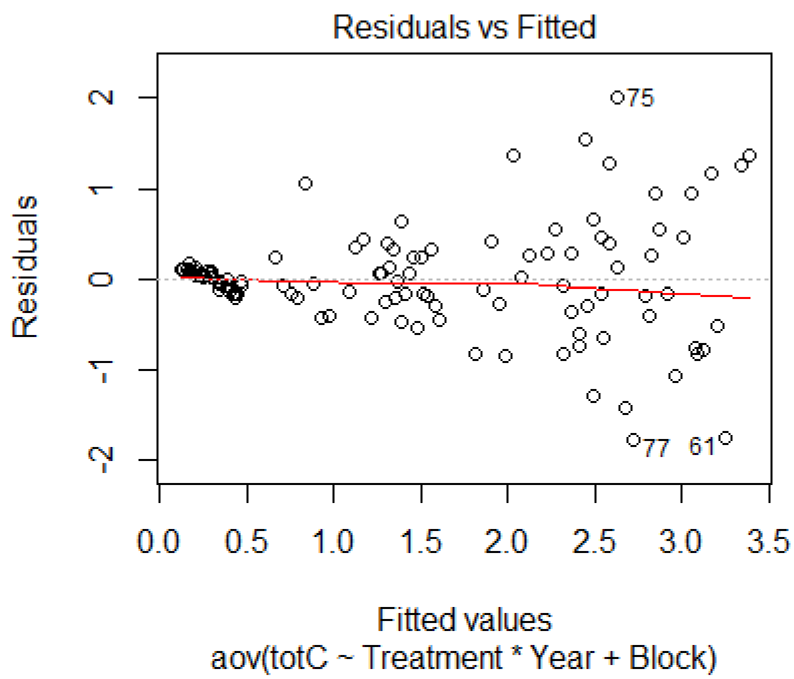
Silt Loam, depth 2

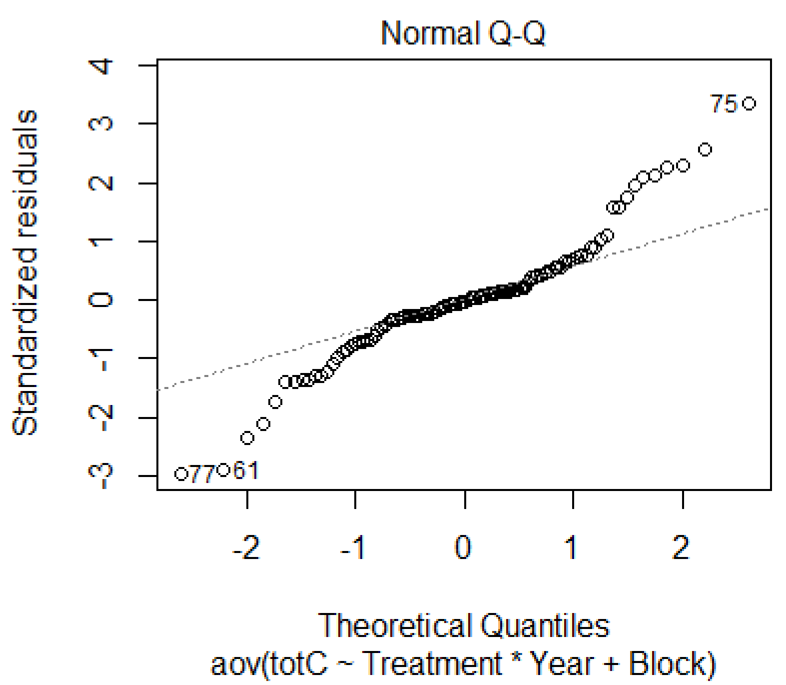
Parameter	Transformation	Shapiro Wilks Normality Test	Fligner-Killeen Test of Homogeneity
pH	None	Fail	Fail
Total B	None	Fail	Pass
Total C	None	Fail	Fail
Total Cr	None	Fail	Pass
Total Cu	None	Fail	Pass
Total Fe	None	Fail	Pass
Total K	None	Fail	Pass
Total Mg	None	Fail	Pass
Total Mn	None	Fail	Pass
Total Mo	None	Pass	Pass
Total N	Log	Fail	Pass
Total Ni	None	Fail	Pass
Total P	Log	Fail	Pass
Total Pb	None	Fail	Pass
Total Zn	Log	Fail	Pass
Available Cu	None	Fail	Pass
Available Fe	None	Fail	Pass
Available K	Log	Fail	Pass
Available Mn	Log	Fail	Pass
Available Mo	Square Root	Fail	Pass
Available P	None	Fail	Pass
Available Zn	None	Fail	Fail

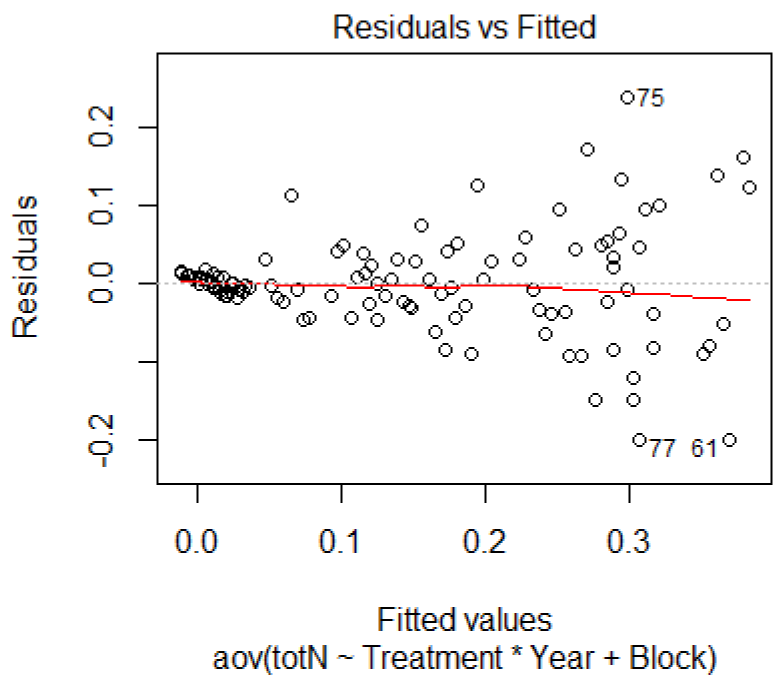
Trojan Depth 1 Residuals vs. Fitted plots and normality Q-Q plots

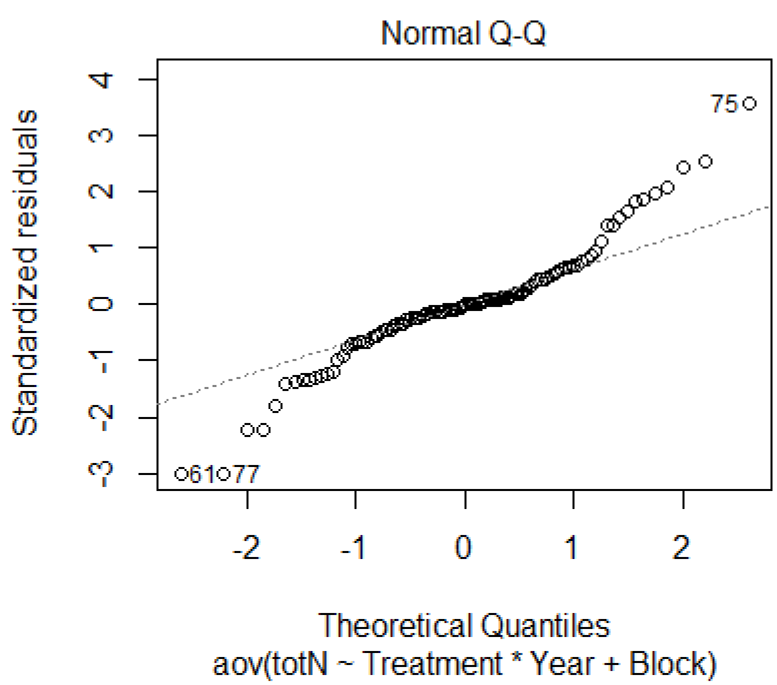


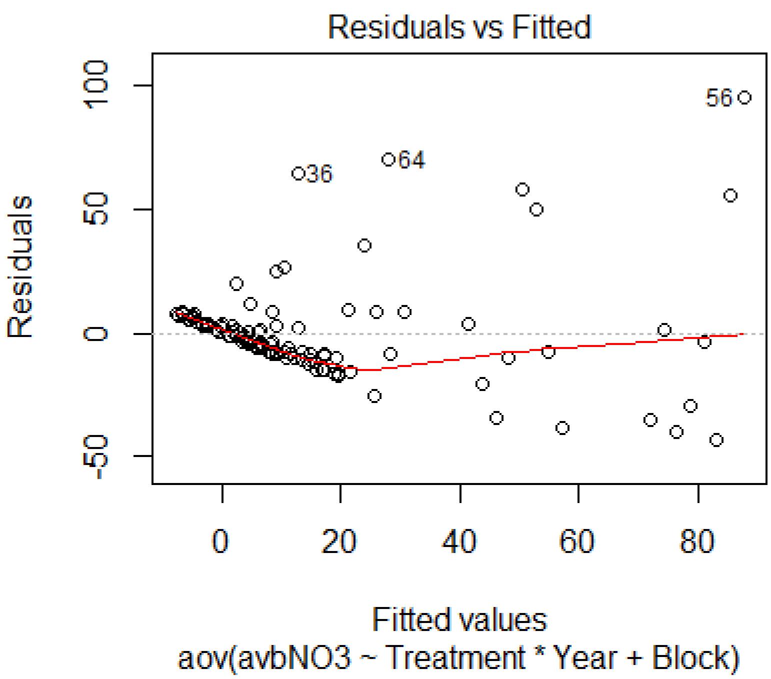


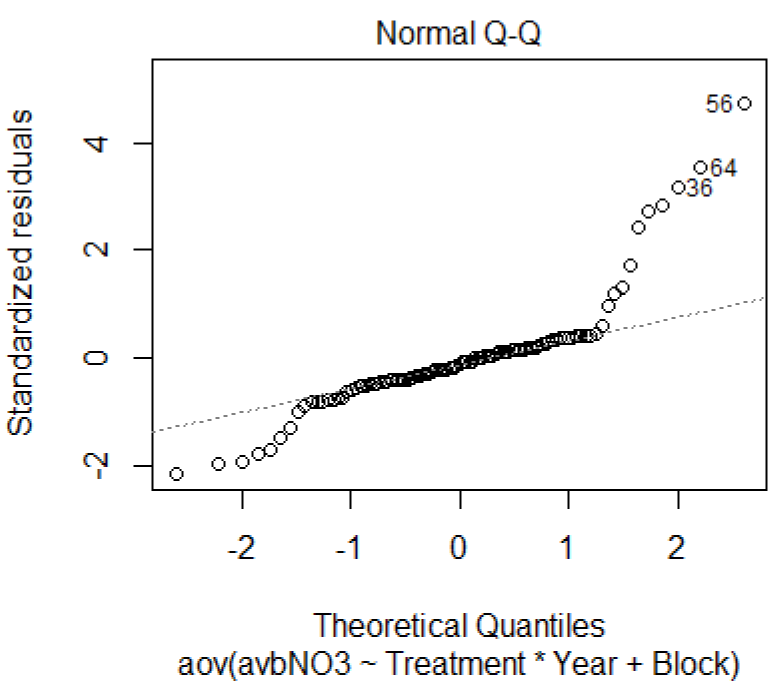

Residuals vs Fitted
Residuals
Fitted values
aov(totC ~ Treatment * Year + Block)
75
77
61
2
1
0
-1
-2
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5


Normal Q-Q
Standardized residuals
Theoretical Quantiles
aov(totC ~ Treatment * Year + Block)
75
77
61
4
3
2
1
0
-1
-2
-3
-2
-1
0
1
2

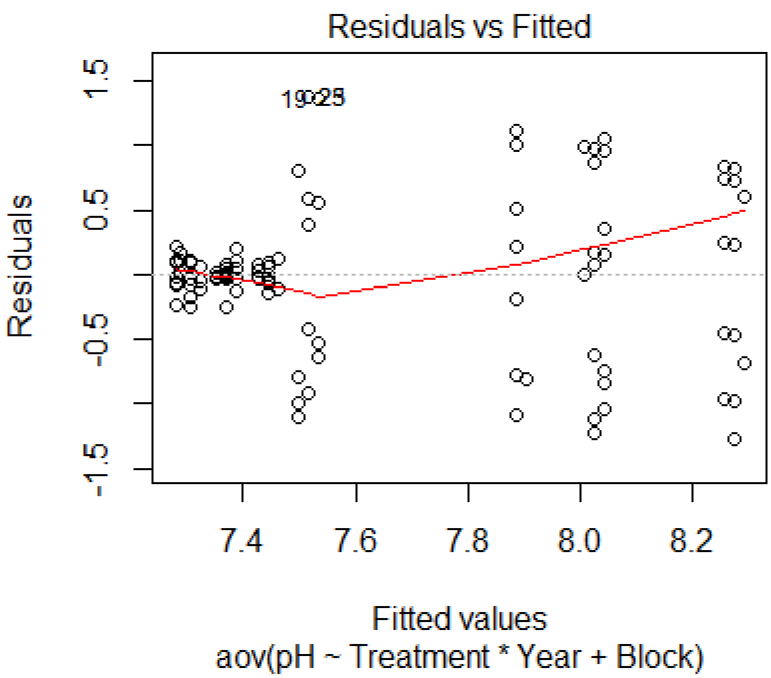

Residuals vs Fitted
Residuals
0.2
0.1
0.0
-0.2
75
77
61
0.0
0.1
0.2
0.3
Fitted values
aov(totN ~ Treatment * Year + Block)

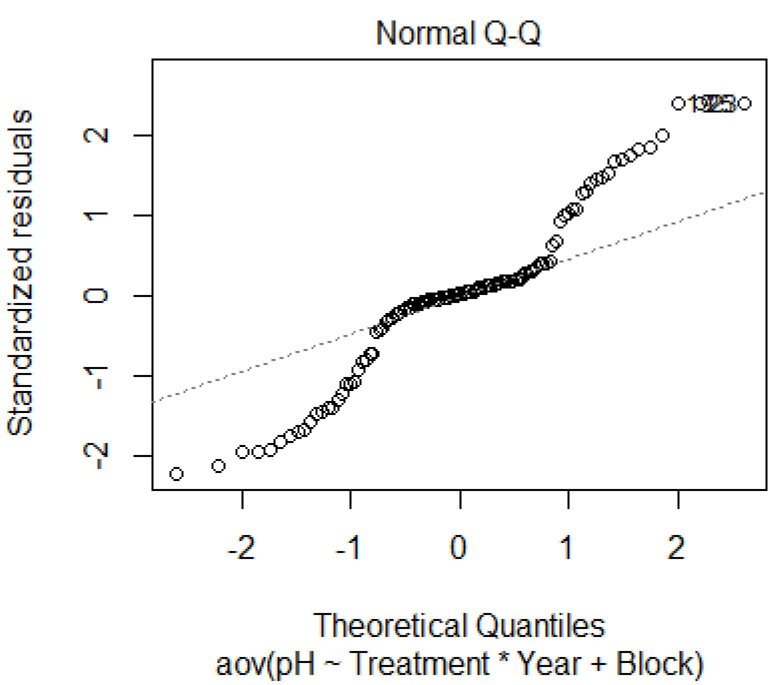

Normal Q-Q
Standardized residuals
4
3
2
1
0
-1
-2
-3
75
61
77
-2
-1
0
1
2
Theoretical Quantiles
aov(totN ~ Treatment * Year + Block)

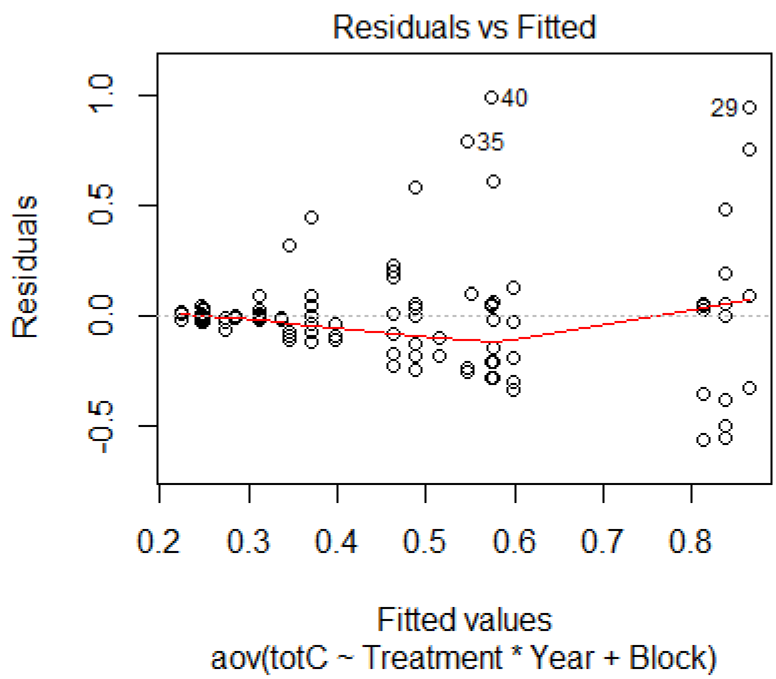

Residuals vs Fitted
Residuals
100
50
0
-50
0
20
40
60
80
56
64
36
Fitted values
aov(avbNO3 ~ Treatment * Year + Block)


Normal Q-Q
Standardized residuals
4
2
0
-2
-2
-1
0
1
2
56
64
36
Theoretical Quantiles
aov(avbNO3 ~ Treatment * Year + Block)

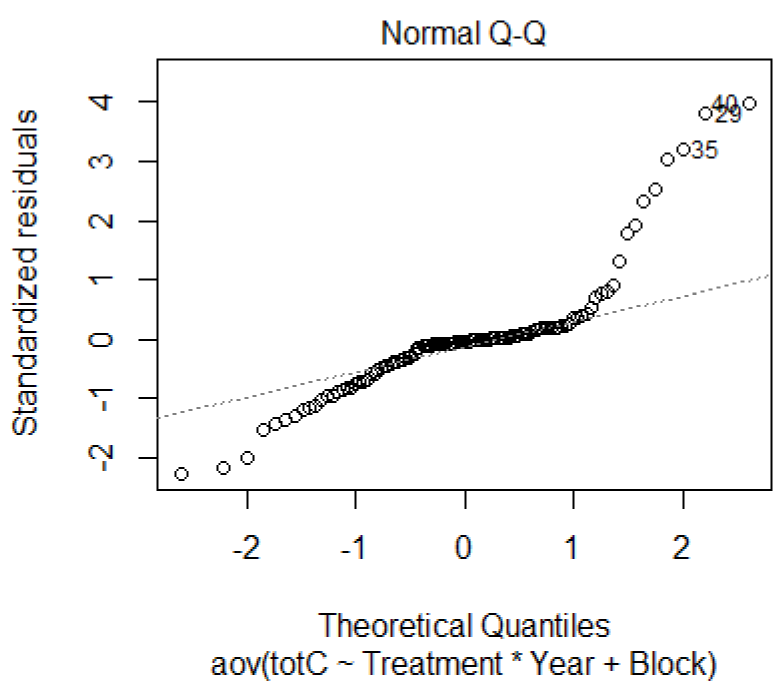
Trojan Depth 2 Residuals vs. Fitted plots and normality Q-Q plots



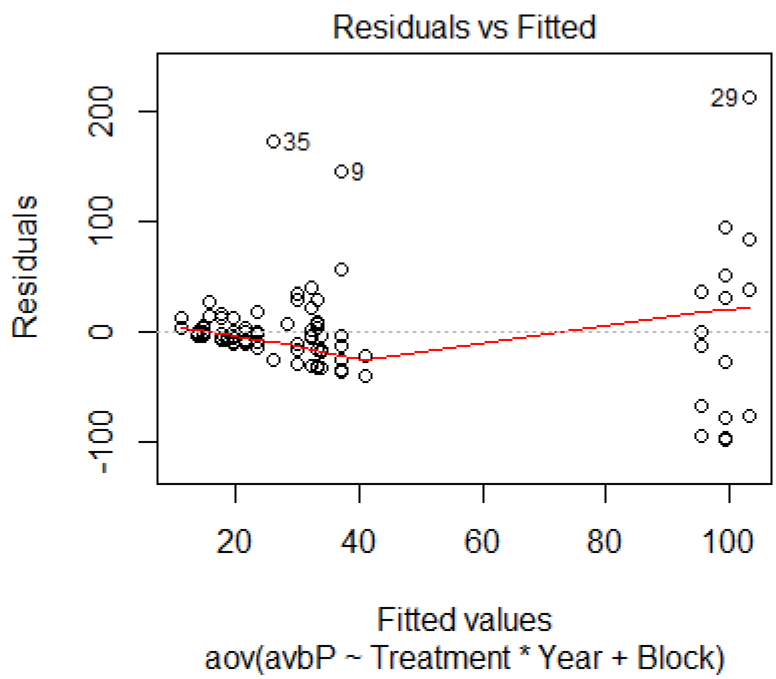




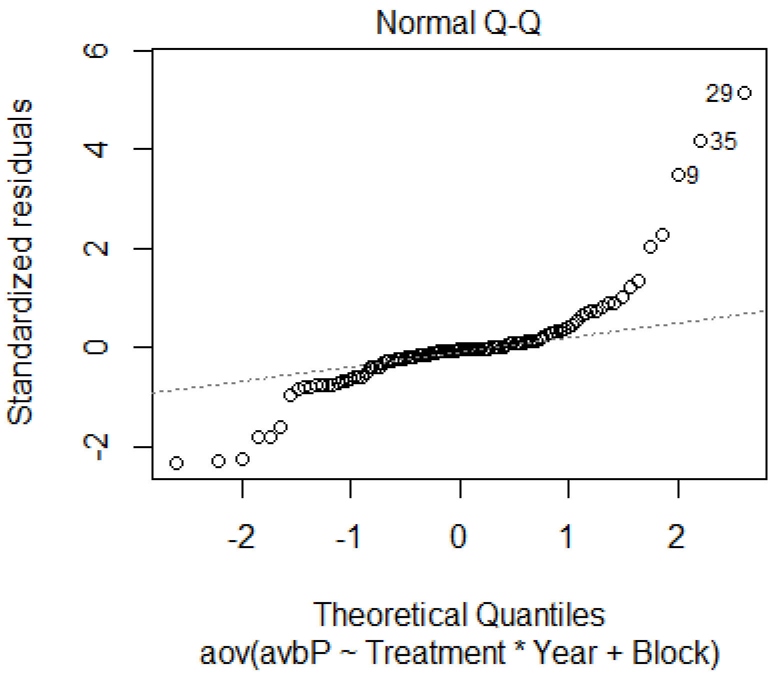
Residuals vs Fitted
Residuals
1.0
0.5
0.0
-0.5
0.2
0.3
0.4
0.5
0.6
0.7
0.8
40
35
29
Fitted values
aov(totC ~ Treatment * Year + Block)



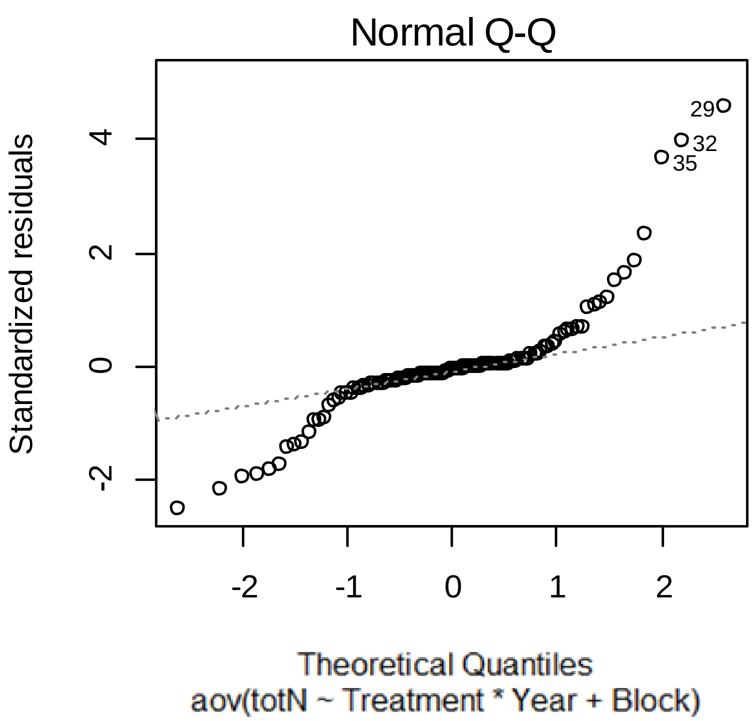
Normal Q-Q
Standardized residuals
4
3
2
1
0
-1
-2
40
29
35
Theoretical Quantiles
aov(totC ~ Treatment * Year + Block)



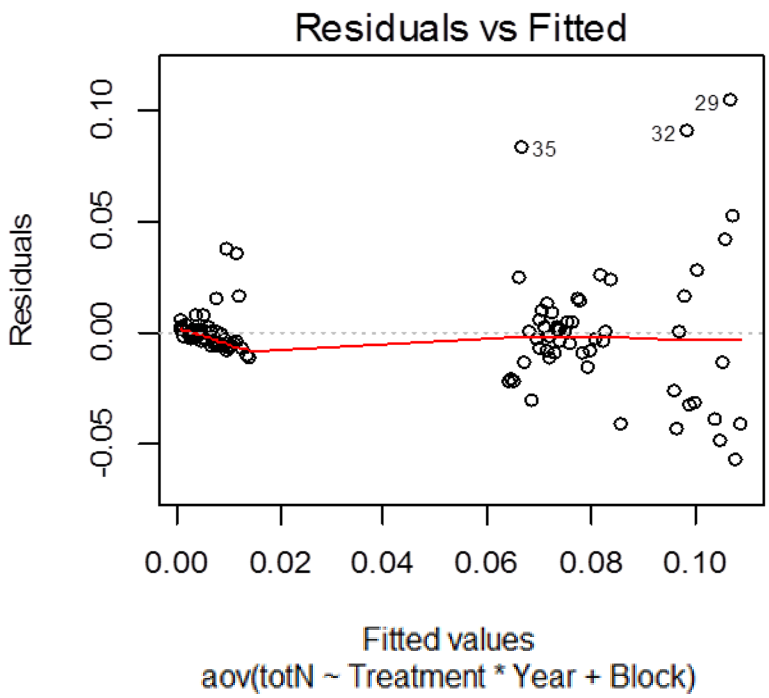
Residuals vs Fitted
Residuals
200
100
0
-100
35
9
29
20
40
60
80
100
Fitted values
aov(avbP ~ Treatment * Year + Block)



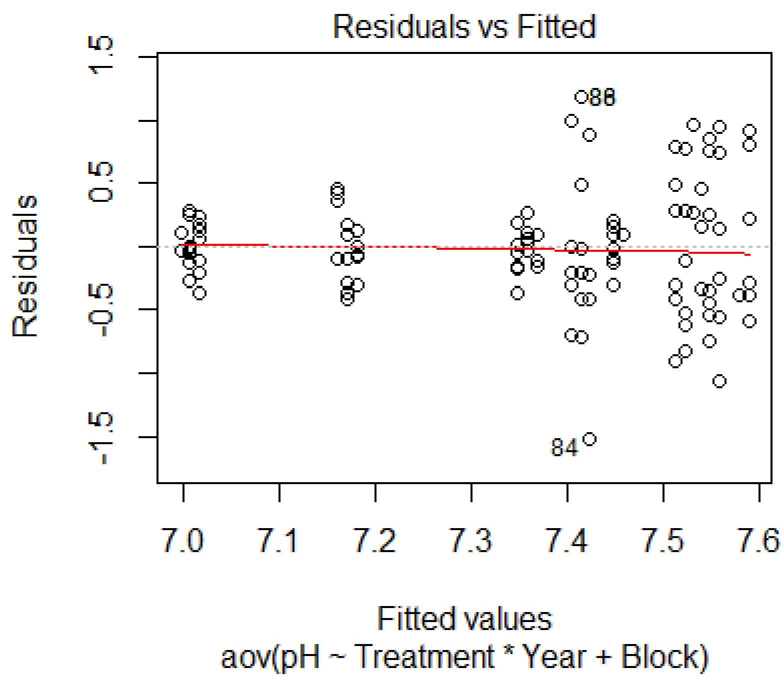
Normal Q-Q
Standardized residuals
6
4
2
0
-2
29
35
9
-2
-1
0
1
2
Theoretical Quantiles
aov(avbP ~ Treatment * Year + Block)

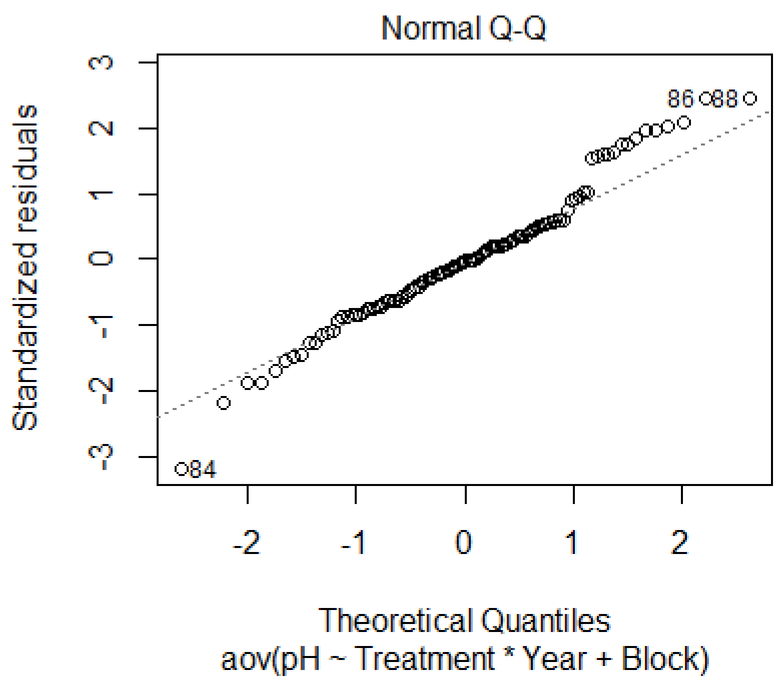


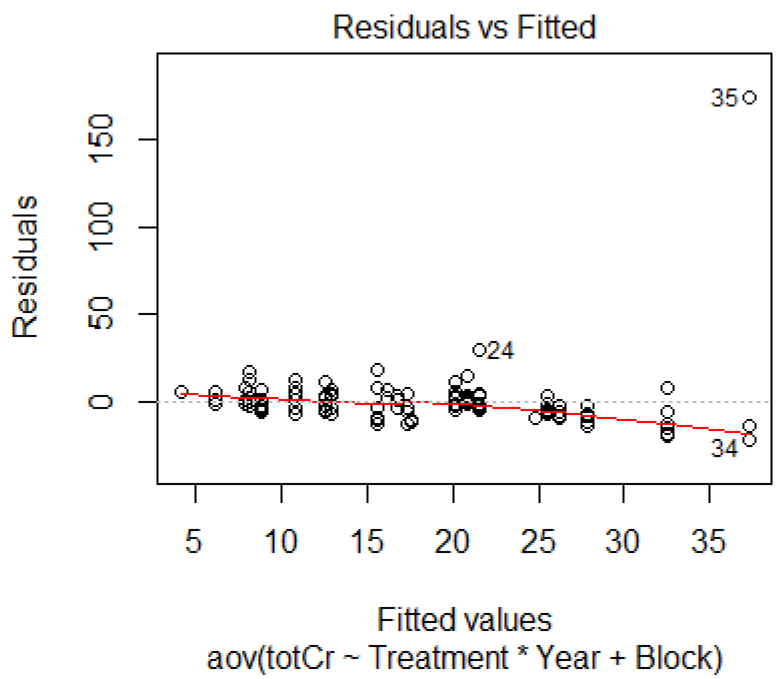
Normal Q-Q
Standardized residuals
4
2
0
-2
29
32
35
-2
-1
0
1
2
Theoretical Quantiles
aov(totN ~ Treatment * Year + Block)

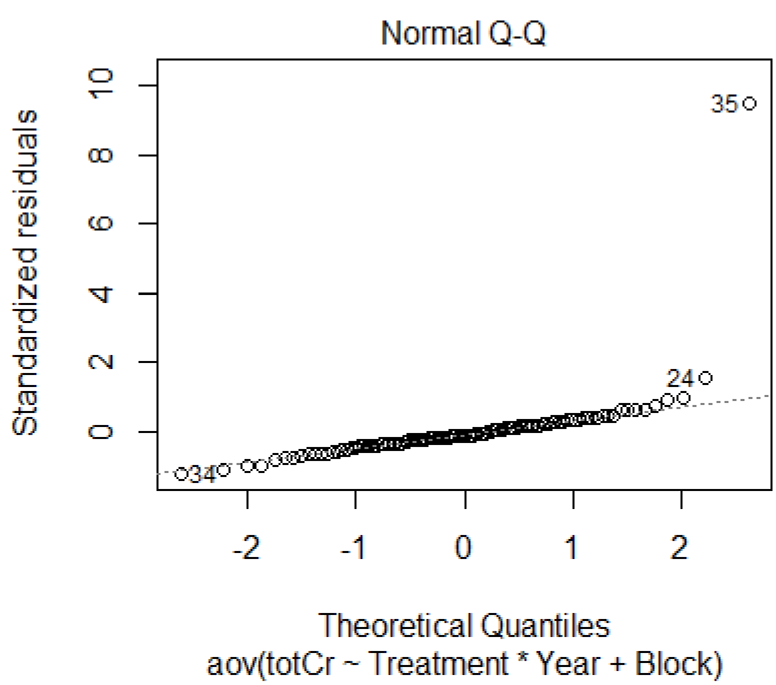

Residuals vs Fitted
Residuals
0.10
0.05
0.00
-0.05
0.00
0.02
0.04
0.06
0.08
0.10
29
32
35
Fitted values
aov(totN ~ Treatment * Year + Block)

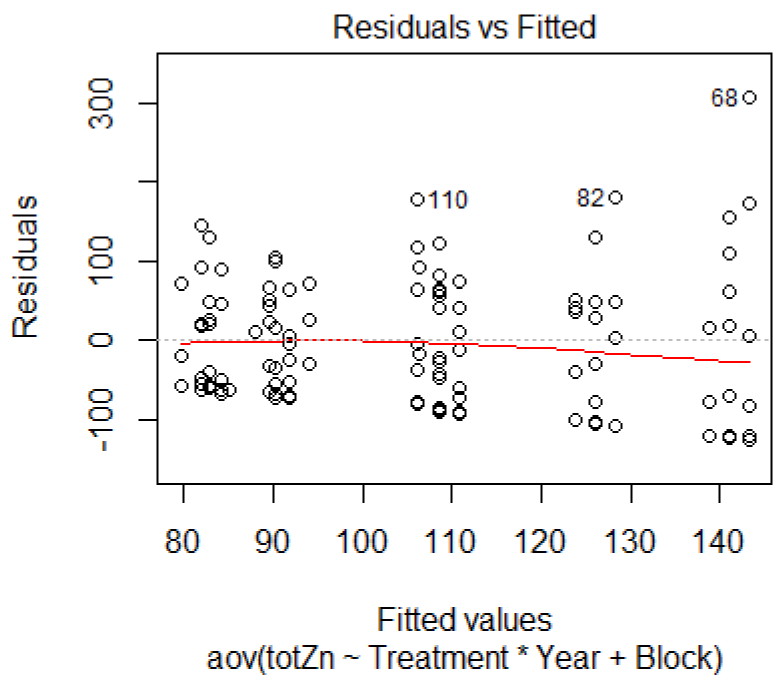
Bethlehem Depth 1 Residuals vs. Fitted plots and normality Q-Q plots



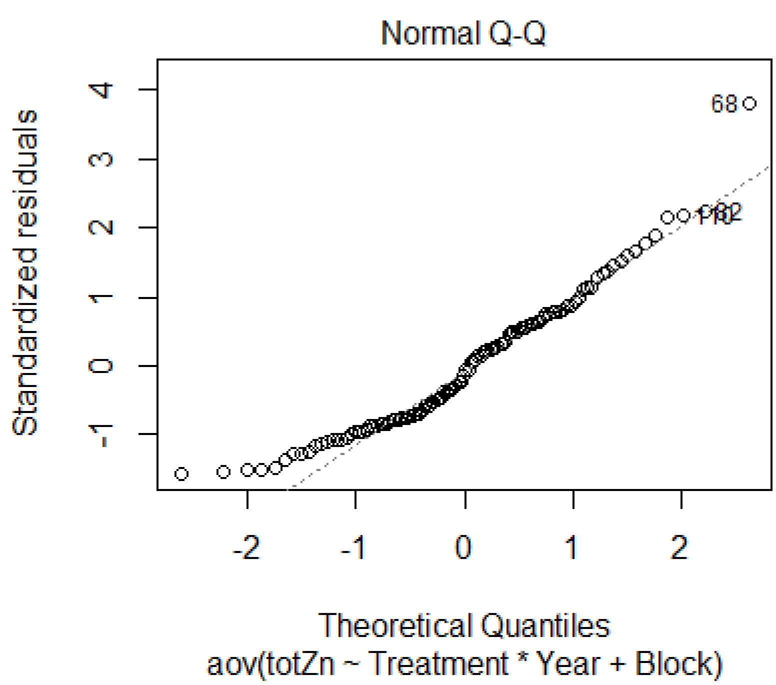



Residuals vs Fitted
Residuals
0
50
100
150
35
24
34
5
10
15
20
25
30
35
Fitted values
aov(totCr ~ Treatment * Year + Block)

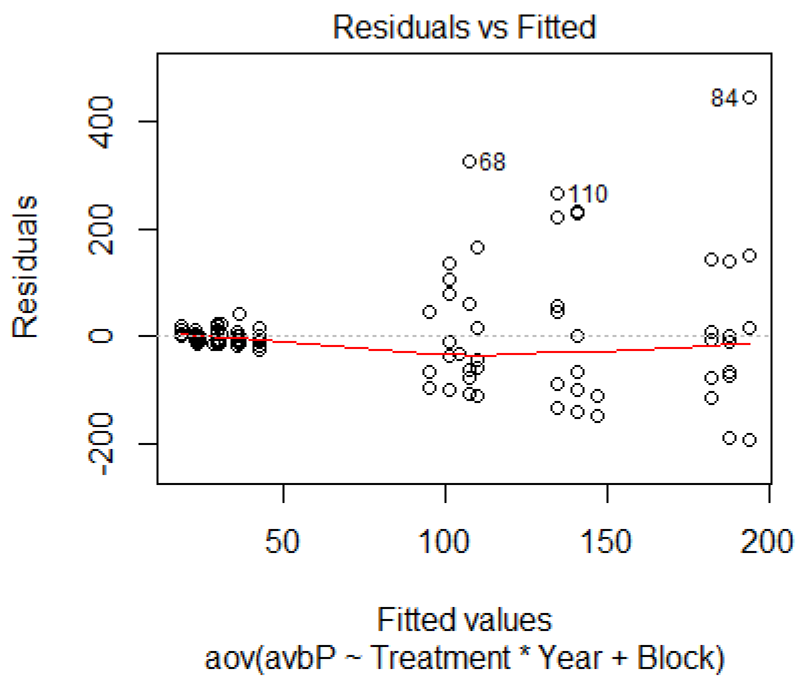

Normal Q-Q
Standardized residuals
0
2
4
6
8
10
35
24
34
-2
-1
0
1
2
Theoretical Quantiles
aov(totCr ~ Treatment * Year + Block)

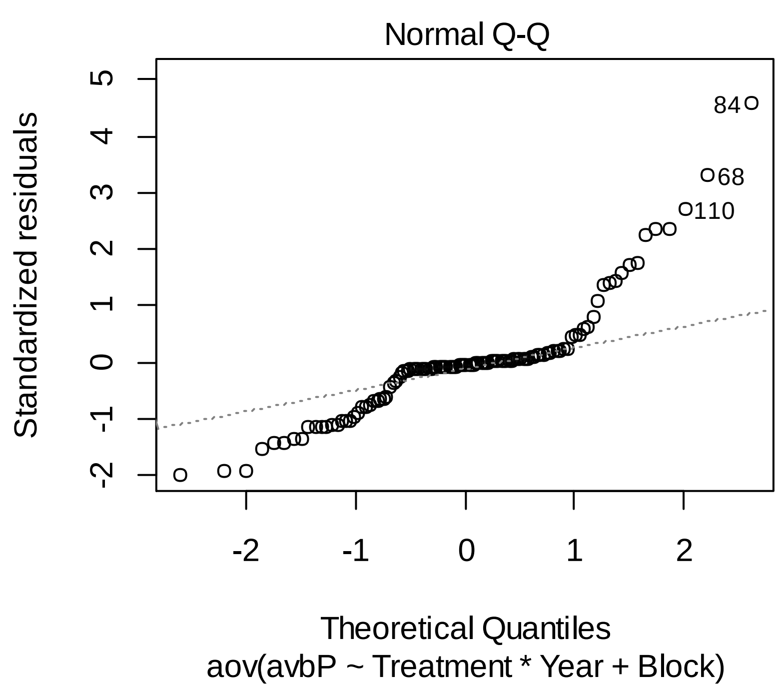


Residuals vs Fitted
Residuals
300
100
0
-100
68
110
82
80
90
100
110
120
130
140
Fitted values
aov(totZn ~ Treatment * Year + Block)

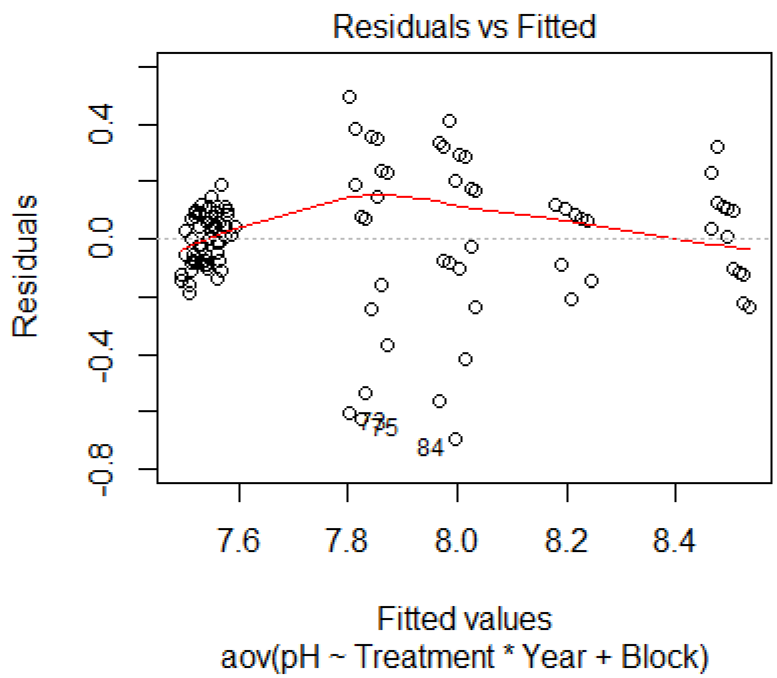


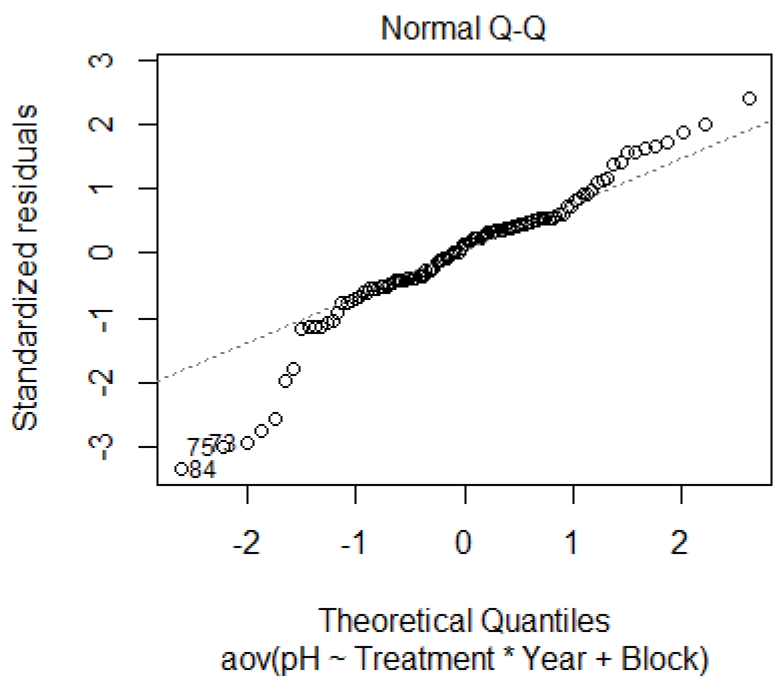
Normal Q-Q
Standardized residuals
4
3
2
1
0
-1
68
-2
-1
0
1
2
Theoretical Quantiles
aov(totZn ~ Treatment * Year + Block)

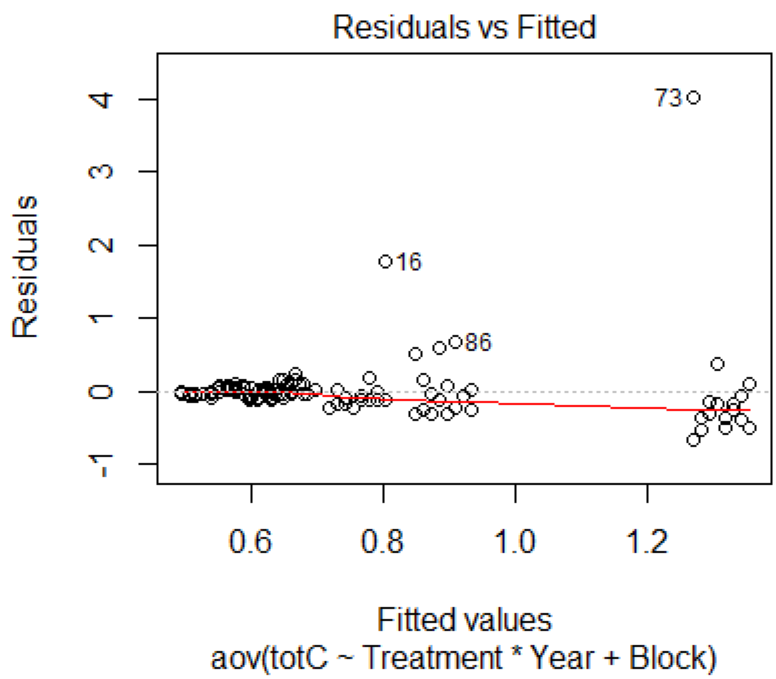

Residuals vs Fitted
Residuals
400
200
0
-200
50
100
150
200
84
68
110
Fitted values
aov(avbP ~ Treatment * Year + Block)


Normal Q-Q
Standardized residuals
5
4
3
2
1
0
-1
-2
-2
-1
0
1
2
84
68
110
Theoretical Quantiles
aov(avbP ~ Treatment * Year + Block)

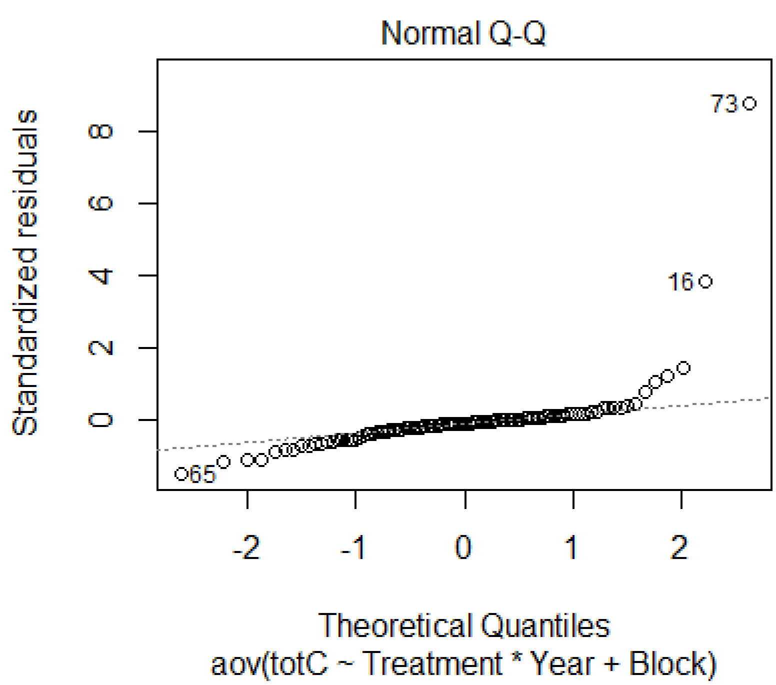
Bethlehem Depth 2



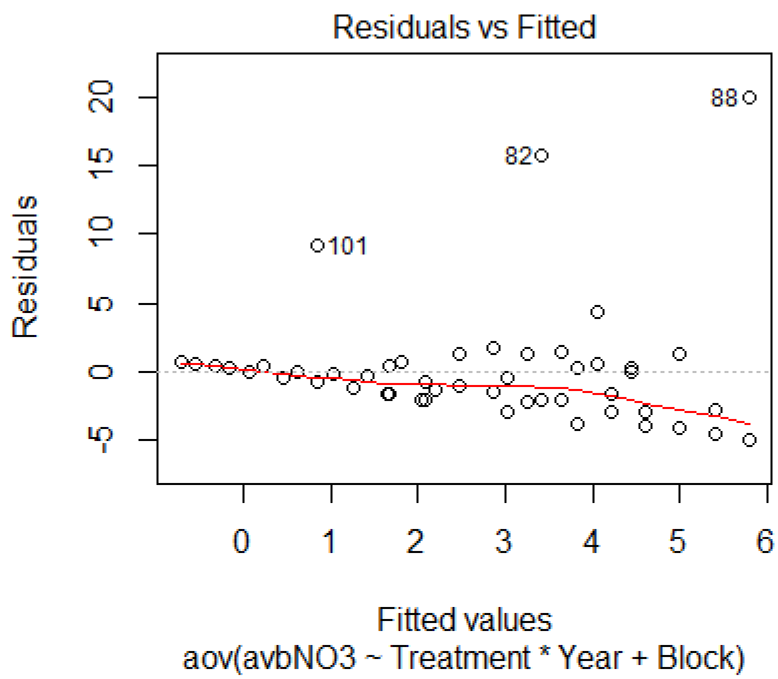


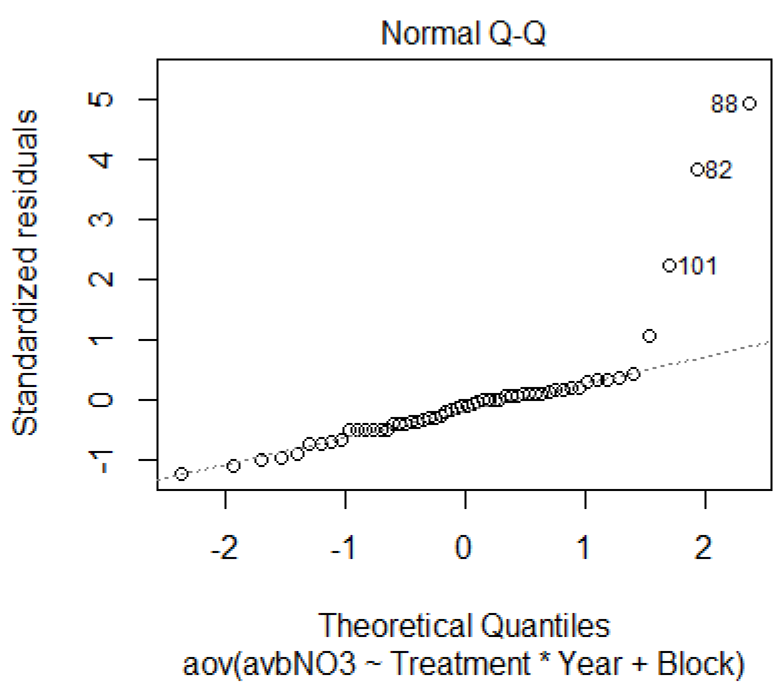


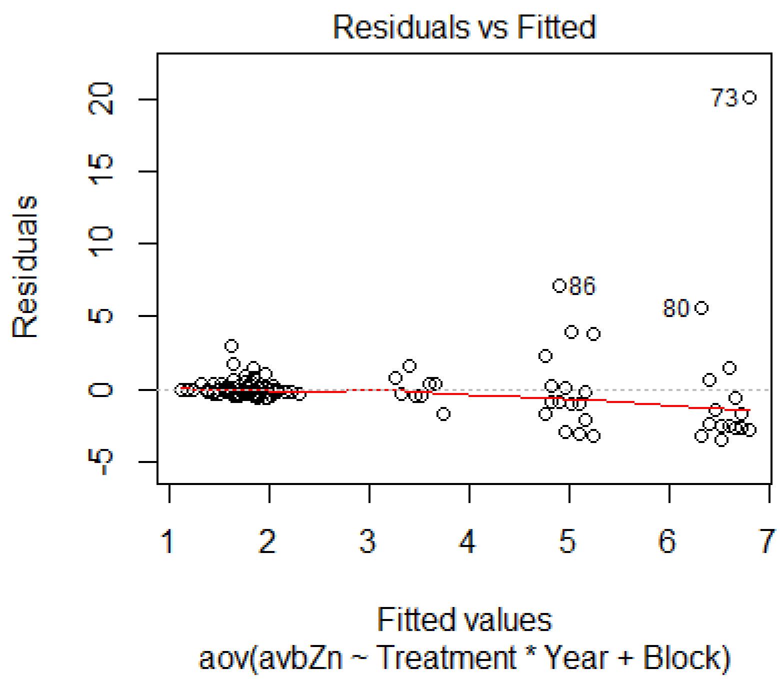
Residuals vs Fitted
Residuals
4
3
2
1
0
-1
73
16
86
0.6
0.8
1.0
1.2
Fitted values
aov(totC ~ Treatment * Year + Block)



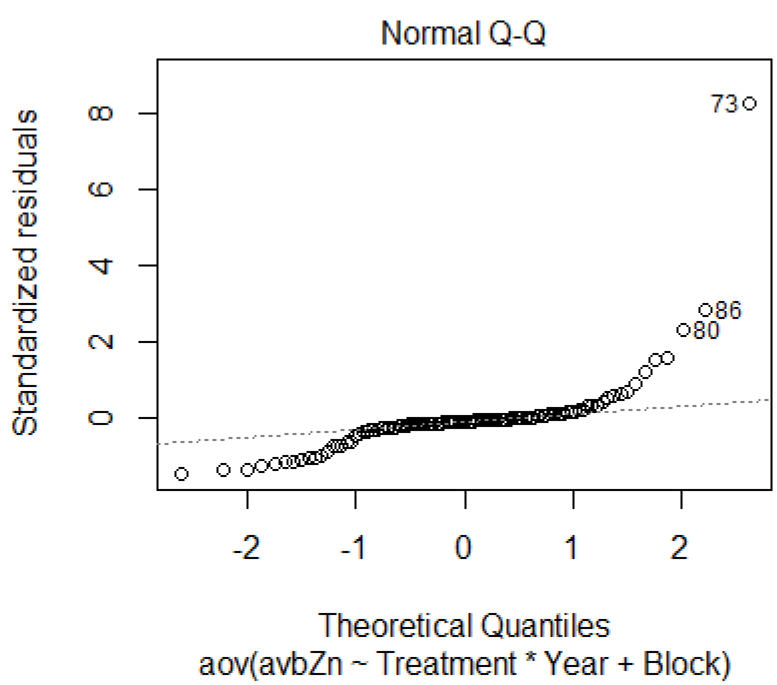
Normal Q-Q
Standardized residuals
8
6
4
2
0
73
16
65
-2
-1
0
1
2
Theoretical Quantiles
aov(totC ~ Treatment * Year + Block)


Residuals vs Fitted
Residuals
20
15
10
5
0
-5
88
82
101
0
1
2
3
4
5
6
Fitted values
aov(avbNO3 ~ Treatment * Year + Block)


Normal Q-Q
Standardized residuals
5
4
3
2
1
0
-1
88
82
101
-2
-1
0
1
2
Theoretical Quantiles
aov(avbNO3 ~ Treatment * Year + Block)



Residuals vs Fitted
Residuals
-5
0
5
10
15
20
73
86
80
1
2
3
4
5
6
7
Fitted values
aov(avbZn ~ Treatment * Year + Block)



Normal Q-Q
Standardized residuals
0
2
4
6
8
73
86
80
-2
-1
0
1
2
Theoretical Quantiles
aov(avbZn ~ Treatment * Year + Block)

Appendix 2. Two-way ANOVA results used in chapter 2 results. Each TSF and depth was tested separately, examining year, treatment, and year x treatment effects.

		Treatment Effects							Year Effects						
Tailings Storage Facility	Parameter	Depth	F value	P value	Depth	F value	P value	Degrees of Freedom	Depth	F value	P value	Depth	F value	P value	Degrees of Freedom
Sand	pH	1	173.5	<0.0001	2	98.1	<0.0001	6	1	482.5	<0.0001	2	235.9	<0.0001	1
	total As		6.1	<0.0001		4.1	<0.01			1378.8	<0.0001		2908.8	<0.0001	
	total B		0.7	0.656		0.7	0.6			319	<0.0001		336.9	<0.0001	
	total C		43.57	<0.0001		8.9	<0.0001			5.3	<0.05		70.5	<0.0001	
	total Cd		16.2	<0.0001		9.7	<0.0001			515.5	<0.0001		7786	<0.0001	
	total Co		4.1	0.001		3.8	<0.01			12.2	<0.001		33.3	<0.0001	
	total Cr		16.5	<0.0001		0.7	0.7			1286.9	<0.0001		332.5	<0.0001	
	total Cu		1.2	0.33		1.3	0.2			1.7	0.2		0.6	0.5	
	total Fe		9.2	<0.0001		1.8	0.1			8.3	<0.01		38.9	<0.0001	
	total K		1.6	0.151		0.3	0.9			0.6	0.4		65.9	<0.0001	
	total Mg		22.4	<0.0001		3.2	<0.01			8.5	<0.01		5.1	<0.05	
	total Mn		1.2	0.194		2.8	<0.05			6.3	<0.05		32.7	<0.0001	
	total Mo		0.5	0.77		2.7	<0.05			3.3	0.07		11.9	<0.001	
	total N		47.4	<0.0001		1.5	0.2			14.7	<0.001		265.3	<0.0001	
	total Ni		11.2	<0.0001		0.4	0.9			37.1	<0.0001		36.4	<0.0001	
	total P		119.1	<0.0001		6.4	<0.0001			34.4	<0.0001		4.6	<0.05	
	total Pb		28.9	<0.0001		2.4	<0.05			27.5	<0.0001		78.1	<0.0001	
	total Zn		86.4	<0.0001		4.4	<0.001			2.1	0.1		10.15	<0.01	
	available Cu		2.2	0.05		0.1937	0.9779			0.9	0.3		1.2	0.3	
	available Fe		6.8	<0.0001						2.1	0.1				
	available K		21.8	<0.0001		1.6	0.2			72	<0.0001		877.3	<0.0001	
	available		37.7	<0.0001		1.9	0.09			0.6	<0.0001		0.5	0.5	

	Mn														
	available Mo		1.5	0.2		2	0.07			0.04	0.8		0.7	0.4	
	available NO3		11.6	<0.0001		NA	NA			19.8	<0.0001		NA	NA	
	available NH4		10	<0.0001		NA	NA			21.5	<0.0001		NA	NA	
	available P		22.4	<0.0001		6.8	<0.0001			95.5	<0.0001		25.8	<0.0001	
	available Zn		51.9	<0.0001		9.8	<0.0001			0.8	0.4		143.2	<0.0001	
Silt Loam	pH	1	69.9	<0.0001	2	19.7	<0.0001		1	58.9	<0.0001	2	367.4	<0.0001	
	total As		23.8	<0.0001		7	<0.0001			911.8	<0.0001		4934.4	<0.0001	
	total B		0.8	0.6		0.184	1			187.6	<0.002		197.9	<0.0001	
	total C		153.7	<0.0001		3.9	<0.01			0.6	0.4		6.3	<0.05	
	total Cd		17.9	<0.0001		3	<0.01			69.6	<0.0001		1234.8	<0.0001	
	total Co		4	<0.01		0.5	0.8			288.3	<0.0001		306.9	<0.0001	
	total Cr		11.5	<0.0001		0.3	0.9			103.3	<0.0001		397.3	<0.0001	
	total Cu		2.8	<0.05		0.8	0.5			1.6	0.2		11.2	<0.01	
	total Fe		10.9	<0.0001		0.7	0.7			0.3	0.6		3.9	0.05	
	total K		0.5	0.8		0.5	0.8			6	0.02		0.9	0.3	
	total Mg		6.5	<0.0001		0.6	0.7			7.3	<0.01		0.8	0.4	
	total Mn		1.5	0.2		0.2	1			10.9	<0.01		6.9	<0.05	
	total Mo		2.9	<0.05		5.1	<0.001			0.08	0.8		34.4	<0.0001	
	total N		173.9	<0.0001		5.3	<0.0001			19.1	<0.0001		247.4	<0.0001	
	total Ni		28.5	<0.0001		0.3	0.9			0.01	0.9		2.6	0.1	
	total P		195.8	<0.0001		18.1	<0.0001			43.1	<0.0001		4.5	0.04	
	total Pb		27.9	<0.0001		1.4	0.2			10.5	<0.01		76.1	<0.0001	
	total Zn		53.7	<0.0001		9.1	<0.0001			12.2	<0.001		16.4	<0.001	
	available Cu		1.1	0.4		1.6	0.1			196.6	<0.0001		31.4	<0.0001	
	available Fe		6.4	<0.0001		9.3	<0.0001			99.5	<0.0001		3.6	0.06	
	available K		2.5	<0.05		0.4	0.9			123.1	<0.0001		155.2	<0.0001	

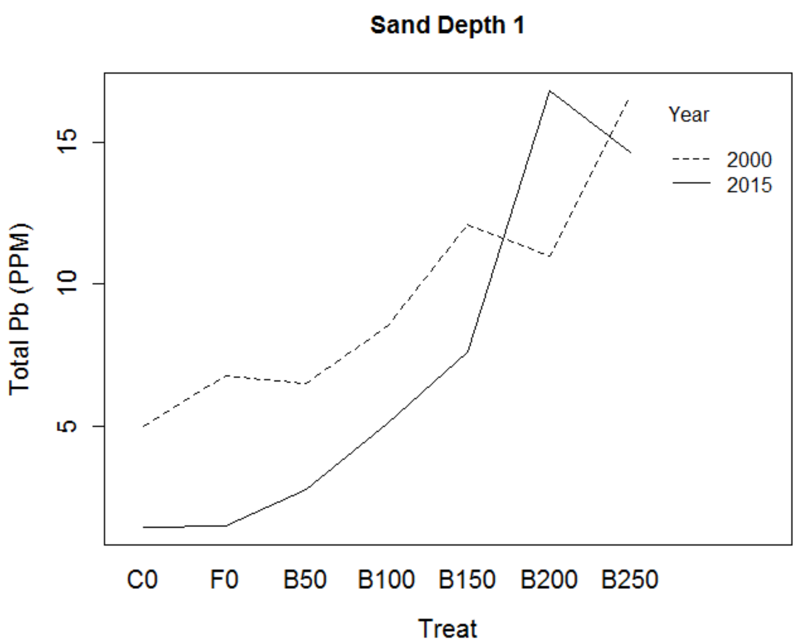
	available Mn	41.8	<0.0001	4.4	<0.001		89.4	<0.0001	17.1	<0.0001	
	available Mo	6.8	<0.0001	1.6	0.2		0.2	0.7	0.4	0.5	
	available NO3	23.4	<0.0001	NA	NA		211.5	<0.0001	NA	NA	
	available NH4	9.7	<0.05	NA	NA		6.9	<0.05	NA	NA	
	available P	19	<0.0001	2.4	0.04		88.8	<0.0001	5.8	0.02	
	available Zn	128	<0.0001	5.5	<0.0001		4.9	<0.05	37.8	<0.0001	

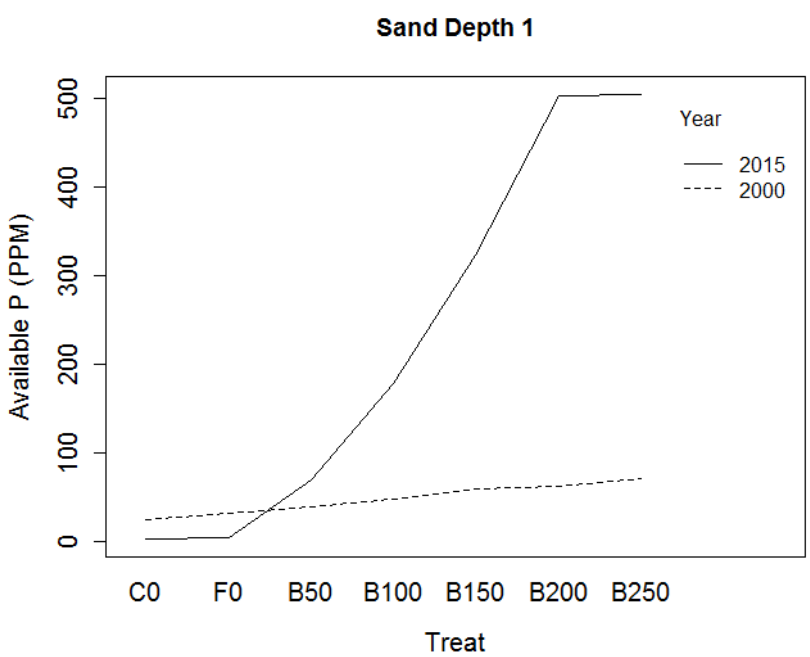
Appendix 3. 2015 total metal concentrations (ug g^{-1}) in comparison to CCME guidelines for agricultural soils

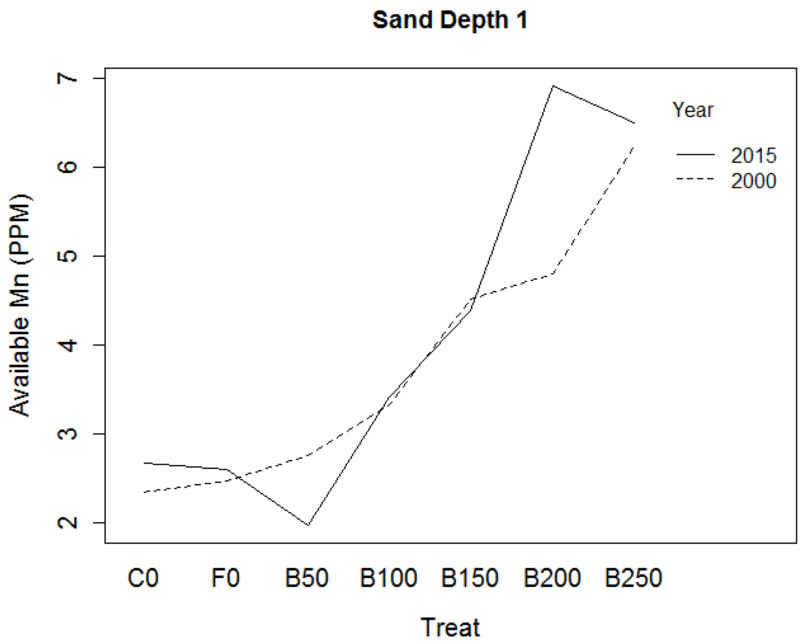
TSF	Treatment	Depth	pH	B	Cr	Cu	Mo	Ni	Pb	Zn
CCME Guidelines for Agricultural Soils			N/A	2	64	36	5	45	70	200
S	C0	1	8.9	0.1	1.0	1481.4	8.3	3.3	1.5	16.0
S	F0	1	8.9	0.1	1.2	1450.0	10.4	3.7	1.5	15.3
S	B50	1	7.6	0.1	2.5	1335.8	12.1	4.4	2.8	31.1
S	B100	1	7.3	0.2	3.8	1387.4	13.4	5.4	5.1	69.6
S	B150	1	6.8	0.4	5.4	1154.0	12.4	4.7	7.6	82.0
S	B200	1	7.0	0.4	11.3	1264.1	9.8	7.3	16.8	170.3
S	B250	1	6.5	0.4	11.7	925.7	16.3	7.3	14.7	160.6
S	C0	2	9.0	0.1	1.9	1118.9	9.5	4.5	1.3	14.4
S	F0	2	9	0.1	1.0	982.9	7.6	5.0	1.4	12.4
S	B50	2	8.3	0.1	1.4	1137.0	18.2	4.6	1.7	15.8
S	B100	2	8.0	0.2	1.5	1226.4	11.3	4.5	1.8	21.4
S	B150	2	7.1	0.1	3.2	1168.7	15.2	4.3	3.7	42.3
S	B200	2	7.0	0.1	2.4	1364.6	14.0	4.1	2.8	28.0
S	B250	2	6.9	0.1	2.6	997.6	8.8	4.9	3.3	36.0
SiL	C0	1	8.4	0.3	5.1	772.8	42.6	6.0	1.9	21.4
SiL	F0	1	8.3	0.3	4.8	665.3	33.8	5.6	1.8	20.1
SiL	B50	1	7.8	0.3	7.0	740.4	28.9	6.4	5.5	57.0
SiL	B100	1	7.3	0.6	9.8	679.3	29.5	6.8	11.2	109.1
SiL	B150	1	7.1	0.7	13.8	700.9	30.9	7.8	17.2	160.4
SiL	B200	1	7.0	0.7	18.6	860.1	33.3	10.1	26.6	230.6
SiL	B250	1	6.6	0.7	20.3	908.1	29.9	10.2	27.2	241.4
SiL	C0	2	8.5	0.1	5.1	608.4	15.1	5.3	2.3	18.0
SiL	F0	2	8.5	0.1	4.8	606.8	13.3	5.6	1.7	18.1
SiL	B50	2	8.3	0.1	4.4	626.4	15.2	4.8	2.0	21.5
SiL	B100	2	8.2	0.1	4.9	604.8	18.3	5.1	2.3	24.3
SiL	B150	2	8.1	0.2	5.5	642.6	21.6	5.2	3.0	32.3
SiL	B200	2	7.6	0.3	7.8	687.8	21.1	5.8	7.7	67.3
SiL	B250	2	7.7	0.2	6.9	624.1	23.4	5.4	5.0	47.5

Appendix 4. Interaction plots displaying elemental concentrations for each year (2000 and 2015) across biosolids application rate.

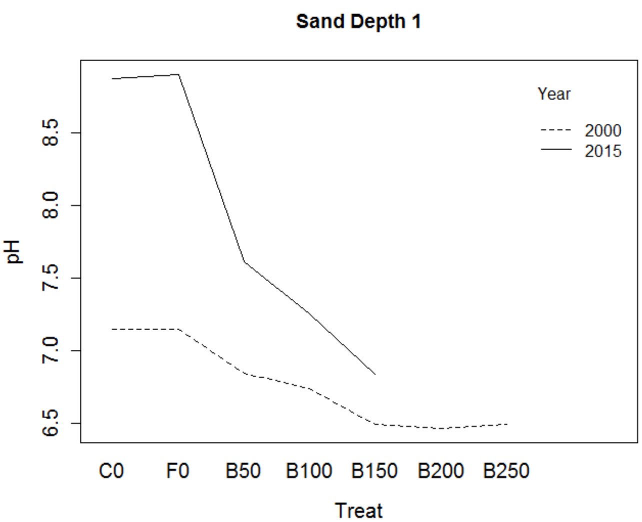
Sand TSF Depth 1





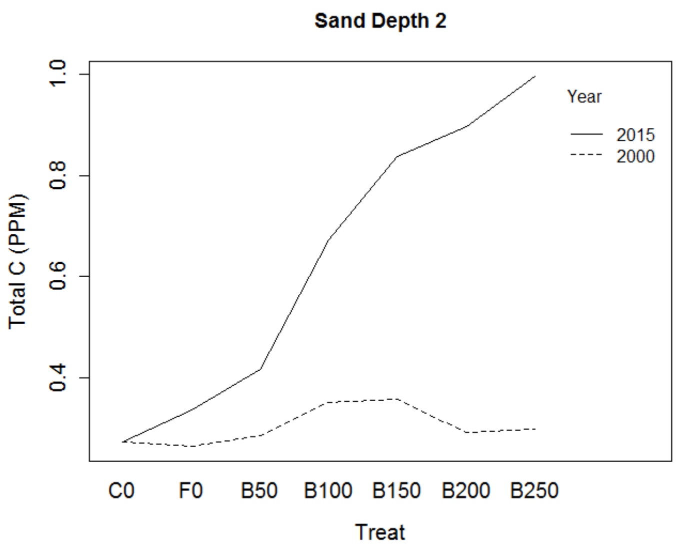


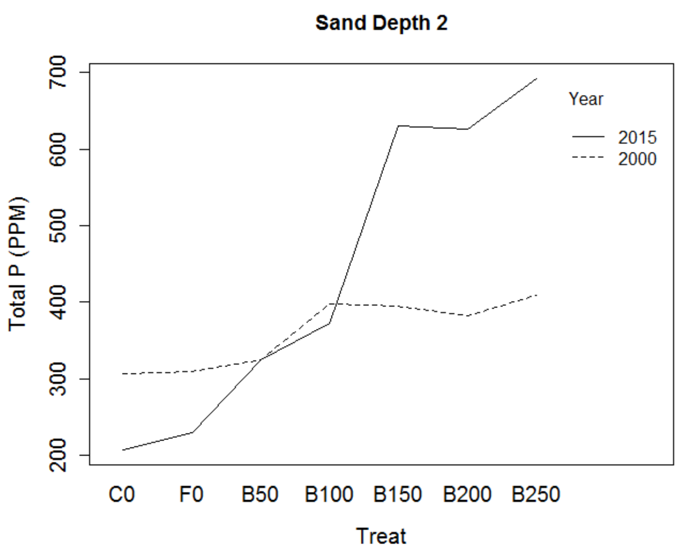
Sand Depth 1
Available Mn (PPM)
2
3
4
5
6
7
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treat

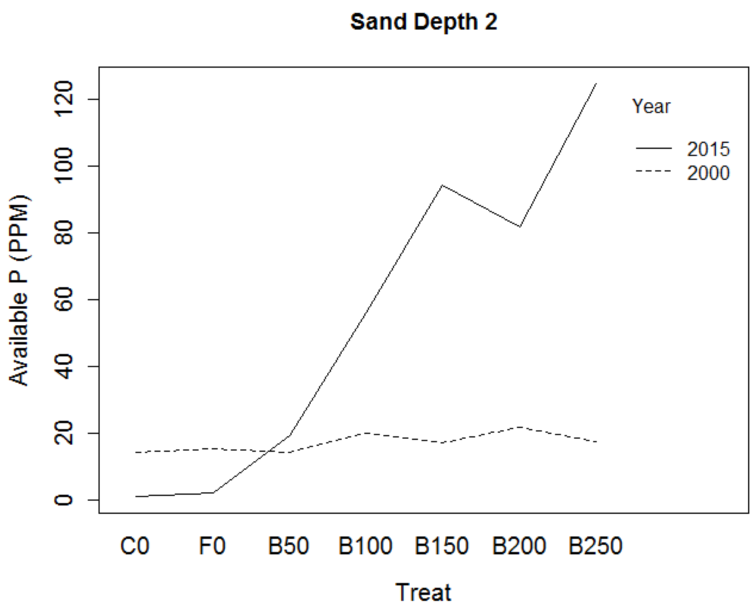


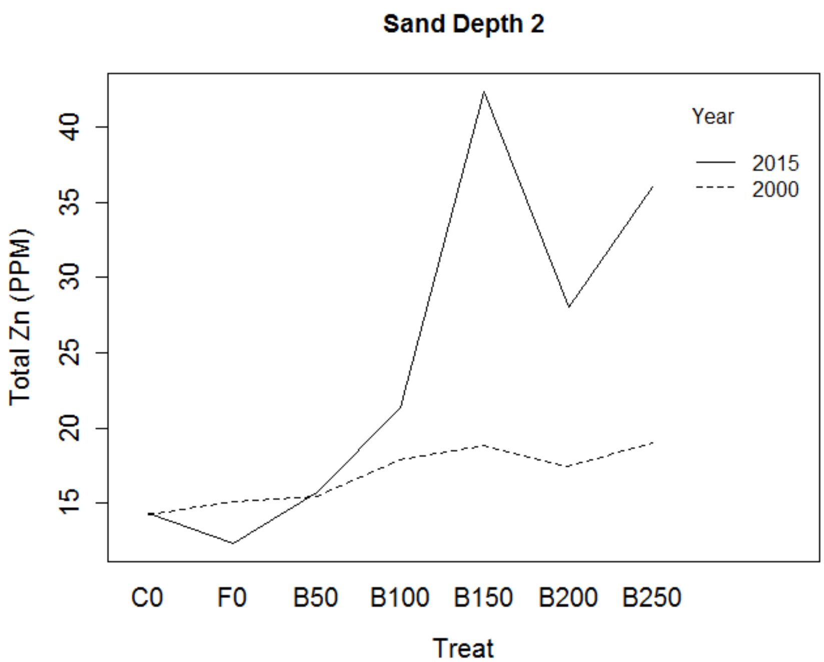
Sand Depth 1
pH
6.5
7.0
7.5
8.0
8.5
Year
2000
2015
C0
F0
B50
B100
B150
B200
B250
Treat

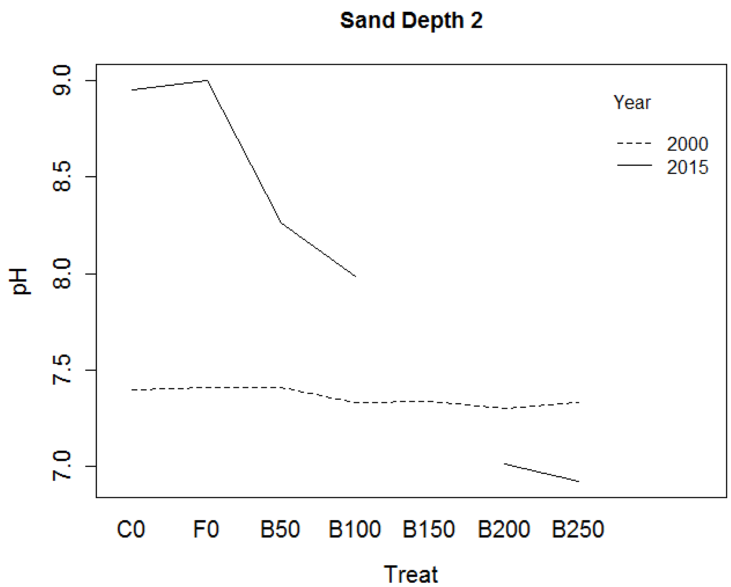
Sand TSF Depth 2



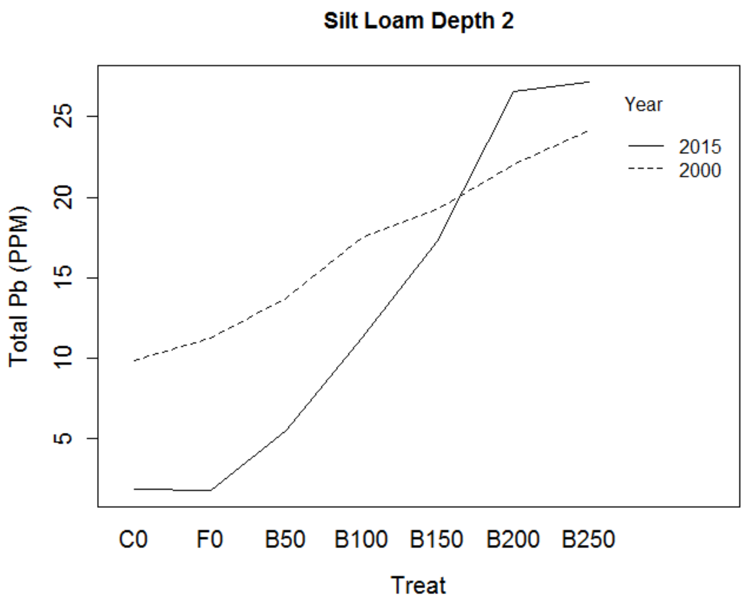


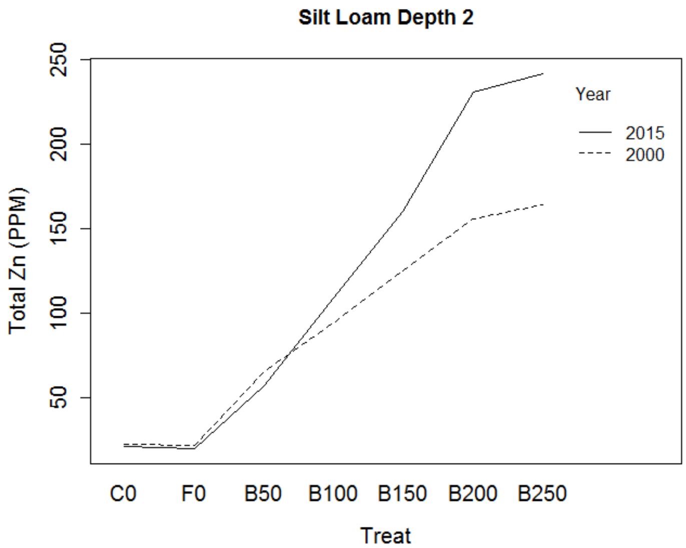

Sand Depth 2
Available P (PPM)
0
20
40
60
80
100
120
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treat

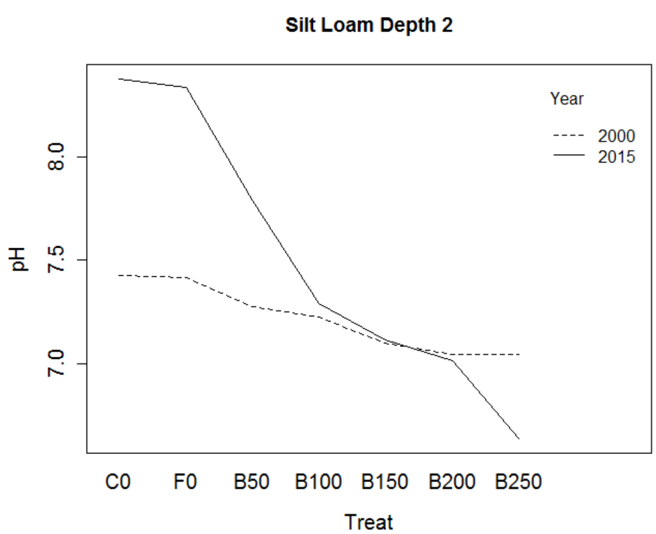

Sand Depth 2
Total Zn (PPM)
15
20
25
30
35
40
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treat


Sand Depth 2
Year
2000
2015
pH
9.0
8.5
8.0
7.5
7.0
C0
F0
B50
B100
B150
B200
B250
Treat

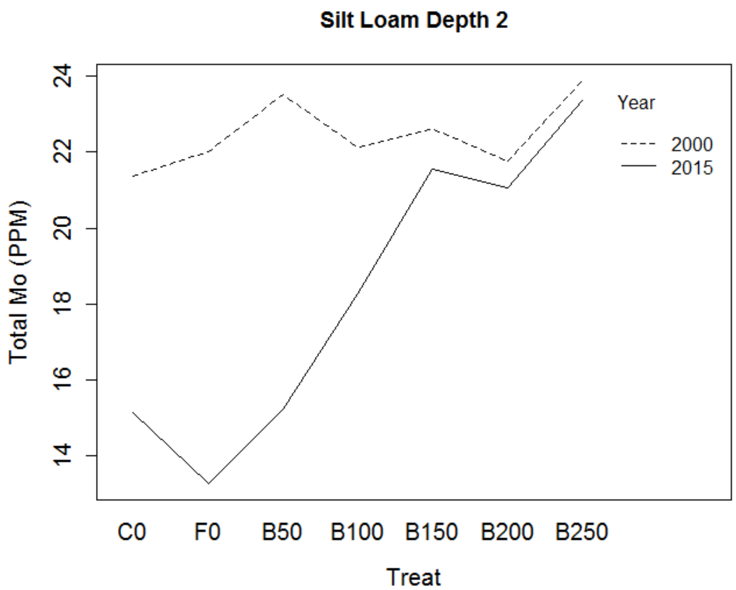
Silt LoamTSF Depth 1

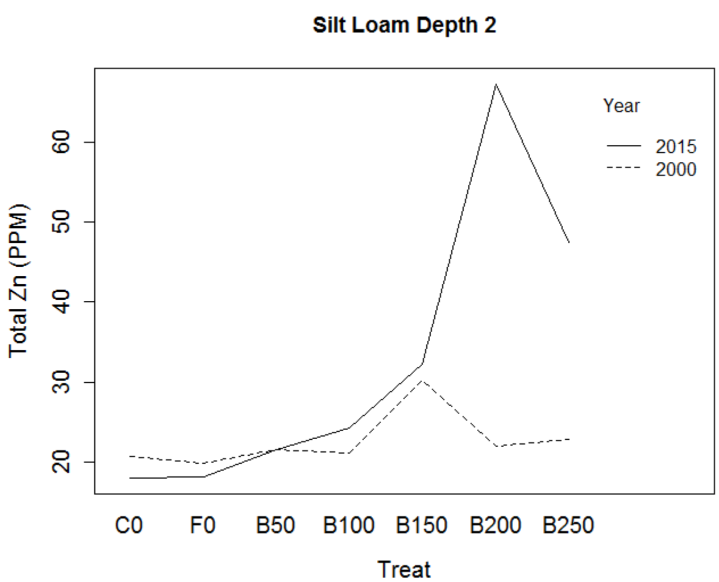


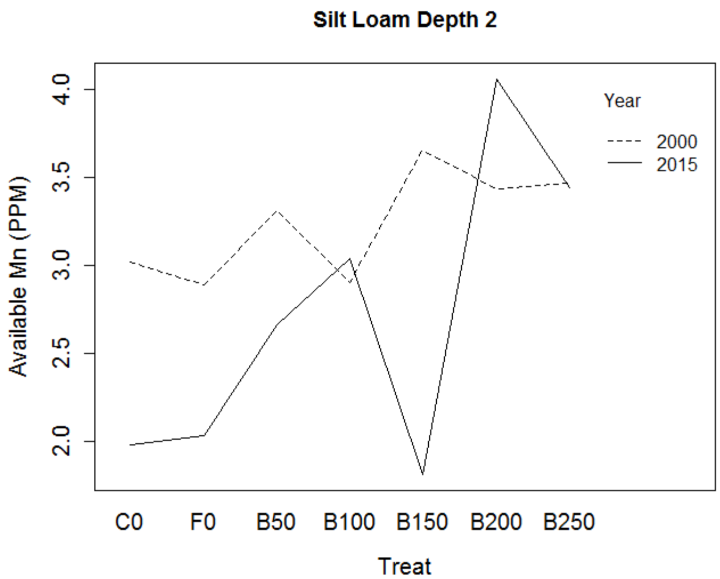

Silt Loam Depth 2
Total Zn (PPM)
50
100
150
200
250
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treat

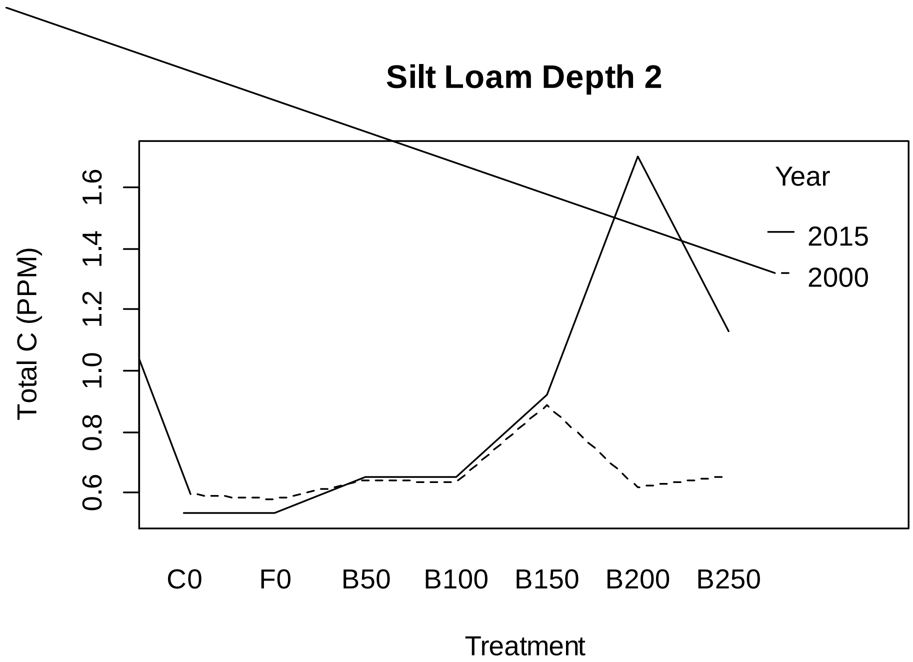

Silt Loam Depth 2
pH
7.0
7.5
8.0
Year
2000
2015
C0
F0
B50
B100
B150
B200
B250
Treat

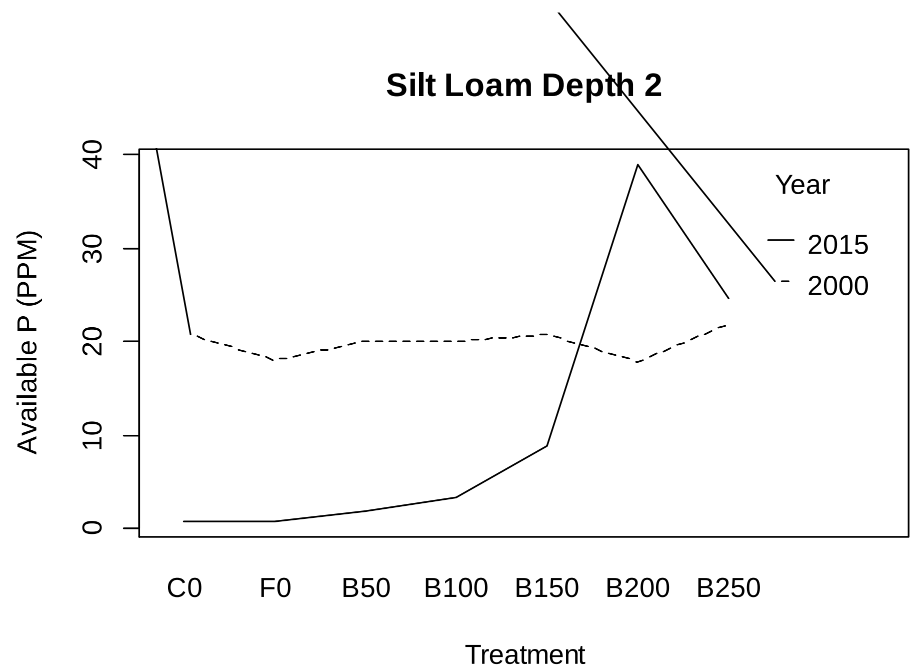
Silt Loam TSF Depth 2

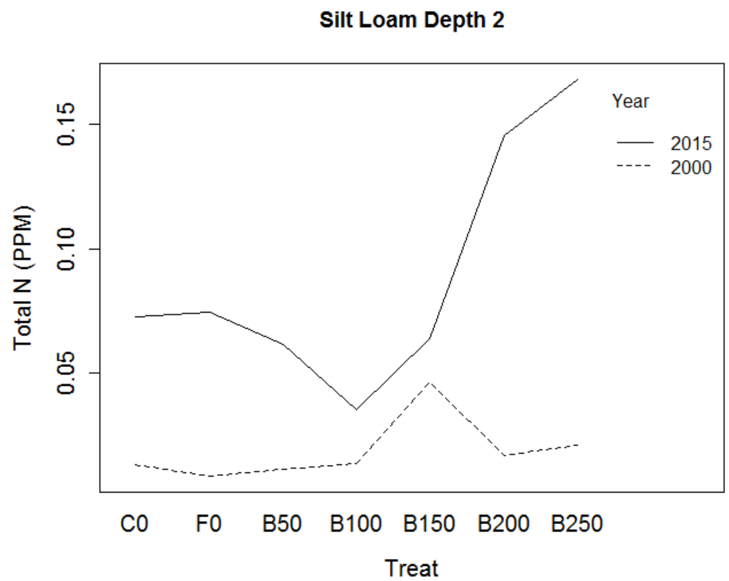


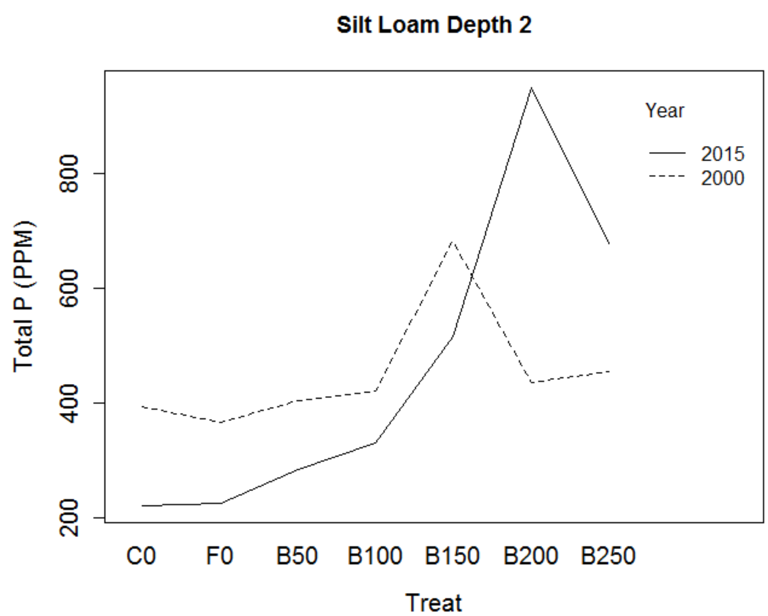


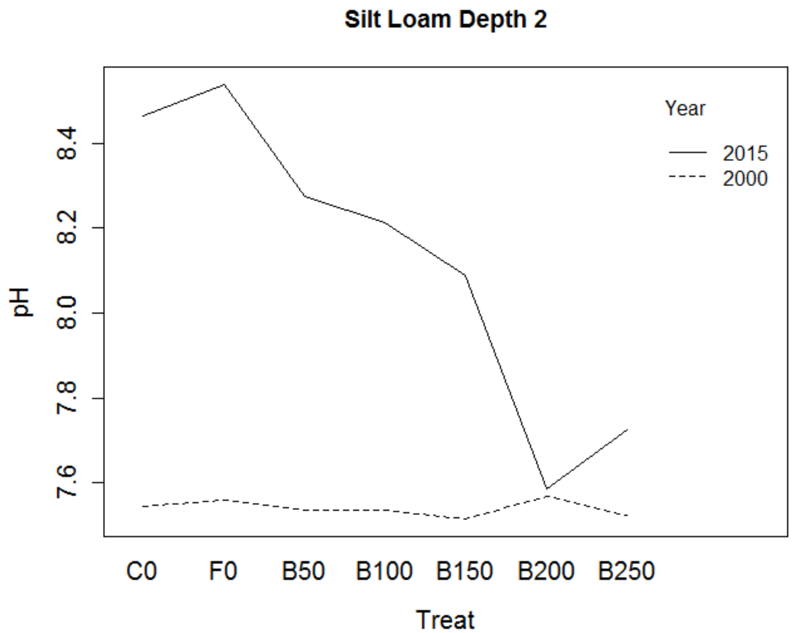

Silt Loam Depth 2
Available Mn (PPM)
4.0
3.5
3.0
2.5
2.0
Year
2000
2015
C0
F0
B50
B100
B150
B200
B250
Treat


Silt Loam Depth 2
Total C (PPM)
0.6
0.8
1.0
1.2
1.4
1.6
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treatment


Silt Loam Depth 2
Available P (PPM)
0
10
20
30
40
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treatment


Silt Loam Depth 2
Total N (PPM)
0.05
0.10
0.15
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treat


Silt Loam Depth 2
Total P (PPM)
200
400
600
800
Year
2015
2000
C0
F0
B50
B100
B150
B200
B250
Treat


Silt Loam Depth 2
Year
2015
2000
pH
8.4
8.2
8.0
7.8
7.6
C0
F0
B50
B100
B150
B200
B250
Treat