PHOSPHOGYPSUM RECLAMATION: EVALUATING ALTERNATIVE AND TRADITIONAL COVER SYSTEMS

by

Martin John Cameron Robinson

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University of Alberta

ABSTRACT

Phosphogypsum (PG) is a by-product from phosphorus fertilizer production that is stored in large piles called stacks. Stacks are formed as PG particles settle to the bottom of large holding ponds and excess water is returned to the production facility resulting in piles of dry PG. PG is comprised of gypsum with low concentrations of fluoride, trace elements and naturally occurring radioactive material from parent ore. PG stack reclamation focuses on limiting exposure to the surrounding environment by capping with soil. Environmental risks have been controlled using topsoil caps vegetated with grass. Alternative methods such as short rotational forestry plantations may provide added benefits by utilizing rapidly growing woody species to produce biomass for wood products and renewable energy, and sequester atmospheric carbon.

Five soil amendments were used in a field experiment to determine effects on *Picea glauca* (white spruce) and *Populus balsamifera* (balsam poplar) growth. After two years amendments had little effect on either species for the parameters measured. The healthiest and most successful trees were from control treatments. Tree survival was low relative to other studies with significant mortality over the 2016 to 2017 winter in all treatments. Nutrients from amendments significantly affected some soil properties but did not impact trees. Despite efforts to control unwanted vegetation such as weeds and competitive grass, use of amendments may have inadvertently benefited competing vegetation more than the planted trees.

Pure soil, PG and mixtures of both were below Canadian Council of Ministers of the Environment guidelines for all elements. There was a poor correlation between soil and plant concentrations for most elements which suggests that elements were not in a bioavailable form. Grass had lower tissue concentrations than trees for most elements analyzed. Washed and unwashed vegetation had few differences. Radium-226 activity in pure PG and highly mixed soils were above Canadian guidelines for naturally occurring radioactive material. All plant species were likely not taking up radioactive isotopes from PG. Vegetation covers differed significantly in radon-222 emissions, likely due to canopy structure, not uptake or changes from plants growing on the site.

Pure PG is not suitable as a soil to support trees on its own; by using topsoil and amendments the limitations of PG can be ameliorated. Short rotational forestry plantations appear to be meeting reclamation objectives for PG stacks, similar to traditional grass covers.

“We don’t make mistakes,
we just have happy accidents”
- Bob Ross

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1. PHOSPHOGYPSUM

1.1. Fertilizer Production

Phosphogypsum (PG) is a waste by-product from production of phosphoric acid fertilizer by wet acidification of phosphorus rock (Tayibi et al. 2009). Fertilizer production is represented by the reaction equation $Ca_5F(PO_4)_3 + 5H_2SO_4 + 10H_2O \rightarrow 3H_3PO_4 + 5CaSO_4 \cdot 2H_2O + HF$. In 2010 Canada used an estimated 1.1 million tons of phosphate fertilizer (Government of Canada 2014). Every ton of phosphate fertilizer produced yields approximately 5 tons of PG (Rutherford et al. 1994). A 2009 (Yang et al.) estimate of 280 million tons of PG produced annually around the world is expected to exceed 300 million tons in the next decade (IAEA 2013).

PG is comprised mainly of calcium sulphate dihydrate or gypsum (> 90 %); however, it contains impurities such as aluminum, iron, magnesium and sodium oxides, fluoride, organic matter and naturally occurring radionuclides (Rutherford et al. 1994). The rock source used to make fertilizer and the process used to extract phosphoric acid affects concentrations of these impurities. PG pumped from the production facility contains residual acidity from phosphoric, sulfuric and hydrofluoric acids and can have a pH of 2.1 to 5.5 (Rutherford et al. 1994, Tayibi et al. 2009). Phosphate ore used in fertilizer production can be of sedimentary or igneous origin. Sedimentary ore (phosphorites) comprises 85 % of the ore used in production of phosphorus fertilizer worldwide (Rutherford et al. 1994). Apatite is the primary mineral in most phosphate ore deposits used for phosphate fertilizer. Phosphorites are mined in many African countries and shipped to North America for fertilizer production.

PG is typically placed in large piles called stacks that can be multiple hectares in size (Hentati et al. 2015). Stacks are usually engineered with impermeable liners and leachate collection systems to prevent ground water contamination. The only currently active stack in Canada is at the Nutrien Redwater fertilizer facility in Redwater Alberta. There are many stacks in the United States and elsewhere in the world. Wet stacking is the most common PG disposal method where a PG and water slurry is pumped into settling ponds. Over time PG particles settle to the bottom of the stack and pond water is returned to the production facility for reuse. Dry stacking involves piling dewatered PG and is common in areas where water reserves are limited (Lottermoser 2010). Alternative disposal methods include back filing mines and discharging into adjacent water bodies (Hentati et al. 2015).

1.2. Chemical And Physical Properties Of Phosphogypsum

PG is comprised of 85 to 93 % gypsum and a variety of other trace elements whose concentrations can vary greatly based on the source of phosphorite ore used in production (Alcordo and Rechcigl 1993). Phosphorites are reported to have high concentrations of cadmium, uranium, silver, yttrium, selenium, ytterbium, molybdenum, lanthanum, strontium, lead and zinc which can be retained in PG after fertilizer production (Altschuler 1980, Alcordo and Rechcigl 1993, Rutherford et al. 1994). Fluoride remains in PG following fertilizer production and results in soluble fluoride in pond water from 4 to 14 g / L (Weinstien and Davidson 2004).

Fresh PG from the production facility contains residual acidity from phosphoric, sulfuric and hydrofluoric acids and can have a pH of 2.1 to 5.5 (Rutherford et al. 1994, Tayibi et al. 2009). This low pH causes many of the trace elements to be in a mobile form in solution. Over time pH in the stack increases as treatment water within the stack is flushed out and treated for reuse in production (Rutherford et al. 1995). After the pore water in the stack has been flushed multiple times, pH in the stack increases and contaminant mobility decreases.

PG has high proportions of medium (0.250 to 0.045 mm) and fine (< 0.045 mm) diameter sized particles and has a silty texture (Alcordo and Rechcigl 1993, Rutherford et al. 1994). PG colour ranges from whitish grey to brownish yellow depending on rock source and production process. PG water content is 25 to 30 % and can form a surface crust that prevents particulates from being blown off the stack (Rutherford et al. 1994, Wissa 2002). The density, strength, compressibility and permeability of PG varies with source of rock, age and location in the stack (IAEA 2013). PG at the bottom of the stack is compressed due to the weight of the stack above, resulting in low permeability. Bulk density of PG is 0.7 to 0.9 g / cm^3 (Rutherford et al. 1994).

1.3. Chemically Concentrated Isotopes In Phosphogypsum

Ore used to produce phosphate fertilizer is naturally enriched in uranium-238 and its decay products (Rutherford et al. 1994, Rutherford et al. 1996). During fertilizer production uranium-238 is partitioned into the fertilizer and radium-226, the daughter product of uranium-238, is concentrated in PG (Rutherford et al. 1996). During fertilizer production radium substitutes with calcium in gypsum crystals. Radium-226 has a half life of 1620 years and is generally considered a perpetual issue for PG management. Isotope concentration can vary in PG, although most samples are above Canadian criteria for diffuse naturally occurring radioactive material (0.3 Bq / g) (Rutherford et al. 1996, Health Canada 2011).

The main risk to humans of PG is inhalation of radon-222 gas, a daughter product of radium-226. When inhaled into the lungs, radon-222 decays into polonium-218 and can be absorbed by the lungs. Polonium-218 goes through a series of alpha and beta decay steps which can damage lung cells and increase the probability of cancer (Rutherford et al. 1996, Health Canada 2014). Radon emission from soil is dependent on radium-226 content, soil texture, humidity and soil water content (Mullerova et al. 2018). Radon-222 is the product of radioactive decay and therefore the only way to reduce its risk in PG is to create a physical barrier reducing exposure. Richardson (1997) found that relative to bare PG, PG vegetated with grass reduced radon emission in half and the placement of a 15 cm overburden cap halved the emission again. Active PG stack surfaces are typically compacted to reduce radon gas emissions until a permanent cover system can be established (IAEA 2013).

1.4. Phosphogypsum Reclamation Objectives

The main objective of phosphogypsum reclamation is to control any risks the material may have of impacting the surrounding environment. Fluoride ions, trace elements and radionuclides in PG make it an environmentally sensitive material requiring special management (Rutherford et al. 1996). Atmospheric contamination, ground water pollution and inhalation of particulates and radon gas are potential risks of PG stacks (Rutherford et al. 1994). Wind erosion of particulates from PG stacks are typically not an issue since crusts form on the surface of stacks over time. If water infiltrates into a stack it can mobilize contaminants and leach into ground water. Most PG stacks are constructed over polyethylene liners with leachate collection systems to control this risk (Rutherford et al. 1994).

Most research on PG stack reclamation focuses on cover systems to mitigate PG exposure to the environment. Cover systems are typically compacted PG or clay, high density polyethylene liners or soil and vegetation (Patel et al. 2002). Cover systems generally attempt to restrict water infiltration into PG stacks and reduce stack emissions to the atmosphere. Soil and grass vegetation covers have been a common and effective way to reduce water infiltration and risk to the environment (Hallin 2009, Hallin et al. 2010, Jackson et al. 2011, Christensen 2013).

1.5. Alternative Uses For Phosphogypsum

PG has beneficial chemical and physical properties that make it useful as an agricultural soil amendment (Alcordo and Rechcigl 1993, Papastefanou et al. 2006, Degirmenci et al. 2007, AbouRisk 2015). PG can be a nutrient source of sulfur, phosphorus and calcium and plant micronutrients when applied to agricultural soils. PG can be used to ameliorate saline, sodic, acidic and calcareous soil and raise cation exchange capacity of highly weathered soil (Rutherford et al. 1994, Lottermoser 2010). Use of PG in agriculture is limited due to concerns of radionuclide, fluoride and metal contamination of plants and water bodies (Lottermoser 2010).

In some countries PG is used in cement and building materials. PG can be used as an additive in cement, bricks and for road base stabilization (Yang et al. 2009). PG has not fully been adopted as a building material due to poor load bearing qualities, fine particle size, high water content and risks of radioactivity (Yang et al. 2009, Lottermoser 2010). Reprocessing PG to extract pure calcium sulfate or sulfur has been explored but is not considered to be economically feasible under current market conditions (Lottermoser 2010).

Utilization of PG is limited since raw gypsum is plentiful, inexpensive and typically does not have as many contaminant issues as PG. Land that could benefit from PG is often far away from production facilities resulting in high transport costs. An estimated 5 to 8 % of PG generated is recycled world wide (IAEA 2013). PG is generated at a large volume, making 100 % reuse unlikely; however, further research is being conducted to find alternative uses.

1.6. Historical Phosphogypsum Stack Reclamation

The literature on reclaiming PG stacks is limited mainly to herbaceous plant species with emphasis on reducing PG exposure to the environment. Hallin et al. (2010) conducted an experiment on PG stacks at Fort Saskatchewan to determine effects of soil capping depths on water balance and quality. After large storm events (27.2 mm) percolation into PG was low with soil caps of 16 to 24 cm. Percolation into PG was greater where PG was less compacted. The soil cap supported grass vegetation and kept runoff quality within provincial and federal regulations in most cases. Exceedances were attributed to PG dust blowing off nearby stacks and roadways into runoff frames where samples were collected for analyses. Vegetation rooted into PG in all samples sites regardless of soil cap depth. Hallin et al. (2010) concluded that the cover system for the PG stack at Fort Saskatchewan effectively prevented water from moving into the stack and reduced erosion.

There are no published studies on woody vegetation establishment on PG stacks. An unpublished study from 2013 in Visakhapatnam India converted an abandoned PG pond into a small tree plantation using no native soil (Coromandel 2016). Five native tree species, *Thespesia populnea* (L.) Sol. Ex Correrea (portia tree), *Salvadora persica* L. (miswak), *Casuarina equisetifolia* L. (beech sheoak), *Parkinsonia aculeate* L. (jelly bean tree) and *Suaeda* maritime L. Dumont. (herbaceous seep weed) were grown on PG by adding fly ash, alkaline sludge, compost from a sugar industry and a bio fertilizer containing mycorrhizal fungi. Each tree had a drip irrigation system that provided two liters of water to each tree daily. Each year after the experiment was established, each tree received 0.5 kg of compost to sustain micro and macro nutrients and enhance microorganisms in the substrate. Individual trees were weeded to remove competition and pesticides were used to eliminate pests. All planted species grew well over the two year study and there was evidence of roots penetrating untreated PG. This intensively managed study showed trees could be grown in PG but the quality of the vegetation is unknown. It is unknown whether PG can support tree growth without such inputs to the soil.

2. SHORT ROTATIONAL FORESTRY

2.1. Alternative Reclamation Strategy

Short rotational forestry systems, also called bioenergy plantations, utilize rapidly growing woody plant species to grow large quantities of biomass on marginal land (Laureysens et al. 2004b). Typically, *Populus* and *Salix* species are used in these plantations since they can produce a large amount of biomass in a short period of time and can tolerate marginal growing conditions. *Picea* and *Pinus* species are sometimes used when larger wood products are prioritized. Biomass grown from these plantations can be used as a fossil fuel substitute and for application of carbon credits from carbon sequestration. Short rotational forestry plantations provide an opportunity to convert marginal land into low input forests capable of generating capital to offset production costs.

Short rotational forestry plantations have been used in Canada since the 1980s and has recently received federal funding through the Canadian Biomass Innovation Network (Kenny et al. 1991, Government of Canada 2015). Trees are typically planted in rows with variable spacing based on the stems required per hectare. High yield afforestation, concentrated woody biomass and mixed wood afforestation are types of plantations utilized across Canada (Keddy 2017). Plantations can be established for different harvest cycles based on species used, planting density and timber requirements. Often a portion of a plantation will be harvested before the end of a cycle to reduce competitive pressure on surrounding trees.

Forestry plantations are a potential reclamation strategy for PG stacks that provide environmental and economic benefits to the land owner relative to traditional grass cover.

Although forestry plantations provide economic and environmental benefits with PG stacks from carbon sequestration; the risks associated with PG must be managed. Forestry plantations have a different canopy structure than grass covers and vegetation between trees is removed to promote tree growth, resulting in bare soil. This may affect the amount of radon being emitted into the atmosphere and may alter the water dynamics in soil and PG. Trees are physiologically different than grass and may uptake trace elements and radionuclides differently from PG.

2.2. Carbon Sequestration

Since 1880 mean annual temperature on earth has increased by 0.85 ºC (0.65 to 1.06 ºC) due to anthropologic activity (IPCC 2013). Industrial and commercial emissions of carbon dioxide, methane, nitrous oxide and chlorofluorocarbons have led to changes in the earth's climate. Earth's ability to control carbon dioxide in the air is thought to have decreased due to deforestation, wetland drainage and conversion of natural land to agricultural land (Lal 2004). Some believe climate change is not the fault of humans, as higher temperatures have been observed and documented before humans evolved. On the contrary, the Intergovernmental Panel on Climate Change states with virtual certainty that atmospheric warming is a result of human impact (IPCC 2013). Climate change has gained global attention and many countries have set emission reduction goals and signed international treaties to reduce their emissions. Applying conservative agricultural practices such as reduced tilling, crop rotation, leaving crop residues on the field and reducing chemical inputs to soil are ways producers can reduce carbon emissions by reducing soil disturbance and promoting carbon sequestration.

Carbon sequestration is the removal of atmospheric carbon by plants and storage of fixed carbon in soil organic matter (Lal 2004). Plant biomass eventually degrades in the soil, increasing soil carbon. The rate of sequestration is reliant on climate, growing conditions, plant species and management practices. Global carbon sequestration potential has been estimated at 0.9 ± 0.3 Pg / yr and could offset nearly one third of anthropogenic emissions (IPCC 2013).

Grass accumulates carbon rapidly as it establishes, whereas trees initially accumulate it slower since litter inputs are small when trees are young (Post and Kwon 2000). Over time this changes as trees get larger and inputs from trees exceed the amount from grass. Carbon sequestration from trees varies with species, planting regime, management and climate of the area. Nair et al. (2009) compared numerous global studies and found carbon sequestration from 0.29 to 15.21 Mg / ha / y from above and below ground biomass. The North American studies estimate potential sequestration between 0.83 to 1.37 Mg / ha / y from agroforestry systems planted with hybrid poplar clones. Natural grasslands in North America have been estimated to sequester 0.2 Mg / ha / y of carbon in cool arid climates and 0.4 Mg / ha / y in warmer areas (Bruce et al. 1999). Arevalo et al. (2011) found total carbon sequestered in biomass and soil was greater with *Populus deltoides* (cottonwood) than *Brassica napus* L. (canola) after 9 years. Soil carbon decreased by 7.9 Mg / ha after planting but was equal to initial levels after 7 years. The researchers concluded that with greater than four year rotations *Populus deltoides* will sequester significantly more carbon than *Brassica napus.*

3. AMENDMENT EFFECTS ON TREE GROWTH

3.1. Mycorrhizal Fungi

Mycorrhizal fungi form symbiotic relationships with roots of host plants to increase nutrient uptake efficiency, provide access to unavailable minerals, increase productivity and provide stress resistance (Bonfante and Anca 2009). Mycorrhizal hyphae are very fine branch like cells that grow in the soil and access nutrients that would be unavailable to the root. Host plants provide the fungi with carbohydrates necessary to complete their life cycles. The relationship between host plant and fungi is often species specific; however, some hosts can be colonized by different fungal species and some fungi can colonize multiple host species (Linderman and Call 1977). Ectomycorrhizae and endomycorrhizae are the two major groups of fungi that colonize plant roots. Ectomycorrhizae colonize the outside of host roots and significantly alter root morphology of the host plant (Bonfante and Anca 2009). Endomycorrhizae penetrate the root cells of host plants and establish intracellular symbiosis.

3.2. Black Earth

Black Earth is a mined product from naturally occurring oxidized coal that is rich in humified organic matter, humic and fulvic acids with very low impurities (Black Earth 2013). Humic acids, humates, humate based products, bio stimulants and natural organic fertilizers are common names used for products such as Black Earth (hereafter humates). Humates are rich in organic matter and are often marketed to as a natural supplement for plant growth and productivity.

Plant growth has been positively correlated with humate addition with a greater impact on below ground than above ground biomass (Chen and Aviad 1990). Typically, there is an increase in growth with humate application, with a diminished return when applied excessively. Black Earth

(2014a, 2014b) completed two studies on their products and found a significant increase in above ground *Brassica* biomass and total *Avena sativa* L. (wheat) biomass when applying 44.8 kg / ha and 46.8 L / ha of humate, respectively, relative to untreated plants. Although humates generally improve plant growth, some studies had apposing outcomes. Kelting et al. (1998) assessed three humate products on *Acer rubrum* L. (red maple) and all had no effect on root length or volume. Fraser and Percival (2003) found that two of three humate products increased plant growth, although not all were equally beneficial on different tree species. Thus individual humate products have different effects on vegetation and do not have as consistent and predictable yields as chemical fertilizers.

3.3. Municipal Waste Compost

Municipal waste production has increased as the world population increases. Many cities and governments are making commitments to reduce the volume of waste deposited in landfills by recycling the organic fraction of municipal waste. The City of Edmonton is a leader in this regard; diverting 90 % of their waste from landfills and generating 160,000 tons of organic compost each year (City of Edmonton 2016). When applied to soil, municipal waste compost can improve soil physical and chemical properties and enhance soil microbial activity (Shiralipour et al. 1992). Compost quality varies as the waste used to generate it is not consistent throughout the year. The City of Edmonton attempts to standardize the contents of its compost by adding different amounts of waste, plant residue and biosolids throughout the year (City of Edmonton 2016). Bengtson and Cornette (1973) found that incorporating compost into soil increased soil water holding capacity but decreased nitrogen from immobilization by soil microorganisms. Compost with low nutrients and high carbon content should be applied conservatively to prevent immobilization of soil nutrients.

A concern with municipal waste compost is the possibility of contaminating soil with metals, organic contaminants and microbial pathogens (Shiralipour et al. 1992). The Canadian Council of Ministers of the Environment guidelines for compost quality outlines maximum allowable concentrations of contaminants in compost (CCME 2005). These guidelines separate compost into categories based on the concentrations of contaminants. Category A compost can be used unrestricted in any application because it is below all standards of the guidelines. Category B compost has one or more parameters above the standards outlined in category A and has restricted application. Category B compost can be used conservatively to prevent over application of one or more contaminant.

3.4. Manure Pellets

Animal manure contains useful plant macro and micro nutrients that increase plant growth when applied to soil (Moore et al. 1995). Manure amendments typically includes all material on farms including feces, urine, bedding, waste feeds and feathers or hair. Larcheveque et al. (2011) studied impacts on hybrid poplar growth with manure and equal amounts of chemical fertilizer. Initially manure significantly increased soil nitrogen and water holding capacity but after two years the effect was not significant. Growth of chemically fertilized *Populus* trees was greater than those with manure. Although not statistically significant, manure at low and high application rates increased height, leaf area and total biomass relative to controls.

Concerns with the use of manure on land include the potential for over application of nitrogen and phosphorus causing eutrophication in water bodies, pathogenic microorganisms infecting animals and trace contaminants from pharmaceuticals given to animals (Moore et al. 1995). Logistical problems with manure include the presence of weed seeds, unappealing odour and high mass making transport expensive. Nutrients in manure are often heterogeneous because manure is composed of different materials with different chemistry. This makes application of manure imprecise relative to chemical fertilizers (Land Resources Network 1993). Turning and composting manure piles increases homogeneity, making nutrient distribution more consistent.

4. VEGETATION QUALITY

4.1. Browse

Common herbivores in central Alberta are *Odocoileus virginianus* (white tailed deer), *Alces alces* (Western Canadian moose), *Lepus americanus* (snowshoe hare) and various small rodents. At the research site, *Odocoileus virginianus* is frequently seen on and around the stacks. *Odocoileus virginianus mainly* consume grasses and herbs in spring, leaves from shrubs and trees in summer and twigs from woody plants in fall and winter (Government of British Columbia 2000). Its desired vegetation is growing at Nutrien on PG. There is concern that elements in PG can be taken up by plants and passed through the food chain when organisms eat those plants (Pulford and Watson 2003). The potential for these contaminants to accumulate in animals that will then travel off site may be a risk to the surrounding environment.

Different plant species have different ways of accommodating high or low concentrations of nutrients, elements, contaminants and water. Plants deal with contaminants in soil generally by accumulating or excluding them (Baker 1981). Accumulators will take up contaminants from soil and store them within different organs and tissues based on the plant species accumulating. Accumulators can concentrate contaminants in various components of their biomass at a higher concentration than what is found in soil. Excluders maintain a low concentration of contaminants regardless of soil conditions and concentrations. Accumulators efficiently remove contaminants from the soil; however, biomass may become a hazardous material in the process (Pulford and Watson 2003). Accumulators can be used as natural soil cleaners and filters to remediate soil instead of disposing it to landfills, which is a common practice in Alberta (Landberg and Greger 1996, Pulford and Watson 2003). Biomass from plants used for remediation may need to be handled with special consideration depending on concentrations in tissues.

Radioactive isotopes in soil can be taken up by plants, although correlation between soil and plant transfer is variable (Million et al. 1994, Rutherford et al. 1996, Gerzabek et al. 1998). The amount an isotope that is taken up by a plant depends on availability of the isotope in soil and the species of plant. Most radioactive isotopes are elements with availably that is greatest at low pH (Gerzabek et al. 1998). If animals eat radioactively contaminated vegetation, cancer risk is increased (Health Canada 2011). There is a poor understanding and lack of documentation on how radioactivity affects plant growth and plant processes.

Determining effects of individual elements on a specific plant or animal species can be difficult due to the complexity of biological processes, animal diet and metabolism and availability of the elements in soil (Natural Resource Council 2005). As a result, criteria for vegetation quality are not available for most elements. Often a more acceptable approach is to determine the maximum tolerable levels that likely present no negative impact to animals (Natural Resource Council 2005). Maximum tolerable levels are determined for specific elements by doing meta analysis on data from a large number of studies. Recommended concentrations are determined for chronic (> 10 day) ingestion by animals. Although this approach is not inclusive for all elements or species, it provides a reference that can be used for comparison and for determining potential impacts for given elements and species.

4.2. Plant Uptake Of Trace Elements

4.2.1. Woody vegetation

Populus species and clones can differ in uptake of contaminants and partitioning into different plant structures (Laureysens et al. 2004a). *Populus* stores significantly more metals in leaves than in wood or bark. Laureysens et al. (2004a) found *Populus* species allocated more metals to senescing leaves than to young or mature leaves. One hypothesis states that as photosynthesis declines by the end of the growing season the plant will transport metals to leaves before they fall from the tree. A contradictory hypothesis states that as leaves enter senescence the cuticle breaks down and presents a pathway for airborne metals to bind to callus tissue in the leaf.

Landberg and Greger (1996) found that *Salix* and *Populus* species can exhibit similar uptake variability. They concluded that some *Salix* species accumulate more metals in roots than in shoots to preserve the health of the shoots and to prevent an impact to photosynthesis. This conclusion was reached because the soils were contaminated with cadmium, copper and zinc which was shown to inhibit photosynthesis in other studies (Landberg and Greger 1996, Stiborova et al. 1986). Rosselli et al. (2003) found that even under highly toxic conditions *Salix viminalis* L. (basket willow) did not show any deficiency symptoms or reduced growth even though root and shoot concentrations of metals were high. Some *Salix* species are better at tolerating contaminated soils while some have reduced growth or cannot survive harsh conditions (Pulford et al. 2002). When selecting species to grow on a contaminated site planting multiple species or clones to determine variability in uptake and growth response is necessary.

4.2.2. Herbaceous vegetation

Much like woody vegetation, grass species exhibit capabilities of accumulating or tolerating different contaminant concentrations in soil (Ernst et al. 1992). Grasses tend to allocate the least amount of metals to reproductive organs; however, in metal rich soils non-tolerant grasses rarely survive to reproductive stages. Elevated metal concentrations in soil can inhibit root growth by reducing root cell division and size (Cox and Hutchinson 1979, Ernst et al. 1992). Grasses differ from trees in that their life cycle is shorter, creating a greater potential for grass populations to adapt to contaminants from genetic variability (Cox and Hutchinson 1979). Cox and Hutchinson (1979) found that *Deschampsia cespitosa* L.P. Beauv. (tickle grass) grown in contaminated soil tolerated elevated contaminant concentrations better than those growing in native soil. This relationship is dependent on the genetic variability of the species.

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II. TREE RESPONSE TO SOIL AMENDMENTS IN PHOSPHOGYPSUM STACK RECLAMATION

1. INTRODUCTION

Phosphogypsum (PG) is a waste by-product from production of phosphorus fertilizer via the wet process (Tayibi et al. 2009), where sulfuric acid and water are mixed with phosphate rock. PG is commonly pumped as a slurry from the production facility and placed in large holding ponds where it settles to the bottom; excess liquid is skimmed off to be treated and recycled (Rutherford et al. 1994). Over time PG accumulates and ponds become large stacks. For every ton of phosphate fertilizer produced an average of five tons of PG is produced, with a global estimate of 280 million tons produced per year (Tayibi et al. 2009, Yang et al. 2009).

PG is mostly comprised of gypsum and phosphoric acid with low concentrations of fluoride, trace elements and naturally occurring radioactivity from radionuclides in the parent ore (Tayibi et al. 2009). PG stacks can pose a risk of contaminating the surrounding environment and ground water through wind and water erosion. Thus, PG stack reclamation is focused on reducing erosion to prevent impact to the surrounding environment. Vegetation and soil create a barrier to reduce water infiltration into the stack and mitigate emissions from the stack to the atmosphere (Patel et al. 2002). Typically, PG stacks are covered with grass, although there is potential for PG to be used more beneficially to sequester carbon dioxide from the atmosphere and provide ecosystem services.

Short rotational forestry systems utilize rapidly growing woody plant species to grow large quantities of biomass on marginal land (Laureysens et al. 2004). Typically *Populus* (poplar) and *Salix* (willow) species are used in these plantations since they can produce a large amount of biomass in a short time and can tolerate marginal growing conditions. Biomass grown from these plantations can be used as fossil fuel substitutes and as carbon credits from carbon sequestration. Both markets provide an opportunity to convert land into short rotational forest plantations capable of generating capital to offset production costs.

Reclamation often requires large volumes of topsoil to reach end land use objectives. Nutrien is currently required to cap its PG stacks with 1 m of suitable topsoil. When soil is not stored during the initial disturbance, it can be brought from off site or built. Hauling soil can be very expensive for large sites with long haul distances. Thus soil amendment or building is an important alternative. Many industries produce waste materials that are high in organic matter and can contain plant nutrients. Many jurisdictions are committing to reduce the volume of waste deposited in landfills by composting the organic fraction of municipal waste. When applied to soil, municipal waste compost can improve physical and chemical properties and enhance microbial activity (Shiralipour et al. 1992). Animal manure contains plant macro and micro nutrients that increase plant growth when applied to soil (Moore et al. 1995). Humic acids, humates, humate based products, bio stimulants and natural organic fertilizers are common names for products such as Black Earth. Humates are rich in organic matter and are often marketed as a natural supplement for plant growth and productivity.

Other soil building or amending materials include mycorrhizal fungi. Mycorrhizal fungi form symbiotic relationships with roots of host plants to increase nutrient uptake efficiency, provide access to unavailable minerals, increase productivity and stress resistance (Bonfante and Anca 2009). Mycorrhizal hyphae are very fine branch like cells that grow in the soil and access nutrients that would be unavailable to the root. Host plants provide the fungi with carbohydrates necessary to complete their life cycles. The relationship between host plant and fungi is often species specific; however, some hosts can be colonized by different fungal species and some fungi can colonize multiple host species (Linderman and Call 1977).

2. RESEARCH OBJECTIVES

The objective of this experiment is to determine effects of soil amendments on above and below ground biomass, height, vigour, stump diameter, number of branches and survival of *Picea glauca* Voss. (white spruce) and *Populus balsamifera* L. (balsam poplar), and on soil properties and competing vegetation.

3. MATERIALS AND METHODS

3.1. Research Site

Nutrien Nitrogen Operations (53.734471, -113.195037) is located off Highway 15 on the east perimeter of the city of Fort Saskatchewan, which is located 20 km north east of Edmonton Alberta. The research site is located on the south bank of the North Saskatchewan River. Mean annual precipitation is 459 mm, with 353 mm as rain and 104 mm as snow (Government of Canada 2016). During June, July and August the area can receive over 60 mm of precipitation. Mean annual temperature is 2.4 °C; 17.1 °C in July is the highest mean daily temperature and -

10.4 °C in December is the lowest. The site is located in the Central Parkland Subregion of the Parkland Natural Region (Natural Regions Committee 2006). In the Fort Saskatchewan area there are large aspen parkland forests with grassland areas intermixed. Very little native vegetation remains due to extensive cultivation and anthropogenic disturbances.

This experiment was conducted on PG stack 4 at the Nutrien Fort Saskatchewan, Alberta site (Figure 2.1). Stack 4 contains PG produced in the 1970s and was re-contoured in fall 2015 for this experiment (Figure 2.1). Approximately 20 cm of topsoil, procured from an industrial development north of the site, was placed on the entire stack in fall 2015. PG stack 4 was seeded in fall 2015 with a reclamation grass seed mix to prevent soil erosion in spring.

3.2. Experimental Design And Treatments

The experimental design was a randomized complete block with 5 amendment treatments and a control (Figure 2.3). The five amendments were compost, compost with mycorrhizal fungi, manure pellets, Black Earth and mycorrhizal fungi. Blocks were replicates and each consisted of 6 plots, one for each amendment treatment, with treatments randomly assigned to plots. Each plot contained 10 trees; 5 *Populus balsamifera* and 5 *Picea glauca*. Each tree species was planted in rows running north to south in the plots; *Populus balsamifera* on the west side, *Picea glauca* on the east. Trees were spaced 1.25 m apart in a grid. Each plot was 2.5 m wide and 6.25 m long (Figure 2.4). There were 10 replicates on the site with 2.5 m unplanted buffers between each. A slight hill ran east to west so replicates were established going down the hill to account for potential effects of slopes. There was a total of 600 trees from 2 tree species x 6 amendment treatments x 5 trees per species x 10 replicates.

3.3. Amendments

Amendments were selected for their nutrient amending properties, availability and relatively low cost. They needed to be easy to apply in small or large amounts in various scale scenarios.

Compost was provided by the City of Edmonton Waste Management Center. The organic fraction of municipal waste is separated and combined with small portions of biosolids to form compost (City of Edmonton 2016). Compost is spread into long piles and turned until mature; the compost used matured in November 2015. Laboratory analyses show trace amounts of *Escherichia coli*, fecal coliform, plastic and glass. Although these inclusions were not desirable, levels were below Compost Council of Canada guidelines (CCME 2005). Compost was stored outside in large piles at the Waste Management Center prior to transport to the site. It was applied at 20 t / ha, as recommended by the City of Edmonton (Hamilton 2016).

Manure pellets were produced by EarthRenew® in Strathmore Alberta via their patented heat processing technology (Black Earth 2013). Fresh manure is cooked above 315.6 ^{o}C to remove weed seeds, pathogens and unwanted chemical compounds. Pellets provide the same nutrient benefits as typical manure with a lighter product that contains no sand, gravel or other waste material common in manure. Pellets were applied at 20 t / ha (Leskiew 2016). The pellets were provided by Mr. Leskiw and were stored outside under a tarp cover since fall 2015.

Black Earth is a mined product from naturally occurring oxidized coal that is rich in humified organic matter and humic and fulvic acids (Black Earth 2013). Black Earth can be purchased as liquid, coarse granule and powder forms of humate. The coarse granule form was selected for this experiment due to its ease of application and supplier recommendation. The coarse granules have a minimum guaranteed organic matter content of 80 %. The coarse granules were applied at 0.67 t / ha based on supplier recommendation.

MYKE® is a mycorrhizal fungal product sold at many commercial greenhouses to increase plant root development and efficient uptake of nutrients (MYKE 2016). It contains ectomycorrhizal fungi that was expected to colonize the exterior of *Picea glauca* roots and endomycorrhizal fungi which was expected to grow in the roots of *Populus balsamifera*. Fungal spores are suspended by perlite and peat. MYKE® Tree and Shrub was used, with 125 mL applied to each tree based on product recommendation for container size and stem caliper (MYKE N.D.).

In May 2016, compost, manure pellets and Black Earth were procured and calculated amounts of compost and manure pellets were weighed in 20 L pails. Amendments were hand spread evenly across the surface of each plot, then hand raked to spread to the full extent of the plot. After amendments were spread in each replicate they were incorporated into the top 10 to 15 cm of soil using a Power Dog 209 9 hp rear tine rototiller. This depth was expected to avoid incorporating the underlying PG. The rototiller was maneuvered lengthwise on the plots and in five to six passes all amendments were incorporated. Buffers were rototilled in the same way as the plots. Amendment application and incorporation were completed on May 12 and 13 2016.

The mycorrhizal inoculant was added to individual trees during planting. Both species received 125 mL (1/2 cup) based on product manufacturer recommendations (MYKE N.D.). Half of the MYKE was sprinkled on the root ball of *Picea glauca* so that excess fell into the planting hole, and the other half was placed in layers with soil. Sprinkling MYKE on the root ball is recommended for best results; however, *Populus balsamifera* cuttings did not have a root ball so MYKE was placed in layers with soil during planting.

3.4. Soil Analyses

On June 14 2016 soil was sampled for site characterization. A sample was taken in the center of each plot at a depth increment of 0 to 10 cm. Soil from three replicates of the same treatment were combined into a clean bucket and mixed into a single composite sample. This method was repeated 3 times for each treatment, creating 18 samples for analyses. Mycorrhizal fungi treatments were sampled the same way, except during mixing 250 mL of MYKE was added since the center of the plots did not receive inoculation. Samples were submitted to Exova, a commercial laboratory, and analyzed for total carbon, total nitrogen, ammonium, nitrate, potassium, phosphorus, pH and electrical conductivity (Table 2.1).

3.5. Vegetation

3.5.1. Plant stock

Picea glauca stock from the Canadian Forest Service was procured from the greenhouse on May 25 2016. Seedlings were planted in the greenhouse in early 2016 in 2+0 container stock (2 years old and grown in the same place). Height, health, branching and root ball noticeably differed among individual trees when they were received. *Picea glauca* height was measured on June 10 2016 to document the beginning height. Despite the individual plant differences, mean height when planted was very similar in each treatment. Mean tree height was 45 (13.95 standard deviation) cm; smallest was 11 cm and tallest was 78 cm. Some trees had significant branching on the entire stem, others had a few branches at the base. Tall trees with more branching often had a tight root ball with many visible roots. Small trees with less branching had fragile root balls that fell apart in the planter’s hands. Trees were randomly placed on plots to remove bias from these differences.

The *Populus balsamifera* clone Okanese was used since it has desirable growth characteristics for the site and is commonly used in the area. *Populus balsamifera* cuttings were taken from a nursery plantation at the University of Alberta Ellerslie Research Station. Long stems were harvested from a stool bed (nursery bed of woody plants propagated by layering), bagged and stored below -5 ^{o}C until taken from storage and cut into 25 cm long cuttings. Cuttings were bundled and soaked in water for approximately 12 hours prior to planting. Cuttings used in this experiment had a mean length of 25 (0.39 standard deviation) cm, mean diameter of 1.12 (0.24) cm and mean of 6.3 (0.94) buds per cutting. When planted, all the cuttings appeared to be of the same health status with no obvious visible differences. Cuttings came in bundles of 25 and were randomly planted in the plots.

3.5.2. Tree planting

On May 25 and 26 2016 the site was marked and planted. Planting was done within two days of the trees being brought from the greenhouse or cuttings taken from storage. A tree planting spade was pushed into the ground and the planter would lean back on the shovel creating a small hole for each tree. *Populus balsamifera* cuttings were placed in each hole so that one bud was above the surface and the buds were pointing upwards. *Picea glauca* required a slightly larger hole due to the root ball on the stock. Holes were made larger by removing a small amount of soil the width of the root ball (4 cm). Seedlings were planted so the top of the root ball was covered by 2.5 cm of soil. After each tree was planted the ground around the tree was compressed by hand pressing firmly on the surface to secure each tree.

3.5.3. Plot management

Weeds and grasses in the plot areas, particularly *Kochia scoparia* L. (common kochia), *Silene latifolia* Mill. (white cockle), *Thlaspi arvense* L. (stink weed), *Elymus repens* L. (quack grass) and *Elymus trachycaulus* Link. (slender wheat grass), were problematic in the 2016 and 2017 field seasons. The grass seeded for soil erosion control in fall 2015 prior to experiment establishment had potential for competition that could reduce tree growth; however, the entire experimental area was seeded equally and hence should affect treatment response equally. Mowing and cultivation were conducted around individual trees, large weeds close to trees were hand pulled. Mowed vegetation was left on the ground surface and areas around each tree were cleared of excess litter. After vegetation had been mowed a cultivator pulled by a tractor was used to incorporate litter into the substrate, avoiding the rooting area around each tree. During the 2017 growing season cultivation was conducted more frequently, eliminating the need for mowing. Cultivation was effective in removing most grass and weed competition, except in the direct vicinity of a tree (15 cm radius) where grass could not be removed without tree damage.

3.6. Vegetation Assessment

Picea glauca and *Populus balsamifera* vigour was assessed once a month during the growing season using a 1 to 5 scale for an overall health score. The scale used 1 = < 5 % of tree appears dead, 2 = 5 to 30 % of tree appears dead, 3 = 30 to 60 % of tree appears dead or conditions of 2 with significant wilting, 4 = 60 to 99 % of tree appears dead, 5 = tree appears 100 % dead. The percent of the tree deemed dead was determined from leaves or needles showing necrosis and chlorosis. Healthy leaves and needles were green with no discolouration or wilting. Vigour was assessed on June 10, June 29, July 20 and August 23 in 2016 and on May 23, June 28, August 10 and September 17 in 2017.

Midway and at the end of the growing season, height of each tree was measured using a ruler placed at the base of the trunk to the end of the tallest branch. Height measurements of *Picea glauca* were taken on June 10 and August 23 during the 2016 growing season. *Populus balsamifera* was only measured on August 23 in 2016 since they were all the same height when planted. Tree height was measured on May 23 and September 25 during the 2017 field season. Tree height for *Picea glauca* was analyzed as change in height, as each tree was not the same height when planted. Using this method, some trees had negative growth from snapped branches, so these were changed to 0 for analysis.

On September 25 2017, final measurements of each tree were taken for base stem diameter, branching pattern and number of living branches. Base stem diameter was measured using a tree caliper at the ground surface.

In each treatment and species, 6 poor (vigour = 3 or 4) and 6 healthy (vigour = 1 or 2) individuals were selected for above ground biomass assessment at the time of final measurements. Not all treatments had enough specimens in each health category and in these cases the maximum number possible were sampled (Table 2.2). While excavating a tree, measuring rooting depth was not possible; however, any occurrence of roots penetrating the PG layer was recorded. Above ground biomass was clipped at the soil surface and bagged. Everything below this point constituted below ground biomass. Above ground biomass was dried at 80 °C for 48 hours or to constant weight. *Picea glauca* had new growth separated from old growth prior to drying due to variability at planting. New growth was defined as new needles that had emerged from buds during the 2017 growing season (Keddy 2017).

On June 12 2017, prior to plot management activities, competing vegetation (which included all vegetation except the planted trees) in each of the plots was assessed. A 0.1 m^2 quadrat was place in the center of each plot and percent canopy cover of grass, forb and bare ground were visually assessed. Any vegetation rooted within the quadrat had all above ground biomass clipped at ground level and bagged. Biomass was oven dried at 80 °C for 24 hours or to constant weight, then weighed.

3.7. Soil Assessment

Portions of PG stack 4 had significantly greater mortality than other areas. The area of this experiment showed poorer growth than other areas planted with the same species. An abrupt boundary line was observed where trees on one side were 2 to 3 m tall and healthy and on the other side approximately 10 % of trees survived and were less than 1 m tall. Soil depth across the entire stack was assessed to determine if topsoil depth and soil horizons differed. A Dutch augur was used to determine the depth to PG at each sample location. A Garmin 62s GPS was used to map sample locations on a 50 m by 50 m grid. Upon completion of the grid sampling, 30 samples were taken on the boundary of where tree mortality became significantly noticeable on the stack. Samples were procured approximately 25 m away on either side of the line to determine if soil differed in good and poor growth areas.

3.8 Statistical Analyses

All statistical analyses were completed using R version 3.4.2 (R Core team 2017). Data collected at the end of the 2017 growing season were used for most of the analysis. Descriptive statistics were calculated for all measured tree variables. Normality was assessed with the Shaprio Wilk test and residual plots observation. Data were not normally distributed and not balanced due to variable survival of trees in each treatment.

Mixed models were used to detect significance of treatment on tree height, stump diameter, number of branches, above ground biomass and vigour. Replicate was included as a random effect in the mixed models. A repeated measure mixed model was used to detect significance of treatment on vigour over time. Grass biomass and cover in each treatment were assessed using analysis of variance (ANOVA) since all assumptions of normal distribution, equal variance between treatments and independence of samples were met. The p values from pairwise comparisons were adjusted using the HSD Tukey method to reduce a chance of type one error.

Tree survival in each treatment was compared using a chi-squared test and pairwise Z-tests. The p values were adjusted using the Holm method for pairwise comparisons. Tree survival and competing grass biomass were correlated using Pearson correlation coefficient.

Soil data were analyzed using ANOVA to compare the effect of the measured soil parameters by treatments. Significant effects were followed by pairwise comparisons using HSD Tukey method to adjust p values for multiple inferences. Descriptive statistics for each soil parameter measured in each treatment were calculated.

4. RESULTS

4.1. Meteorological Data

Weather during winter 2016 to 2017 was compared to a 20 year climate normal from the Government of Canada (2016) records (Figure 2.5). There was much higher than normal precipitation in October 2016 and April 2017. Precipitation was lower during 2016 from November to February than the climate normal. Mean monthly temperature was warmer than normal and only slightly cooler in December during winter 2016.

4.2. Effect Of Amendments On *Populus Balsamifera* And *Picea Glauca*

The control treatment tended to have the best overall tree growth relative to the other treatments (Tables 2.3, 2.4, 2.5, 2.6). Many tree parameters had high standard errors due to low sample sizes from low survival percentages and high variance within treatments, reducing the ability to detect significant effects.

The only statistically significant effect of treatment on *Populus balsamifera* was vigour ($p < 0.05$) (Tables 2.3, 2.5). Trees in the control were significantly healthier than those in compost and MYKE as depicted by the lowest vigour scores (Figure 2.6). Above ground biomass was highly variable (Figure 2.7, Table 2.3) and numerically greater with compost and MYKE than other treatments. Number of branches was highly variable (Figure 2.8), with no obvious treatment trend. Mean tree height ($p > 0.05$) was numerically highest with manure and Black Earth and the control was lowest (Figure 2.9). Stump diameter was larger for trees with manure ($p = 0.084$) than with Black Earth, compost and the control (Figure 2.10). When dead trees were removed from the data there was no significant effect of treatment on mean vigour ($p = 0.64$).

Treatment effects were not statistically significant for *Picea glauca* new growth biomass, old growth biomass and total biomass, change in height or vigour ($p > 0.05$) (Table 2.4). Trees in the control tended to have numerically higher biomass than in the other treatments. New growth biomass was similar for all treatments ($p > 0.05$) (Figure 2.11). Old growth and total growth biomass were similar for all treatments except the control which had numerically highest values ($p > 0.05$). Change in height for all treatments was very low (Figure 2.12), with numerically highest values for Black Earth and the control.

Mean vigour was similar for all treatments ($p > 0.05$) with the control numerically lowest (Figure 2.13). Treatment had no significant effect on tree vigour over time for either species ($p > 0.05$)

(Figure 2.14). On the first assessment date, all treatments were similar for both species; tree health then declined significantly after winter ($p < 0.05$) for all treatments, with no significant change over the 2017 growing season. By the end of the study trees were healthiest in the control for both species (Tables 2.3, 2.4).

Cumulative mortality increased greatly over the winter for both species in all treatments (Figure 2.15, Table 2.6). During the 2017 growing season mortality did not decline further and was lowest in the control at the end of the study. Survival was very low in some treatments at the end of the study (Table 2.6, Figures 2.15, 2.16). Survival was significantly highest in the control for *Populus balsamifera* (36 %) and *Picea glauca* (58 %). *Picea glauca* had significantly higher survival than *Populus balsamifera* in all treatments ($p < 0.05$). Both species showed a similar survival trend in each treatment. Survival was numerically lowest in both species in the compost and MYKE treatment.

4.3. Effect Of Amendments On Soil Parameters

Some soil properties were affected by amendments ($p < 0.05$) (Table 2.7). In general, soil nutrients increased with the organic amendments, compost and manure, with very little change to soil pH or electrical conductivity. Black Earth and MYKE had no significant effect on any soil properties relative to the control ($p > 0.05$). Both compost treatments had significantly higher nitrate and phosphorus than Black Earth, control and MYKE ($p < 0.05$). Potassium in the manure treatment was significantly highest ($p < 0.05$). Ammonium had high variability within replicates of compost and MYKE. Carbon to nitrogen ratio was highest in MYKE treatments and significantly lower in compost and compost and MYKE ($p > 0.05$). Organic matter, inorganic carbon, organic carbon, pH and electrical conductivity were not significantly affected by amendments ($p > 0.5$).

4.4. Effect Of Competing Vegetation On *Populus Balsamifera* And *Picea Glauca*

Grass biomass and cover were not significantly affected by amendments (Table 2.8), with numerically lowest values in the control (Figure 2.17). A trend was visibly obvious between tree survival and grass competition. Survival was highest in the control, which also had lowest grass yield (Tables 2.6, 2.8). All amended treatments had increased grass yield and decreased tree survival (Figure 2.16). Grass yield was similar in MYKE, Black Earth and control treatments. All amendment treatments had numerically increased grass cover relative to the control (Table 2.8). Grass cover was 10 % lower in the control than in Black Earth, compost and MYKE.

Grass biomass was not statistically significantly correlated with tree survival ($p > 0.05$) (Figure 2.18). Nevertheless, *Populus balsamifera* ($R = -0.69$) and *Picea glauca* ($R = -0.40$) both had negative Pearson correlation coefficients, which were stronger in *Populus balsamifera* than *Picea glauca*. Thus tree survival decreased with greater grass biomass (competition). Grass responded to amendments, which affected tree growth as shown by the negative correlation.

5. DISCUSSION

Amendments likely had an unintentionally greater influence on competing vegetation than on the desired tree species, as exemplified with the equal or better tree growth in the control than in other treatments. The increased nutrients from addition of amendments, particularly nutrient rich compost and manure, may have given surrounding vegetation a competitive advantage in amended plots relative to the control. Over the two year study the grass likely reduced potential tree growth despite efforts to control vegetation. Competition not only reduced nutrients available to trees, but also water, sunlight and root space. Although this hypothesis cannot be confirmed on such limited grass data and short observation time, it is a possible explanation for the higher survival and good growth in the control.

It was not unexpected that only one significant effect was observed for both species in this experiment. Most studies on short rotational forestry compile at least 4 or 5 growing seasons of data to compare the effects of clone and site characteristics on trees. Fortier et al. (2010) found that different soil nutrients at different sites lead to significantly different growth of one hybrid poplar clone over a six year study. Although our experiment is only at one site, the treatments altered soil properties significantly and some change in growth over time would be expected. Extending this study may reveal significant effects of treatment on either tree species.

Cultivating vegetation around each tree may not be the most effective way to reduce competition. The cultivator could only get within a 15 cm radius of each tree since any closer would risk tree damage. This method is likely effective with larger stock or in older plantations, but for small cuttings and seedlings used in this experiment it is likely not close enough since the root systems are not well developed. Henkel-Johnson et al. (2016) found that cultivation was the least effective method for controlling perennial grasses in a hybrid poplar plantation in northern Alberta. Vegetation control treatments did not affect survival of trees, although herbicide treatments had a significantly higher basal area than cultivation. All treatments had substantially higher survival (92.3 to 100 %) than our study after three growing seasons.

Landhausser and Lieffers (1998) found a significant decrease in height, stem caliper growth, stem and leaf dry weight of *Populus tremuloides* Michx. (aspen) when grown in competition with *Calamagrostis canadensis* Michx. (blue joint grass) in a greenhouse experiment. Removing competition of adjacent weeds and grass more effectively should improve establishment and growth of trees in future studies on PG.

Competition around each tree was likely a key driver of poor tree survival, other factors could contribute. Although mortality of both species was significant in the 2016 to 2017 winter in all treatments temperature and precipitation did not differ considerably from a 20 year climate normal. This could mean trees were stressed by the end of the 2016 growing season and did not have stored resources or strength to tolerate a winter. Poor tree growth could be related to a shallower soil profile than in a natural system. An average of 20 cm of topsoil was placed across the stack with a relatively high sand content. High sand content in soil reduces water holding capacity and rapidly drains following precipitation events (Brady and Weil 2010). These soil conditions could result in periods of water stress when precipitation is infrequent. Under the topsoil horizon is compacted PG that is designed to prevent water infiltration into the stack. This boundary layer may have restricted the rooting depth of trees relative to a natural soil. Cultivation removes plant biomass on the surface exposing dark soil to direct sunlight, resulting in higher soil temperatures and greater evaporation of water from the soil (Brady and Weil 2010). The combination of these factors could have resulted in low water availability to trees. Organic amendments used in this study improve water holding capacity but likely were not used in great enough proportions or incorporated deep enough to affect water holding capacity.

The Canadian Forest Service conducted a study adjacent to ours on PG stack 4. It used the same species and *Populus* clone, along with 2 other *Populus* clones, and the same soil building procedure but without amendments. In general, our study had lower survival than the rest of the site (visual observations) which had an average survival of approximately 80 %. Soil assessment peripheral to this study found that soil depth was likely not a result of this difference in mortality since very little fluctuation was seen across the site (Figure 2.19). The more successful areas of the site had less topsoil than areas where survival was low. Soil quality is not known for the rest of the site but no visible differences in soil texture or structure were seen during soil assessment. Soil nutrients could be the reason for the differences but without data a conclusion can only be speculated. On the perimeter of the stack, where results are poorest (Figure 2.19), water was either percolating into the stack or running off in under ground channels. We hypothesize that slope position, wind direction and drainage of water into the PG stack may have resulted in tree performance discrepancies. This hypothesis cannot be confirmed from the data in this study. Replicating the study in different areas of the stack may reveal an effect of amendments.

Despite being marketed to improve tree growth, MYKE did not improve tree parameters of either tree species relative to the control. Root assessments conducted in a companion study on the same site found MYKE significantly reduced colonization and intensity of mycorrhizal fungi (Boldt-Burish et al. in preparation). Healthy trees had significantly higher mycorrhizal colonization and intensity than unhealthy trees in the same treatment. A decrease in colonization and intensity of fungi from MYKE could be attributed to competition between native fungi and what was added from the inoculant. Many fungal pathogens including *Fusarium* sp., a known tree pathogen, were found on both species roots in all treatments (Boldt-Burish et al. in preparation). The percent of pathogenic fungi was significantly correlated with tree health. This suggests that poor health of trees could be the result of a fungal infection and reduced mycorrhizal abundance. It is possible the infection came from either the greenhouse or the soil cap at Nutrien. The source of infection could be confirmed by growing trees in both soils in a greenhouse setting.

Results from this study were unexpected and should not discredit the possibility of using amendments for establishing short rotational forestry plantations. The chemical and physical attributes of PG likely did not impact the trees during this short study; however, the soil profile built could have limited tree success. Altering depths and horizons in the soil profile may improve success of trees in future studies. Adjusting the proportions of different soil building materials could be an effective way to better test the effect of amendments on trees. Altering the amount of soil and amendment in the soil and incorporating organics into the PG layer could improve rooting depth and soil water holding capacity. Using a roundup type herbicide at the start of the study to remove as much of the seed bank as possible could be a more effective way to control vegetation in the early stages of a plantation. Comparing the effectiveness of herbicide and cultivation may be useful in the first few years after plantation establishment.

6. CONCLUSIONS

After two years amendments had little effect on either *Picea glauca* or *Populus balsamifera* for the parameters measured. The only significant effect of treatment was the control having significantly healthier *Populus balsamifera* trees. In general, the healthiest and most successful trees of both species were from the control treatment. Organic amendments, compost and manure, had significantly higher nitrate and phosphorus than most treatments. Black Earth and MYKE did not affect any soil properties significantly relative to control. Despite efforts to control unwanted vegetation, the use of amendments inadvertently benefited competing vegetation more than the planted trees.

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Table 2.1. Analytical methods for soil properties.

Soil Property	Method	Reference
Available nitrate, ammonium	Extractable (2N KCl)	McKeague 1981
Electrical conductivity, pH	Saturated paste	Carter, Gregorich 1983
Available phosphorous, potassium	Modified kelowna	Ashworth, Mrazek 1995
Total inorganic and organic carbon, nitrogen	Leco combustion	Sparks et al. 1996

Table 2.2.Number of trees for above ground biomass assessment and roots growing in PG.

Species	Treatment	Number of Trees Sampled	Number of Trees with Roots Growing in PG
Populus balsamifera	Black Earth	9	4
	Manure	3	1
	Compost	7	5
	Compost and MYKE	4	0
	MYKE	4	2
	Control	10	4
Picea glauca	Black Earth	11	0
	Manure	10	0
	Compost	8	0
	Compost and MYKE	8	0
	MYKE	8	0
	Control	12	0

Table. 2.3. Descriptive statistics of measured tree parameters for *Populus balsamifera.*

Parameter	Treatment	N	Mean	Sig	Standard Deviation	Minimum	Maximum
Height (cm)	Black Earth	13	95.9		47.2	17	205
	Manure	9	155.0		33.7	89	201
	Compost	7	113.3		43.4	37	152
	Compost and MYKE	5	142.4		66.3	65	230
	MYKE	6	139.2		36.4	75	175
	Control	17	102.9		61.7	21	236
Stump Diameter (mm)	Black Earth	13	12.2		7.3	4	30
	Manure	9	25.1		8.1	8	38
	Compost	7	16.9		10.2	4	35
	Compost and MYKE	5	20.6		14.6	7	45
	MYKE	6	20.8		7.4	10	31
	Control	17	16.7		11.5	4	48
Vigour (category)	Black Earth	50	4.2	ab	1.4	1	5
	Manure	50	4.4	ab	1.3	1	5
	Compost	50	4.6	ab	1.1	1	5
	Compost and MYKE	50	4.7	a	1.1	1	5
	MYKE	50	4.6	ab	1.1	1	5
	Control	50	4.0	b	1.5	1	5
Branches (number)	Black Earth	13	3.6		4.6	1	13
	Manure	9	7.4		6.7	1	19
	Compost	7	6.1		6.9	1	17
	Compost and MYKE	5	4.2		6.6	1	16
	MYKE	6	8.8		7.1	2	21
	Control	17	5.9		7.1	1	26
Above Ground Biomass (g)	Black Earth	9	64.3		97.8	0.54	310.2
	Manure	3	119.7		101.1	23.6	225.2
	Compost	7	116.2		113.6	2.65	355.2
	Compost and MYKE	4	342.8		468.2	43.3	1034.0
	MYKE	4	151.8		107.1	18.91	260.1
	Control	10	176.8		335.4	13.7	1109.2

Sig = means with the same letter within property rows are not statistically significant.

Table 2.4. Descriptive statistics of measured tree parameters for *Picea glauca*.

	Treatment	N	Mean	Standard Deviation	Minimum	Maximum
Height Change (cm)	Black Earth	27	2.8	2.4	0	7
	Manure	19	2.2	2.1	0	5
	Compost	24	1.7	2.2	0	8
	Compost and MYKE	19	1.8	2.0	0	6
	MYKE	18	1.7	1.8	0	5
	Control	29	2.7	2.6	0	12
Vigour (category)	Black Earth	50	4.0	1.1	2	5
	Manure	50	4.4	1.0	2	5
	Compost	50	4.2	1.0	1	5
	Compost and MYKE	50	4.4	0.9	2	5
	MYKE	50	4.3	1.1	1	5
	Control	50	3.9	1.2	1	5
Total Biomass (g)	Black Earth	11	17.7	15.5	1.8	44.0
	Manure	10	17.3	9.6	4.3	36.2
	Compost	8	15.8	6.3	6.1	24.3
	Compost and MYKE	8	15.3	10.6	7.5	36.9
	MYKE	8	20.6	15.2	2.6	45.9
	Control	12	26.2	24.1	2.3	82.2
New Biomass (g)	Black Earth	11	4.8	4.2	0.5	12.8
	Manure	10	4.6	2.3	1.3	8.2
	Compost	8	4.6	2.9	1.3	9.4
	Compost and MYKE	8	4.0	2.4	1.3	8.4
	MYKE	8	6.9	6.4	0.8	20.1
	Control	12	8.5	6.7	0.7	20.8
Old Biomass (g)	Black Earth	11	12.9	3.6	1.3	31.1
	Manure	10	12.7	3.8	3.1	28.0
	Compost	8	11.2	4.5	4.9	14.9
	Compost and MYKE	8	11.3	4.3	6.2	28.5
	MYKE	8	13.7	3.4	1.9	25.8
	Control	12	17.8	3.7	1.7	61.4

No significant effects were found for any of the measured tree parameters.

Table 2.5. Statistical significance values of mixed models for tree parameters.

Tree Parameter	Species	P Value
Height Change (cm)	*Picea glauca*	0.27
Vigour (category)	*Picea glauca*	0.13
Total Biomass (g)	*Picea glauca*	0.62
New Biomass (g)	*Picea glauca*	0.25
Old Biomass (g)	*Picea glauca*	0.75
Height (cm)	*Populus balsamifera*	0.053
Stump Diameter (mm)	*Populus balsamifera*	0.084
Vigour (category)	*Populus balsamifera*	0.015
Branches (number)	*Populus balsamifera*	0.54
Above Ground Biomass (g)	*Populus balsamifera*	0.58

Table 2.6. Cumulative percent mortality of *Populus balsamifera* and *Picea glauca*.

Species	Treatment	July 2016	August 2016	May 2017	June 2017	August 2017	September 2017
Populus balsamifera	Black Earth	18	30	82	72	74	74
	Manure	18	22	80	72	76	82
	Compost	32	62	88	78	82	86
	Compost and MYKE	30	48	84	84	90	90
	MYKE	26	48	80	86	88	88
	Control	16	32	70	58	68	66
Picea glauca	Black Earth	4	8	34	32	38	46
	Manure	6	8	44	44	48	62
	Compost	2	4	32	36	48	52
	Compost and MYKE	8	12	46	40	48	62
	MYKE	10	10	38	40	58	64
	Control	2	4	30	22	38	42

Table 2.7. Mean soil properties for each soil treatment.

Soil Parameter	Black Earth	Manure	Compost	Compost and MYKE	MYKE	Control	P Value
Nitrate (ug / g)	3.3 (1.5) b	12 (4.4) ab	19.7 (5.0) a	18 (2.6) a	3.3 (0.6) b	3.3 (2.1) b	0.0002
Phosphorus (ug / g)	40.7 (6.0) bc	53.3 (6.1) ab	61.3 (1.2) a	64 (7.8) a	35.7 (2.5) c	36 (6.1) c	0.0003
Potassium (ug / g)	88.3 (6.5) b	216 (67.9) a	128.3 (5.5) b	128 (16.5) b	87 (3.0) b	85 (1.0) b	0.0017
Ammonium (ug / g)	1 (0.9)	0.4 (0.0)	1.2 (0.6)	2.9 (2.3)	0.8 (0.6)	0.7 (0.5)	0.1736
C:N Ratio	12.3 (0.1) ab	12 (0.2) abc	11.7 (0.0) c	12 (0.1) bc	12.5 (0.1) a	12.3 (0.2) ab	0.0014
Total Nitrogen (%)	0.2 (0.0)	0.2 (0.0)	0.2 (0.0)	0.2 (0.0)	0.2 (0.0)	0.2 (0.0)	0.2000
Organic Matter (%)	4 (0.4)	4.1 (0.4)	4.4 (0.2)	4.4 (0.3)	4.1 (0.3)	4 (0.7)	0.6289
Inorganic Carbon (%)	0.1 (0.0)	0.1 (0.0)	0.1 (0.0)	1 (0.0)	0.1 (0.0)	0.1 (0.0)	0.8511
Organic Carbon (%)	2 (0.2)	2 (0.2)	2.2 (0.1)	2.2 (0.2)	2 (0.1)	2 (0.4)	0.6284
PH	7.4 (0.1)	7.5 (0.1)	7.4 (0.1)	7.4 (0.1)	7.4 (0.1)	7.5 (0.2)	0.5984
Electrical Conductivity (ds / m)	2.4 (0.1)	2.2 (0.7)	2.3 (0.7)	2.4 (0.4)	2.3 (0.1)	1.8 (1.0)	0.7990

Numbers are means followed by standard deviations indicated in brackets.

Means with the same letter in each row are not statistically significant. Rows without letters show no significant effect of treatment.

Table 2.8. Descriptive statistics for grass in each treatment.

	Treatment	N	Mean	Standard Deviation	Minimum	Maximum
Above Ground Biomass (kg / ha)	Black Earth	10	6099.2	633.4	4762	6962
	Manure	10	6326.4	2525.8	3746	12797
	Compost	10	6701.3	1073.0	5295	8579
	Compost and MYKE	10	6639.2	1611.6	4359	9506
	MYKE	10	5893.7	955.4	3949	7039
	Control	10	5664.3	1374.5	3063	7454
Percent Grass Cover (%)	Black Earth	10	69.0	5.7	60	75
	Manure	10	64.5	10.1	50	80
	Compost	10	69.0	11.0	45	80
	Compost and MYKE	10	62.5	12.5	50	85
	MYKE	10	69.5	8.6	60	80
	Control	10	59.0	15.6	35	75

Figure 2.1. Location of study site in Alberta; indicated with a black and white star.

Figure 2.2. Location of amendment plots at the Nutrien Fort Saskatchewan site, indicated with a black and white star.

←**North** Top of Hill

Replicate						
1	Black Earth	MYKE	Control	Compost	Manure	Compost + MYKE
2	Compot + MYKE	Manure	Black Earth	Compost	Control	MYKE
3	Black Earth	Compost + MYKE	Compost	Manure	MYKE	Control
4	Compost + MYKE	Compost	Control	Manure	MYKE	Black Earth
5	Control	MYKE	Manure	Compost + MYKE	Compost	Black Earth
6	Compost	Control	Compost + MYKE	Black Earth	Manure	MYKE
7	Compost	Manure	MYKE	Control	Black Earth	Compost + MYKE
8	MYKE	Manure	Black Earth	Compost	Compost + MYKE	Control
9	Black Earth	Compost + MYKE	Compost	Manure	Control	MYKE
10	Compost	Black Earth	Compost + MYKE	MYKE	Control	Manure

Bottom of Hill

Figure 2.3. Treatment and replicate layout for amendment experiment. Empty space between plots are unplanted buffers.

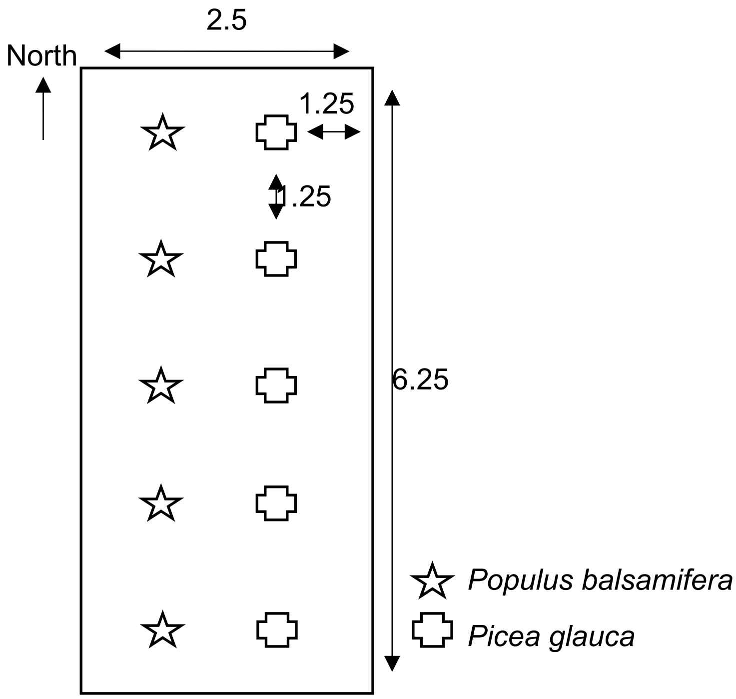


Figure 2.4. Amendment experiment tree spacing and location within each plot (units in meters).

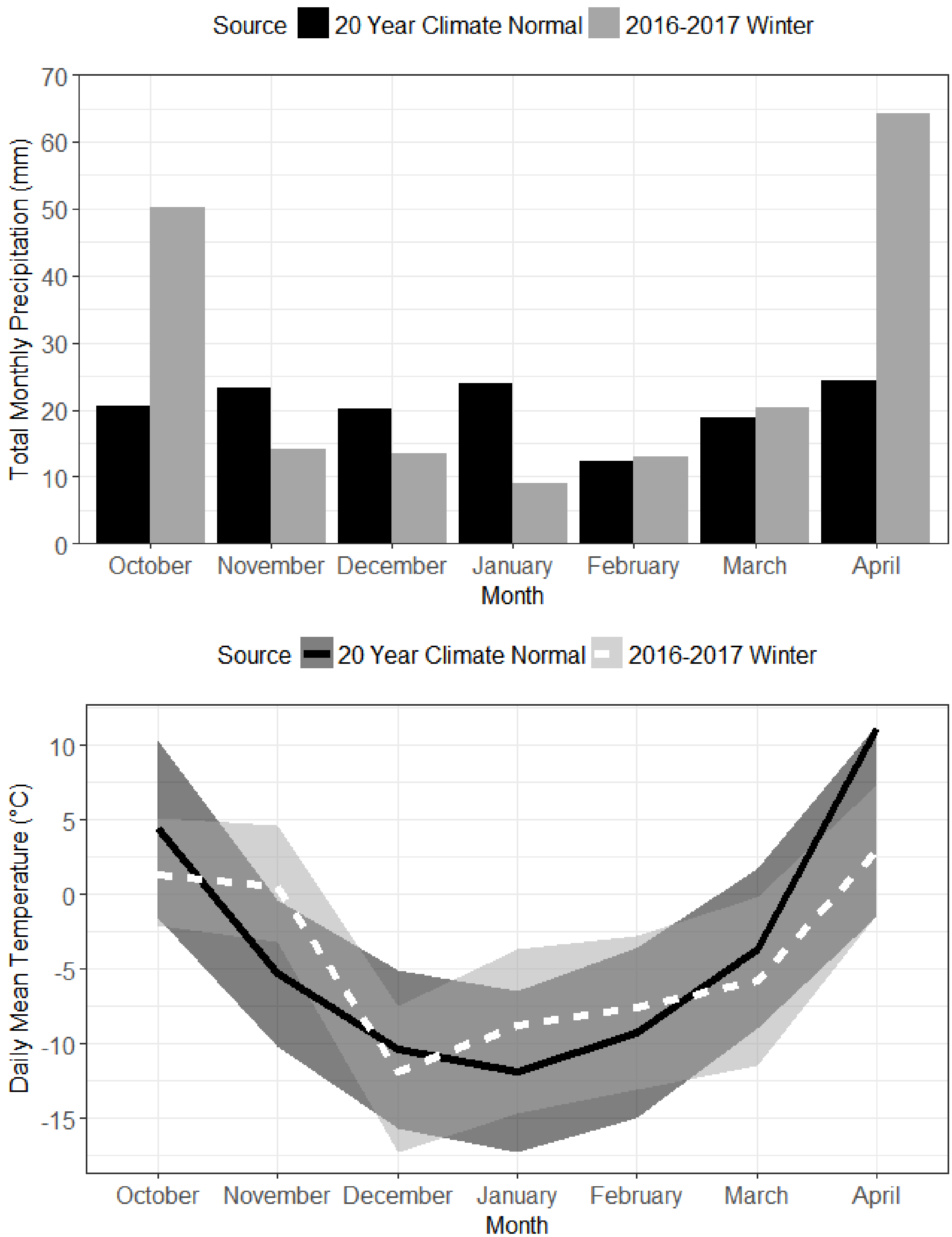


Figure 2.5. Mean monthly precipitation and temperature in 2016 to 2017 winter relative to a 20 year climate normal. Minimum and maximum temperatures are displayed using ribbons. (Adapted from Government of Canada 2016, ACIS 2018)

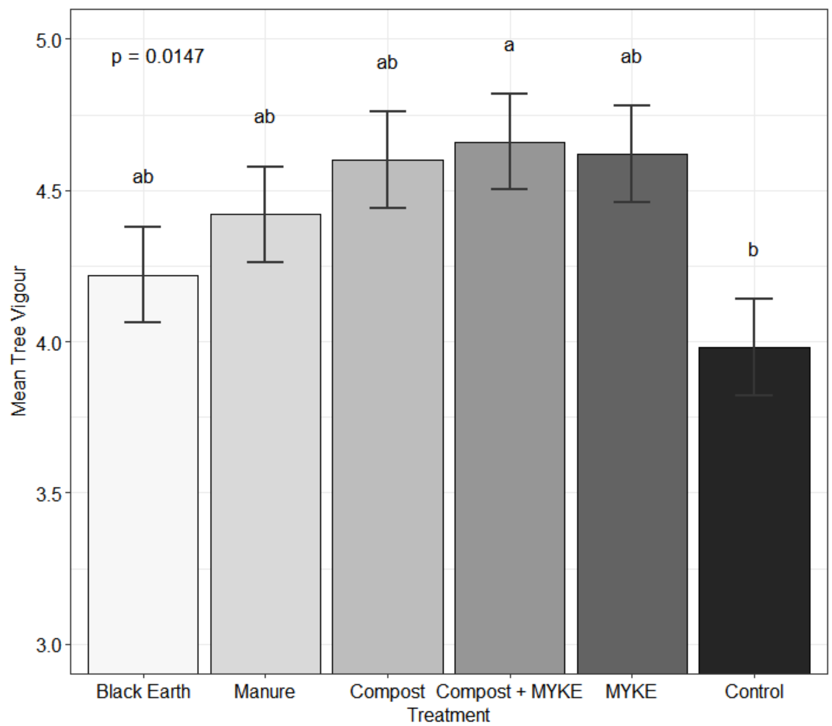


Figure 2.6. Mean *Populus balsamifera* vigour at the end of the study. Standard error is indicated with error bars. Different letters denote statistical significance.

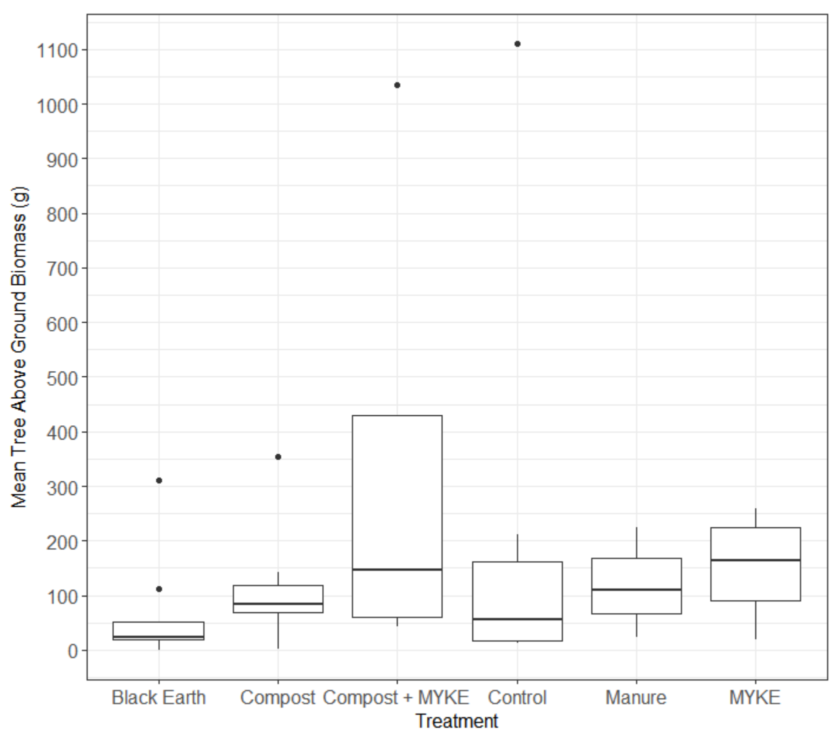


Figure 2.7. Boxplot distribution of *Populus balsamifera* above ground biomass.

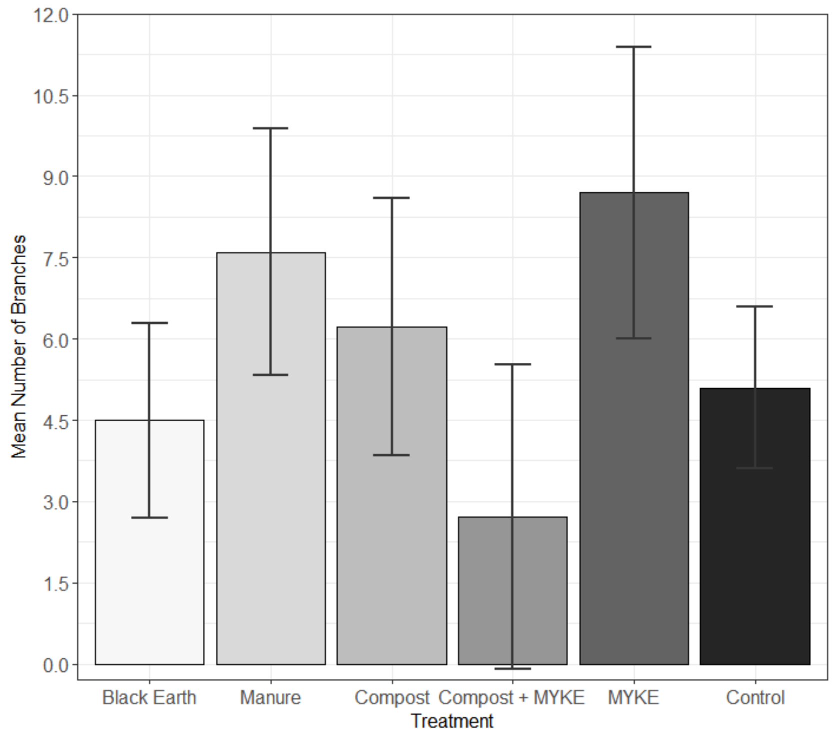


Figure 2.8. Mean *Populus balsamifera* branch number at the end of the study. Standard error is indicated with error bars. No significant effects were found among treatments.

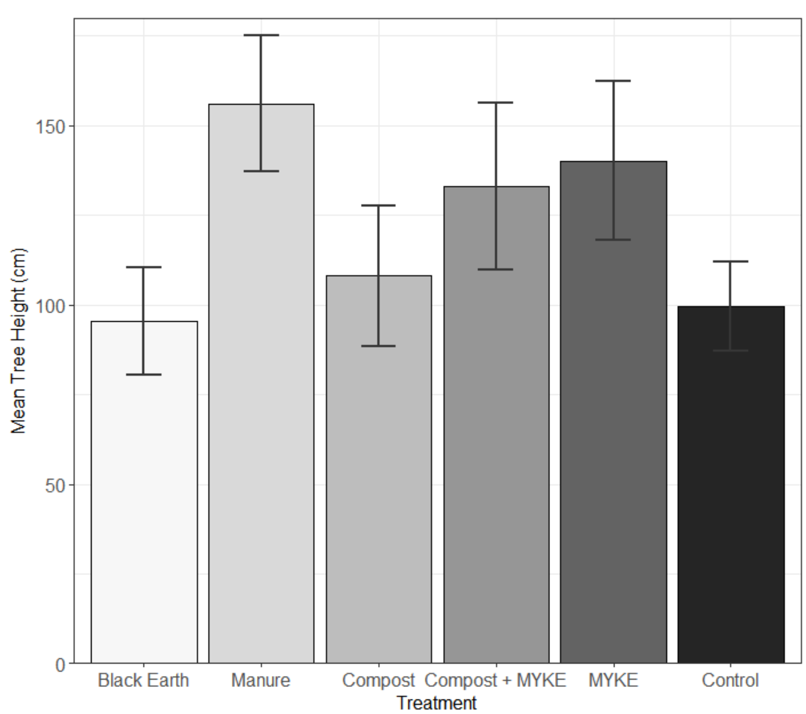


Figure 2.9. Mean *Populus balsamifera* height at the end of the study. Standard error is indicated with error bars. No significant effects were found among treatments.

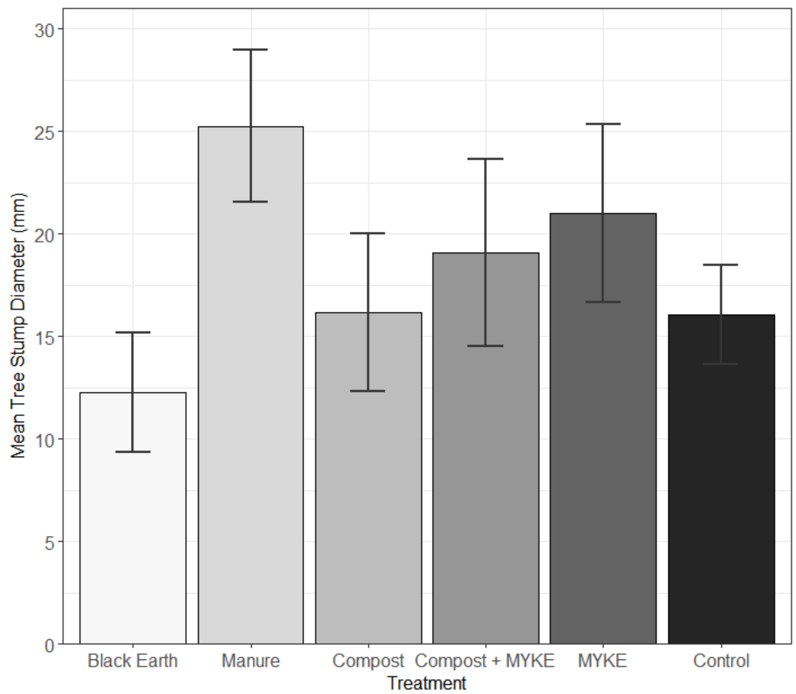


Figure 2.10. Mean *Populus balsamifera* stump diameter at the end of the study. Standard error is indicated with error bars. No significant effects were found among treatments.

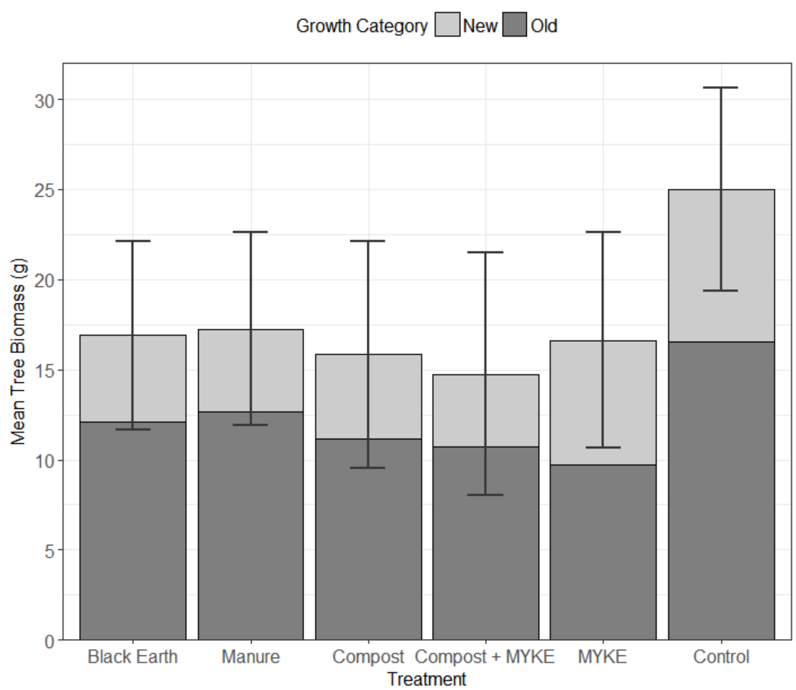


Figure 2.11. Mean *Picea glauca* above ground biomass. Total biomass is the entire bar; new and old growth are differentiated with shade. Standard error of the total biomass is indicated with error bars. No significant effects were found among treatments.

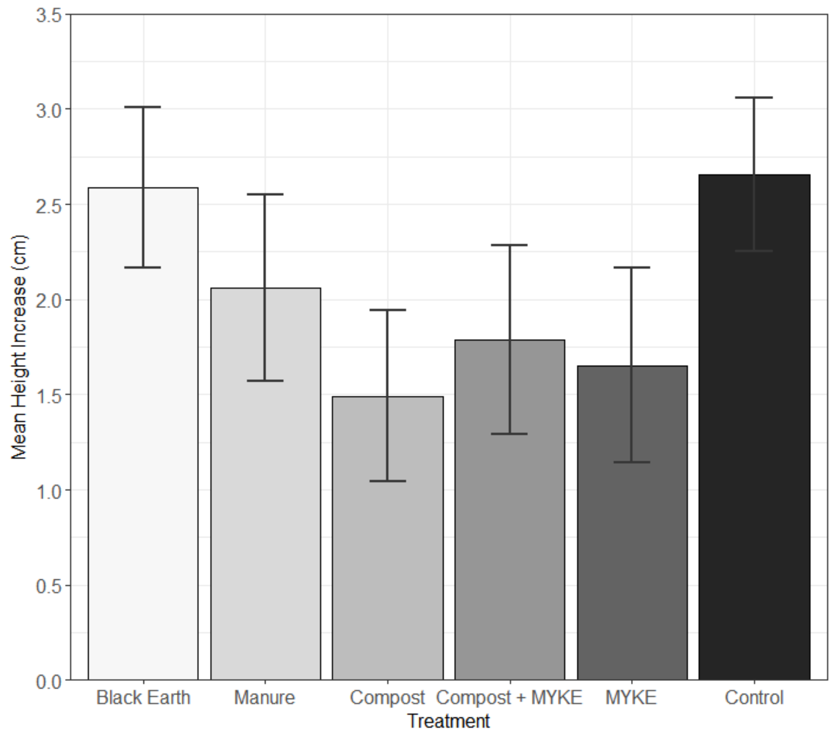


Figure 2.12. Mean *Picea glauca* height increase since planting. Standard error of is indicated with error bars. No significant effects were found among treatments.

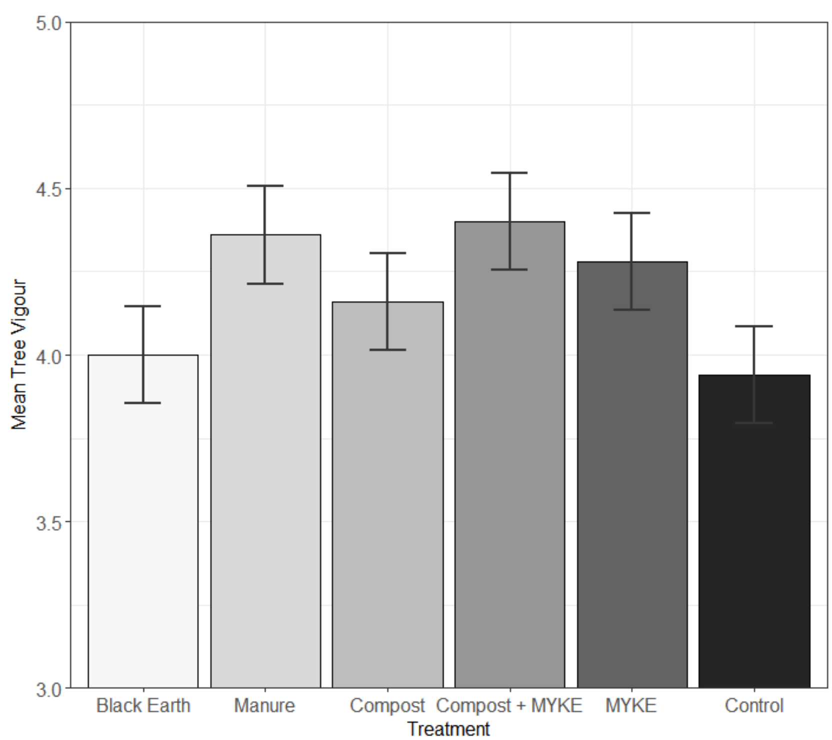


Figure 2.13. Mean *Picea glauca* vigour at the end of the study. Standard error of is indicated with error bars. No significant effects were found among treatments.

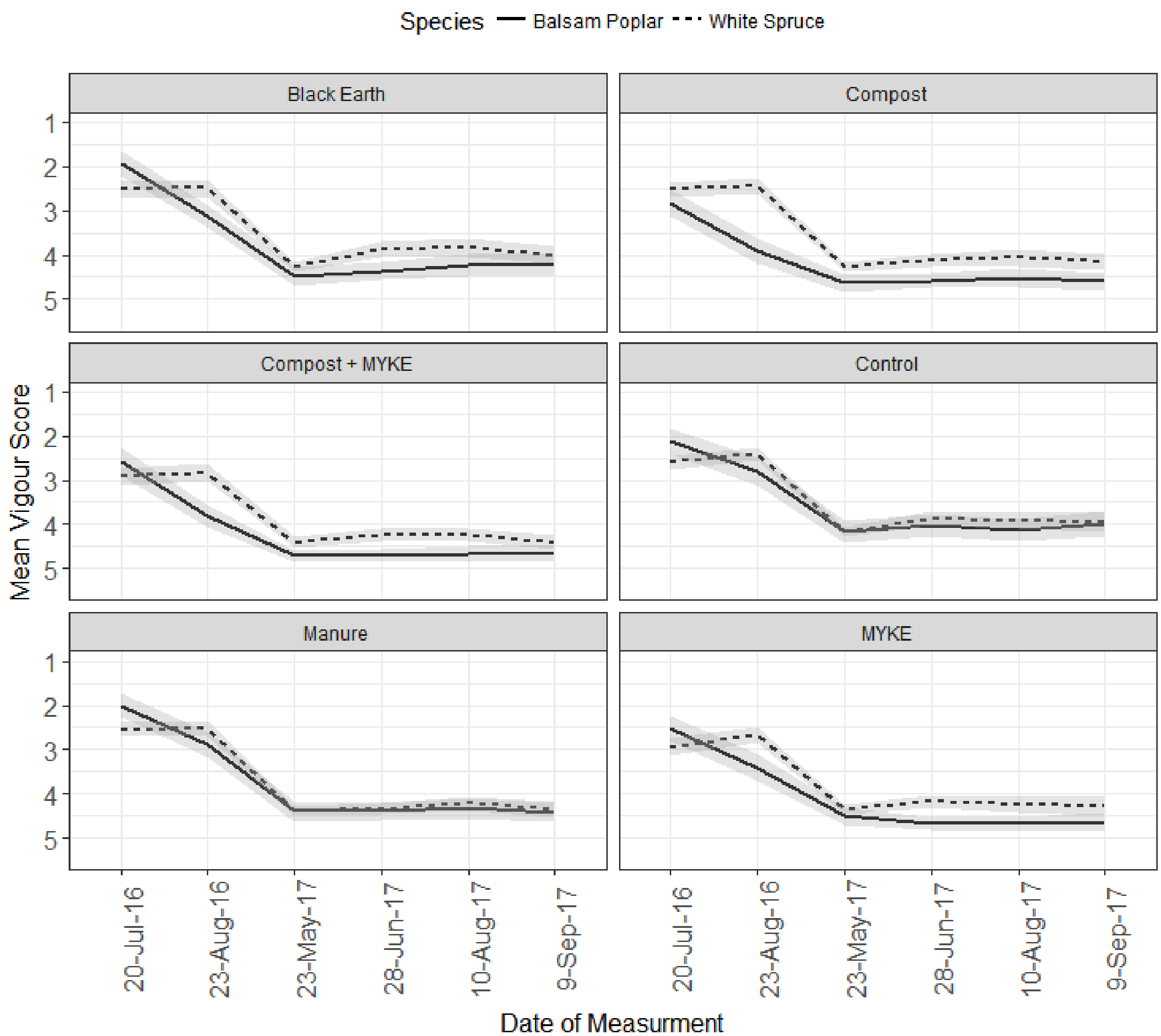


Figure 2.14. Change in mean tree vigour of *Populus balsamifera* and *Picea glauca* over the study. Ribbons above and below the lines indicate standard error. No significant effects were found among treatments.

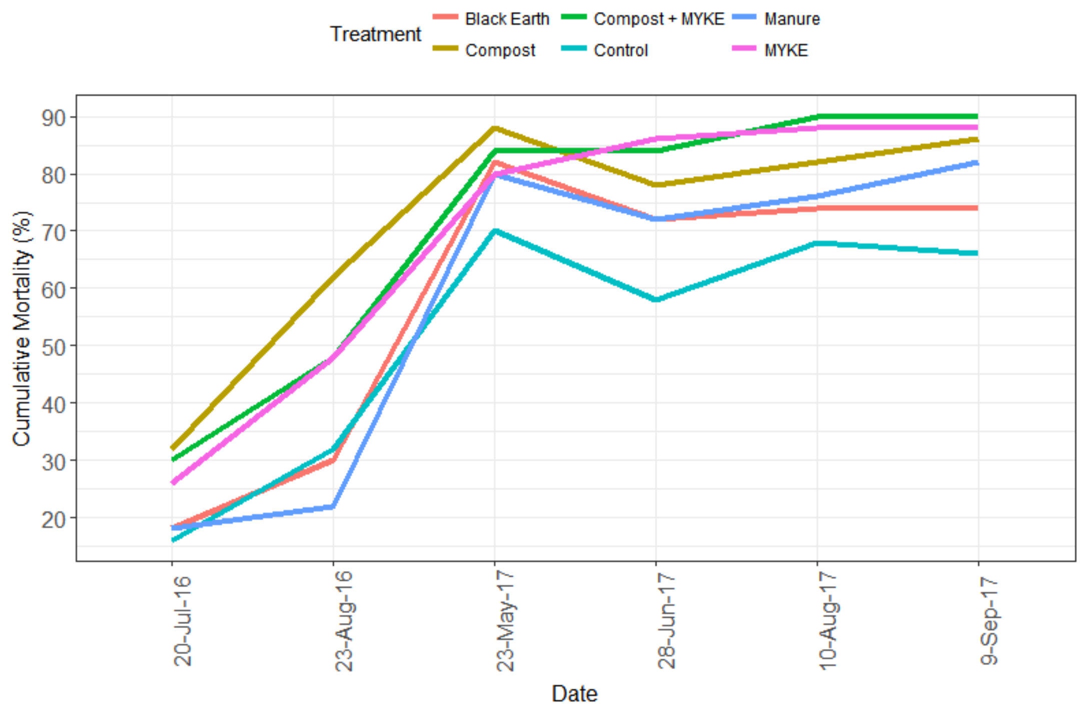


Figure 2.15. Cumulative mortality of *Populus balsamifera* over the study.

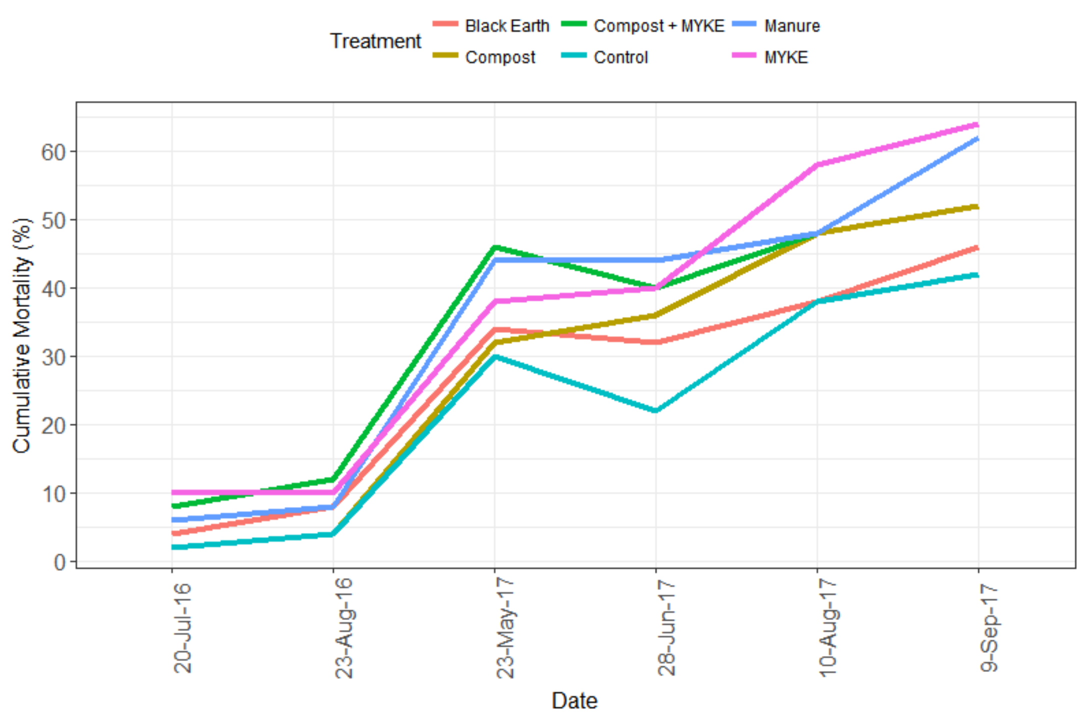


Figure 2.16. Cumulative mortality of *Picea glauca* over the study.

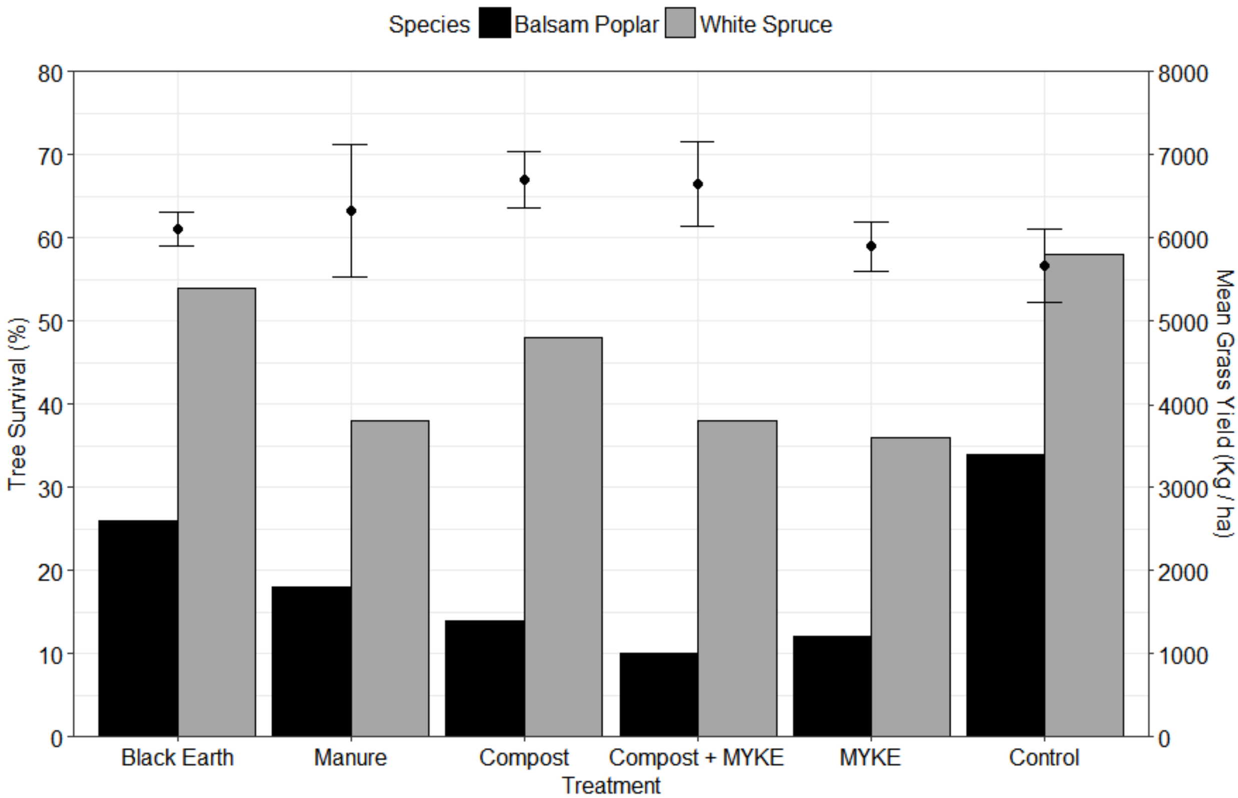


Figure 2.17. Percent tree survival of *Populus balsamifera* and *Picea glauca* in each treatment indicated with bars. Mean grass yield in each treatment shown with dots. Standard error is indicated with error bars for mean grass yield.

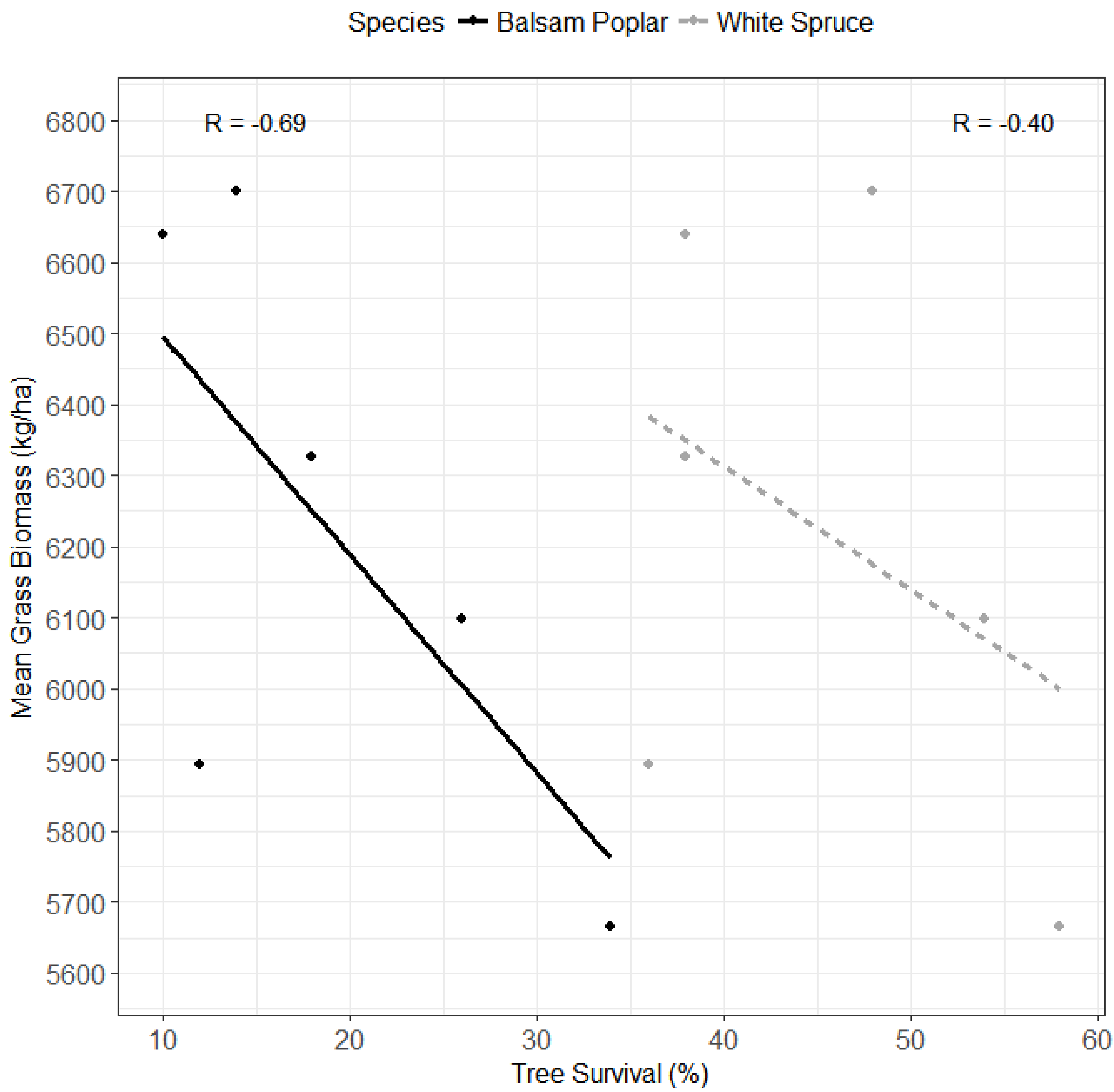
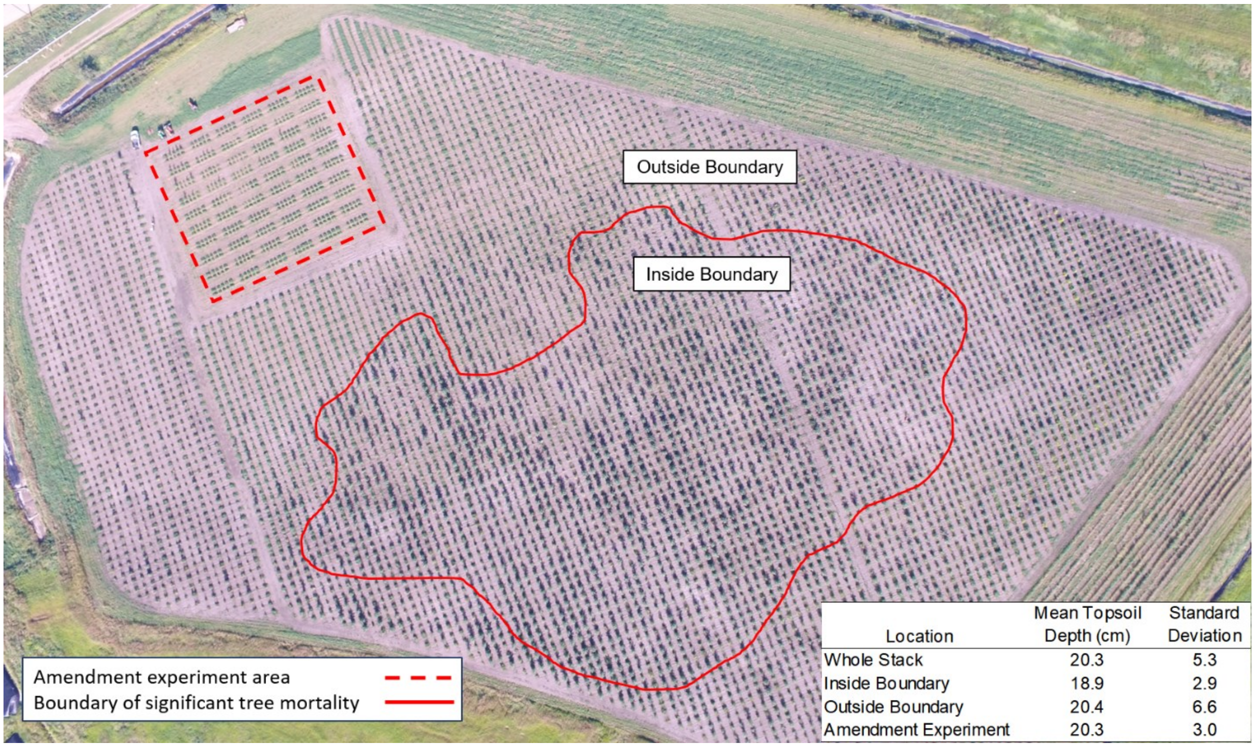


Figure 2.18. Tree survival correlated with mean grass biomass for both species. Pearson correlation coefficients for each species is written above the respective line.



Location	Mean Topsoil Depth (cm)	Standard Deviation
Whole Stack	20.3	5.3
Inside Boundary	18.9	2.9
Outside Boundary	20.4	6.6
Amendment Experiment	20.3	3.0

Figure 2.19. Soil depth assessment of PG stack 4. Boundary line derived from GPS data gathered in 2017. The whole stack is the field of view of this photo.

III. PHOSPHOGYPSUM EFFECTS ON SOIL PHYSICAL AND CHEMICAL PROPERTIES AND VEGETATION TISSUE CONTENTS

1. INTRODUCTION

Phosphogypsum (PG) is a waste by-product from production of phosphorus fertilizer via the wet process (Tayibi et al. 2009). Phosphate fertilizer production involves mixing sulfuric acid and water with phosphate rock represented by the reaction $Ca_5F(PO_4)_3 + 5H_2SO_4 + 10H_2O \rightarrow 3H_3PO_4 + 5CaSO_4 \cdot 2H_2O + HF$. PG is commonly pumped as a slurry from the production facility and placed in large holding ponds (Rutherford et al. 1994). PG settles to the bottom of ponds and excess liquid is skimmed off to be treated and recycled. Over time PG accumulates and ponds become large piles of PG called stacks. For every ton of phosphate fertilizer produced an average of five tons of PG is produced, with a global estimate of 280 million tons produced per year (Tayibi et al. 2009, Yang et al. 2009).

PG is comprised mainly of calcium sulphate dihydrate or gypsum (> 90 %); however, it contains impurities such as aluminum, iron, magnesium and sodium oxides, fluoride, organic matter and naturally occurring radionuclides (Rutherford et al. 1994). The rock source used to make fertilizer affects concentrations of impurities in PG. Newly generated PG contains residual acidity from phosphoric, sulfuric and hydrofluoric acids and can have a pH of 2.1 to 5.5 (Rutherford et al. 1994, Tayibi et al. 2009). Over time pH in the stack increases as treatment water within the stack is flushed out and treated for reuse in production. After pore water in the stack has been flushed multiple times, pH in the stack approaches neutral and contaminant concentrations decrease.

Radionuclides in the parent ore used to make phosphate fertilizers are chemically concentrated in PG during fertilizer production. Radium is concentrated in PG through substitution with calcium in gypsum during fertilizer production (IAEA 2013). High volume, low activity substances are compared to naturally occurring radioactive material (NORM) criteria to evaluate risk (Health Canada 2011). A material is considered to be NORM contaminated if it has radium-226 activity greater than 0.3 Bq / g. Most PG is NORM, but not NORM contaminated since its radioactive properties are inherent in its chemistry (Rutherford et al. 1995, Richardson 1997, IAEA 2013). Radium-226 and its decay products are the primary radionuclides of concern since they are retained in PG (Figure 3.1) (IAEA 2013). Radium-226 has a half life of 1620 years and will be present long after stack closure. Inhalation of radon-222 gas, a daughter product of radium-226, and uptake of radium-226 by plants are common risks surrounding PG (Rutherford et al. 1996). Radium is an alkaline earth metal and tends to follow calcium in biological pathways and typically accumulates in bones if ingested (Rutherford et al. 1994).

PG stacks pose a risk of contaminating the surrounding environment and ground water through wind and water erosion. Thus, PG stack reclamation is focused on reducing erosion to prevent potential impact to the surrounding environment and limit water infiltration. Typical stack reclamation involves placing a soil cap and seeding grass to create a barrier to reduce water infiltration into the stack and mitigate emission by the stack to the atmosphere (Patel et al. 2002). This method has been effective at limiting infiltration and typically stack runoff is within guidelines (Hallin et al. 2010). Limited research is available on how trees grow on PG stacks.

Trace elements in PG can be taken up by plants and passed through the food chain as organisms consume those plants (Pulford and Watson 2003). Plant uptake of impurities in PG has been reported in many studies; however, results from these studies can be highly variable (Rutherford et al. 1994, Komnitsas et al. 1999, Hallin 2009, Petrisor et al. 2001). Plant uptake of specific elements depends on plant species, duration of exposure, availability in the soil to plants and chemical composition of the PG. Previous studies focused on grass tissue analysis since using trees in reclamation was uncommon.

A potential concern of vegetating PG stacks is vegetation accumulating trace elements to toxic levels and negatively impacting animals that forage on it. This impact is very difficult to evaluate since impact of specific metals on individual faunal species is poorly understood and depends mainly on the diet of the animals (National Resource Council 2005). The term maximum tolerable levels is used in the National Research Council's (2005) summary of mineral tolerances of animals as safe concentrations for different elements in food at which no negative impact can be seen to an organism. Maximum tolerable levels are defined as chronic ingestion of food over a minimum of 10 days. Although this document is not inclusive for all elements or species, it provides a reference value that can be used for comparison.

2. RESEARCH OBJECTIVES

The objective of this experiment was to determine whether PG stacks provide suitable growing conditions for short rotational forestry plantations. Specific objectives were as follows.

- To determine whether mixing PG with soil affects specific soil properties.
- To determine whether PG affects metal concentration in washed and unwashed plant tissue

in specific grass and tree species.

- To determine whether metal and radionuclide concentrations differ in specific grass and tree species.
- To determine whether vegetation cover and specific soil treatments affect radionuclides in the surrounding environment.

3. MATERIALS AND METHODS

3.1. Site Description

Nutrien Nitrogen Operations (53.734471, -113.195037) is located on the east perimeter of the city of Fort Saskatchewan, which is located 20 km north east of Edmonton Alberta. The research site is located near the south bank of the North Saskatchewan River and surrounded by petroleum refineries and industrial chemical facilities.

Mean annual precipitation is 459 mm, with 353 mm as rain and 104 mm as snow (Government of Canada 2016). During June, July and August the area can receive over 60 mm of precipitation. Mean annual temperature is 2.4 °C; 17.1 °C in July is the highest mean daily temperature and -10.4 °C in December is the lowest. The site is in the Central Parkland Subregion of the Parkland Natural Region (Natural Regions Committee 2006). In the Fort Saskatchewan area there are aspen parkland forests with grassland areas intermixed; little native vegetation remains due to extensive cultivation and anthropogenic disturbances. The surrounding soils of the region are dominated by Eluviated or Orthic Black Chenozems with small amounts of Gleyed Black Chernozems and Brunisols around the North Saskatchewan River (Agriculture and Agri-Food Canada 2006). Soil texture is mostly loam and slit loam with some sandy clay and loamy sand. Glaciolacustrine is the dominant parent material.

3.2. Experimental Design And Treatments

In 2013 a water holding pond at Nutrien Fort Saskatchewan was emptied and decommissioned to establish a reclamation experiment. PG from an adjacent stack was used to fill the pond and a 15 cm topsoil cap was placed on the PG. In spring 2014 three tillage treatments were implemented to study their effect on grasses. Treatments were heavy disking to 30 cm, deep ripping to 45 cm and deep ripping to 45 cm with addition of manure pellets. Deep ripping treatments were disked after ripping to reduce surface roughness. Treatments were established in a randomized complete block design (Figure 3.2). The site was divided into 4 replicated blocks each 48 m by 82 m. In each block the three treatments and control were placed in 12 m wide strips running east to west. After seedbed preparation each treatment was halved and seeded with one of two seed mixes, an industry standard reclamation mix, or one suggested by Dr. M Anne Naeth (Table 3.1).

Soil auguring in 2016 showed only the ripping treatment incorporated PG with soil. Heavy disking and capping treatments had a clear boundary layer between soil and PG, with little to no mixing. Mr. Dick Purveen (2016), who assisted in plot establishment, said disks bounced and bucked across the site, achieving nearly no incorporation. Therefore, heavy disking and ripping with manure treatments were not assessed. The two treatments assessed were soil ripping (ripped soil) and soil capping (soil cap).

In July 2014 after grass was seeded, 2 of the 4 blocks had a depression form due to settling and consolidation of PG. Grass research on these two blocks was discontinued and the area was filled with PG and capped with 15 cm of soil. This filled in area was used to establish new plots for short rotational forestry research. On April 30 2015, before planting woody vegetation, a 300 hp International 9170 4WD tractor pulled heavy duty disks across the site in 2 passes with the second pass perpendicular to the first. Following disking the site was harrowed to reduce surface roughness. Soil was assessed collectively (tree soil) across the entire plantation area since no substrate treatment was made.

Salix viminalis L. (basket willow) and *Salix dasyclados* Wimm. (india willow) cuttings were planted on June 4 2015 using a mechanical 3 point hitch Model 1000 transplanter. Two *Populus balsamifera* L. (balsam poplar) clones, NM-6 and FFC-1, were hand planted as one year old rooted stock on June 4 2015. A planting spade was used to make a hole for the tree and soil was firmed around each tree after planting. Stock were prepared a day prior to planting by soaking in water overnight. Trees were planted in rows 60 cm apart with 2 m wide row spacing. Each species was planted in 7 rows with each row consisting of approximately 130 trees, for a total of 910 of each species (Figure 3.2) and 5 m buffers between each species. A mortality assessment on July 7 2015 showed that many plants died. On July 24 2015 200 seedlings of each *Populus* clone and 300 *Salix* cuttings were planted to replace the dead stock.

Weeds and grasses between tree rows, particularly *Kochia scoparia* L. (common kochia), *Silene latifolia* Mill. (white cockle), *Thlaspi arvense* L. (stink weed), *Elymus repens* L. (quack grass) and *Elymus trachycaulus* Link. (slender wheat grass) were a significant problem since tree planting and throughout the experiment. Weeds between rows and buffers were mechanically removed using a 1.5 m Sovema rotary tiller approximately every 2 weeks in each of the growing seasons. Noxious weeds within and between each tree row were hand pulled immediately if seen at any growth stage. Non-noxious weeds were hand pulled around trees if they were flowering or appeared to be inhibiting tree growth due to their large size and / or their high plant density.

The University of Alberta Ellerslie research station was used as the non-industrial control site for comparison with Nutrien. The Canadian Forest Service has identical clones at Ellerslie that were used at Nutrien. The plantation spacing is the same as described for the Nutrien site except there were variable numbers of rows of each species. Ellerslie has grass areas of similar grass species near the tree plantation that were used for comparing grass treatments at Nutrien.

3.3. Soil Nutrients

Soil on the water holding pond was sampled with three replicates taken from each of ripped soil, soil cap, tree soil and pure PG locations at Nutrien. Samples were evenly distributed across the water holding pond to determine variability on the site. Soil was sampled from the upper 20 cm or until the PG horizon was reached. Pure PG was sampled by removing the topsoil and taking a core directly into the PG. Cation exchange capacity was not determined for PG since the high calcium content of PG will result in competition on exchange sites. All other methods for CEC determination offered by the laboratory were not suitable for PG.

Samples were divided into halves after sampling since both dry and fresh materials were required for different analyses. Samples were dried at the University of Alberta at 50 ^{o}C for 48 hours since commercial laboratories dry soil above 100 ^{o}C. These temperatures can evaporate water within the PG molecule resulting in a mass overestimation since PG is a hydrated molecule (Strydoma and Potgieterb 1999).

Soil samples were sent to ALS labs for analyses according to the standard methods of Carter and Gregorich (2008), unless otherwise noted. Particle size (sand, silt, clay) was determined physically using sieves. Total carbon and nitrogen were determined by combustion; total inorganic carbon as $CaCO_3$ equivalent; total organic carbon and organic nitrogen (APHA 4500-N) by calculation; cation exchange capacity by ammonium saturation; electrical conductivity and pH by saturated paste; chloride, nitrate and sulfate by ion chromatography (EPA 300.1); available phosphorus with modified Kelowna solution (Ashworth and Mrazek 1995); ammonium by extraction and colorimetry by auto analysis; sodium adsorption ratio by calculation; and calcium, magnesium, sodium and potassium by inductively coupled plasma (EPA 6010B).

3.4. Soil Water

On May 25 2017, three HOBO micro stations (Model H21-002) were set up on the water holding pond in each of the grass soil cap, grass ripped, *Salix* and *Populus* locations. At each station two ECH2O EC-5 soil water sensors were installed, one 10 cm below the surface and the second at the barrier between the soil and PG. Sensors were set to log every hour with a one minute sampling interval.

Soil field capacity and wilting point were determined using pressure plates according to standard procedure (Eijkelkamp 2009). A small volume of soil was placed in 1 cm tall and 3 cm diameter rings. Samples were tamped into the rings, then distilled water was added to the plate by pouring around the rings to prevent soil disturbance. Over 48 hours the samples became saturated. Pressure was set to 33 kPa and 1500 kPa for field capacity and wilting point, respectively. After 24 hours samples were weighed, oven dried at 50 ^{o}C for 48 hours, then reweighed to determine water content.

3.5. Trace Elements In Soil

Soil in the water holding pond and at Ellerslie was sampled on September 12 2017. Three random replicates were taken from each of the areas of ripped soil, soil cap, tree soil and pure PG locations at Nutrien. Three samples were randomly taken from areas within the forest plantation at Ellerslie. Vegetation management in the tree area led to mixing of the soil and PG in some areas of the site and therefore soil samples of the tree soil were selected so there would be variability in mixing. Low, medium and high mixed samples, based on colour, were submitted to determine the range of element concentrations. Soil was sampled from the upper 20 cm or until the PG layer was reached. Pure PG was sampled by removing the topsoil and taking a core into the PG.

Samples were analyzed by inductively coupled plasma mass spectroscopy (ICP-MS) for total dry weight of metals at ALS labs (USEPA 1994). Metals analyzed were aluminum, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, chromium, cobalt, copper, iron, lead, lithium, magnesium, manganese, molybdenum, nickel, phosphorus, potassium, selenium, silver, sodium, strontium, sulfur, thallium, tin, titanium, tungsten, uranium, vanadium, zinc and zirconium. Samples were dried, homogenized and dissolved with nitric and hydrochloric acid prior to analysis. Soluble fluoride in soil was extracted using deionized water and the extractant analyzed using ion chromatography (EPA 300.1).

3.6. Isotopes In Soil

Soil was sampled from Nutrien on September 13 2017 for high resolution gamma spectroscopy analysis. Three randomly located replicates were analyzed for each of ripped soil, soil cap, high and low mixed soil with PG in the tree area and pure PG. The upper 20 cm of soil was sampled except for pure PG, which was obtained by clearing topsoil and auguring to pure PG.

Samples were oven dried at 50 ^{o}C for 48 hours. A mortar and pestle were used to break large aggregates, then soil was passed through a 2 mm sieve to remove rocks. Samples were weighed into 50 mL polypropylene counting vials and heat sealed using a flame heated spatula. Canisters sat for 3 weeks to reach a secular equilibrium state of radium-226 and its progeny.

Samples were individually counted using a 40 % relative efficiency ORTEC hyperpure FX-Series PROFILE Ge detector (FWHM of 1.81 keV for the 1332.5 keV full energy peak of cobalt-60) housed in a 15 cm lead cave lined with copper. This detector type is advantageous, particularly for NORM analysis when lead-210 is of interest, given the high detector counting efficiency at low energy due to the thin detector dead layer and high transmission carbon end cap. Detection limits were calculated using Currie's (1968) method.

Radium-226, radium-228, thorium-228, lead-210 and potassium-40 were quantified using gamma spectroscopy. Radium-226 was quantified using 295 and 352 keV γ (gamma)-emissions of lead-214, together with the 609 and 1764 keV γ-emissions of bismuth-214. Radium-228 was quantified using 338 and 911 keV γ-emissions of actinium-228; thorium-228 was determined using 238, 583 and 2614 keV γ-emissions of lead-212 and titanium-208, respectively. Lead-210 was quantified directly via its 46.5 keV γ-emission; while potassium-40 was measured using its 1460 keV γ-ray. Soil, PG and mixed soil and PG samples were counted for 12,500 – 80,000 seconds. Several background counts up to 500,000 seconds were collected and used to give corrected net sample activities. Canada Centre for Mineral and Energy Technology and New Brunswick Laboratory, U.S. Department of Energy uranium and thorium standards certified for uranium, thorium, radium-226, radium-228, thorium-228 and lead-210 were used for calibration.

3.7. Trace Elements In Vegetation Tissue

Vegetation was sampled at Nutrien and Ellerslie on September 12 2017. Woody samples were taken from the FFC1 and NM6 *Populus* clones and both *Salix* species. Grass species of the *Poaceae* family were sampled at both sites.

Each woody sample was a composite of new growth of 8 to 10 adjacent trees from the same randomly selected row. New growth was the stem, leaves and buds of the last 15 cm of new branches (Keddy 2017). Each species had 3 replicates, one each from the north, middle and south part of the block. Samples were taken from the ground to a height of 1.2 m, the highest animals are expected to browse (Keddy 2017). Samples were bagged and homogenized in the laboratory to create a composite to be divided for each analysis.

On September 13 2017, three grass replicates were collected from ripped and capped treatments and at Ellerslie. A sample was taken from the east, middle and west areas of each treatment at Nutrien from industry and Naeth seed mixes. Grass areas adjacent to the plantation were sampled at Ellerslie. The entire above ground biomass was clipped at ground level and bagged. Only grass that was green in colour was sampled since current year growth was desired for analysis. All grass species sampled were from the *Poaceae* family.

A small sample of tree leaves and grass tissue from Nutrien was taken in 2016 to be analyzed for total element concentrations. A single replicate of each *Salix* species, *Populus* clone and grass were collected. Samples were randomly taken throughout the rows of trees and blocks of grass until enough sample was collected.

Samples from 2017 were divided in half and either washed or left unwashed to compare browse quality with internal concentrations from plant uptake. Samples were washed with LIQUINOX solution in deionized water. LIQUINOX is a concentrated solution containing 200 ml C10-C18 alkylbenaene sulfonate, 50 ml alcohol ethoxylate, 50 ml coconut eiethanolamide, 70 ml sodium xylene sulfonate, 50 ml EDTA and 500 ml water. Solution was diluted by adding 100 mL of deionized water for every one mL of LIQUINOX. Vegetation was rinsed by hand for 30 seconds in a bath containing LIQUINOX then rinsed in four deionized water baths until no bubbles were observed. Water was changed after each sample. All vegetation was air dried, bagged and sent to ALS and Maxxam laboratories for analysis.

Vegetation samples were analyzed by ICP-MS for total dry weight of metals (USEPA 1994). Metals analyzed for were aluminum, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, chromium, cobalt, copper, iron, lead, lithium, magnesium, manganese, molybdenum, nickel, phosphorus, potassium, selenium, silver, sodium, strontium, sulfur, thallium, tin, titanium, tungsten, uranium, vanadium, zinc and zirconium. Samples were dried, homogenized and dissolved with nitric and hydrochloric acid prior to analysis using standard laboratory procedures. Fluoride in vegetation was analyzed using ion selective electrode analysis by Maxxam Analytics (Meyeerhoff and Opdycke 1986, Knight et al. 1988).

3.8. Isotopes In Vegetation

Vegetation for isotope analysis was sampled separately from that for trace metal and fluoride since a different sampling plan was used based on analysis costs and research objectives. Composites of poplar and composites of willow were collected on September 14 2017. Due to vegetation management, certain areas on the holding pond had high mixing of soil and PG. These areas were delineated as high activity based on surface colour and gamma meter readings. Due to budget constraints, three replicates each of poplar and willow were collected in highly mixed areas since they theoretically represent the most contaminated vegetation. Three replicates of grass above ground biomass were sampled in ripped and capped treatments. A single sample of willow leaves from Ellerslie was analyzed for comparison. Samples were not washed since the objective was to assess browse, not uptake. In 2016 one replicate of each woody species and two grass species from the ripped treatment from the water holding pond were sent to ACT laboratory for radium-226 activity determination.

Samples were oven dried at 80 °C for 24 hours or to constant weight and ground into fine powder using a Wiley mill. Approximately 30 g of ground sample was packed into 50 mL vials, heat sealed and left for approximately three weeks to reach secular equilibrium. Two samples from the soil with highest activity were ashed after milling to reduce sample volume by a factor of 10, resulting in higher concentrated samples for lower detection limits. Ashing can volatilize radon gas so samples were heat sealed and left for four weeks to reach secular equilibrium. Ceramic crucibles were sterilized in a muffle furnace at 500 ^{o}C for one hour. Samples were ashed for one hour at 200 ^{o}C then left over night at 500 ^{o}C. Crucibles were weighed before and after ashing to calculate total ash content. Samples cooled in a desiccator until they reached room temperature. Ashed vegetation was packed into 10 mL vials and left to reach secular equilibrium. Vegetation was analyzed as per section 3.6. Count times were increased to 300,000 seconds to increase certainty in measurement due to low activity in samples.

3.9. Radon Canisters

Radtrak radon canisters were used during the 2017 growing season using to determine radon gas emission under different vegetation covers. Radon gas was measured since it is a daughter product of radium-226 and is an environmental hazard of PG. Canisters measure radon activity by quantifying the amount of etching caused by alpha particles from radon decay on the plastic film inside the canister (Landauer 2005). Canisters were in the field for 112 days from May 23, 2017 to September 11 2017. Canisters were fastened to a wooden stake 56 cm above the surface with a protective plastic cover over top to prevent rain damage according to product recommendations (Figure 3). Three canisters were placed in each of the ripped and capped soil treatments in the grass area, within the rows of FFC1, NM6, *Viminalis* and India species, on an unvegetated PG stack at Nutrien and at Ellerslie for a total of 24 canisters. Canisters were removed from the field and immediately sent to Landauer laboratory for analysis.

3.10. Statistical Analyses

All statistical analyses were completed using R 3.4.2. (R Core Team 2017). When analysis of variance (ANOVA) tests were used assumptions of ANOVA were confirmed before each test. Assumptions of normal distribution, equal variance between treatments and independence of samples were assessed using residual plots and boxplots. P values from each ANOVA were adjusted using the Holm method to reduce the chance of type 1 error due to experimental alpha level inflation. If a P value was < 0.05 after Holm adjustment, then pairwise comparisons were made. P values from pairwise comparisons were adjusted using the Tukey HSD method. An alpha level of 0.05 was used to determine statistical significance.

Each soil parameter was subjected to ANOVA to determine significant differences between soils. Statistical analyses could not be completed on HOBO data since many sensors had readings that did not reflect realistic field conditions. Visual assessments between treatments were conducted for June 18 to July 31 2017 since a complete data set was available for the sensors selected and precipitation varied during this time. Soil water content at each probe was compared to precipitation to determine effects of intensity and duration on fluxes in the soil. Soil water content at field capacity and permanent wilting point were compared using an ANOVA. If significance differences existed, then pairwise comparisons were made between soils. P values from pairwise comparisons were adjusted using the Tukey HSD method.

Raw data for soil trace elements were plotted as box plots for initial comparison. Each element was visually assessed and those with no or little differences were removed from statistical analysis. Visual differences in the data were observable when greater than a factor of 3. Each element was analyzed using ANOVA to test significant differences among soils. Radium-226, radium-228, thorium-228, lead-210 and potassium-40 were compared for soil. ANOVA was used to determine significant differences between soil types. Significant results were followed by pairwise comparison with p values adjusted using the Tukey HSD method. Pearson's correlation coefficient was used to correlate radium-226 activity with potassium-40.

The entire data set for the tissue analysis was visualized using boxplots of each element within each treatment level. Elements with similar median values and distributions between treatments were removed since no effect of any treatment was visible. Where possible, treatments were combined to make the data easier to synthesize, present and highlight interesting effects. Each element was analyzed individually using multi-way ANOVAs comparing the effect of location and vegetation type on tissue concentrations. Significant effects were followed by pairwise comparison to determine significant effects of treatment within each element being analyzed. Means and standard errors calculated from ANOVAs are used in figures. Samples below detection had values entered as half of the detection limit to allow for statistical use of the data.

Single vegetation samples taken in summer 2016 were compared to the mean and standard deviation of the more robust 2017 sampling. Since no replication was conducted for the 2016 sampling, no statistical comparison can be made. Due to long analysis time for isotopes in vegetation, not enough samples were analyzed for statistical analysis. A correlation of radium-226 in soil and vegetation was calculated. Canisters below detection had data entered as half the detection limit for statistical analysis. An ANOVA test was used to detect significance effects of radon emission from vegetation cover. Significant results were followed by pairwise comparison with p values adjusted using the Tukey HSD method.

4. RESULTS

4.1. Soil Nutrients

Soil type significantly affected cation exchange capacity, organic and total carbon, sulfate, calcium, base saturation and organic nitrogen (Table 3.2). Calcium and sulfate were significantly highest in PG, with greatest differences between capped soil and pure PG. PG had significantly higher calcium and sulfate than capped soil, which had significantly lower concentrations than any other soil. PG had significantly lower organic carbon, total carbon, organic nitrogen and saturation percentage than capped soil and highest nitrate with a very high standard deviation.

Soil data were compared to Alberta Soil Quality Criteria Relative to Disturbance and Reclamation for the plains region to assess their suitability for reclamation (Table 3.3) (Soil Classification Working Group 1987). Most soils were categorized as good or fair topsoil or subsoil relative to criteria. Ratings were good for all soils for pH and sodium adsorption ratio. Salinity, saturation, texture and inorganic carbon were good or fair for all soils. All soils except PG were good for organic carbon; PG was poor.

4.2. Soil Water

Both *Populus balsamifera* clones had similar trends and therefore NM-6 data were only presented for discussion. Visual trends in soil water content were apparent among vegetation covers and soil treatments (Figure 3.4). With high precipitation intensity, soil water content increased at both measured depths. A daily cyclical pattern at 10 cm depth of high and low water content was observed when precipitation was infrequent. Daily fluctuation of water content at the soil PG barrier did not fluctuate as much as at 10 cm depth. With small precipitation events there were only noticeable changes in soil water at 10 cm depth; PG did not change. With large precipitation events (> 10 mm / day) changes at 10 cm and in PG were seen. Grass treatments appeared to have a more rapid decline in soil water content following intense rain events relative to woody species. All probes at the PG barrier followed the same trend but settled on slightly different values over time (Figure 3.5). *Salix* had highest water content relative to other covers. Both grass treatments had similar values, with greater variability between woody species.

Highest water contents for all treatments were following the June 20 precipitation event (Figure 3.4), when water content at 10 cm depth approached or exceeded field capacity. From July 2 to 16 2017 precipitation was limited, causing a decline in water content at 10 cm. However, this decline was not as apparent for any of the PG sensors. During this prolonged period without precipitation, 10 cm soil depth water content approached or fell below permanent wilting point. Field capacity and wilting point were significantly different in each of the soils (Table 3.2). Capped soil had significantly higher values for field capacity and wilting point than other soils and PG had significantly lower values.

4.3. Trace Elements In Soil

Tin and tungsten were below detection limits and most elements had similar values in different soils (Table 3.4). Aluminum, beryllium, cadmium, calcium, chromium, copper, iron, magnesium, manganese, nickel, sodium, strontium and vanadium had greatest differences among treatments; fluoride did not show large differences.

All metals were below Canadian Council of Ministers of the Environment (CCME 2004) soil guidelines and Alberta Tier 1 Soil and Groundwater Remediation Guidelines for industrial land use (Tables 3.4, 3.5). Although the Nutrien site is considered an industrial area, element concentrations in the soil were below the stricter agricultural values. Fluoride was below CCME criteria but since water soluble fluoride was analyzed not total, this criterion cannot be used.

All 14 elements analyzed differed significantly with soils (Table 3.5). Greatest differences were between pure PG and soils (Figure 3.6). Cadmium, calcium, fluoride, sodium and strontium were highest in PG and aluminum, beryllium, chromium, copper, iron, magnesium, manganese, nickel and vanadium were lowest. Elements high in soil were generally low in PG and vice versa. Ripped soil and soil PG mix had values between capped soil and pure PG. Ellerslie soil and Nutrien capped soil had no significant differences, nor did ripped soil and soil PG mix.

4.4. Isotopes In Soil

Radium-226 activity in PG was above NORM guidelines. Samples with high PG had radium-226 activity higher than 300 Bq/g and were NORM contaminated (Table 3.6). Pure PG had significantly higher radium-226 and lead-210 than other soils. PG had significantly lower radium-228, thorium-228 and potassium-40 than other soils. Measured isotopes were not significantly different in low mixing and ripped soil. Statistical uncertainty of each isotope was good for all soils. Radium-226 and potassium-40 were significantly correlated, ($R = -0.99$) (Figure 3.7).

4.5. Trace Elements In Vegetation

Antimony, arsenic, beryllium, bismuth, thallium and zirconium were removed from statistical analysis since all samples were below detection. Aluminum, boron, cadmium, chromium, cobalt, lead, molybdenum, selenium, sodium, tin, titanium, uranium and vanadium had some samples below detection (Table 3.7).

Washing vegetation had limited effects, with inconsistency among elements (Figure 3.8). Therefore, washed and unwashed samples were combined for all elements to allow for simpler interpretation and greater statistical power. Grass data from the two soil treatments showed little effect of soil treatment (Figure 3.9). Molybdenum was the only element that showed any obvious difference in soil treatment. Grass data for each soil treatment were thus combined since substrate effect was low. The final dataset for interpretation were location = Nutrien or Ellerslie and vegetation type = grass, individual *Populus balsamifera* clones and *Sailx* species. Aluminum, barium, boron, cadmium, calcium, chromium, cobalt, fluoride, magnesium, potassium, sodium, strontium, sulfur and zinc showed treatment variability or were high in PG.

Location and vegetation type significantly affected elements in plant tissue (Figures 3.10, 3.11, 3.12, 3.13, 3.14). Differences were greater between vegetation types than locations. Grass was typically most different from *Salix* species or *Populus balsamifera* clones. Location affected some elements, but generally had little effect. *Salix* species and *Populus balsamifera* clones had few statistical differences. Results from 2017 were comparable to the smaller 2016 sampling event and were within one standard deviation of the mean for most elements (Table 3.8). Aluminum was the only element that differed over two sample years, although only leaves were sampled in 2016 and in 2017 browse was added, which includes new woody growth.

Tissue concentration at each location was significantly different for barium, cobalt, fluoride and zinc. Barium was 5 to 7 times higher at Ellerslie than Nutrien (Figure 3.10). Zinc was 3 to 4 times higher at Ellerslie than Nutrien (Figure 3.14). Cobalt and fluoride were 5 and 3 times higher at Nutrien than Ellerslie, respectively (Figure 3.12). Other elements had some significant differences with location but not in all vegetation types.

Boron, cadmium, calcium, sulfur and zinc concentration in grass were significantly lower than almost all woody species (Figures 3.10, 3.11, 3.14). Chromium was significantly higher in grass than all other vegetation types (Figure 3.11). The only significant effect of location on grass was for barium, with Ellerslie significantly higher than Nutrien (Figure 3.10).

Few effects were seen between *Salix* species at either location. Sodium was the only element with significant differences (Figure 3.13). *Viminalis* had significantly higher sodium than all other vegetation types at Ellerslie and Nutrien, and was the only vegetation type with values above detection for sodium. *Populus balsamifera* clones NM-6 and FFC-1 had few differences in either location. The only significant effect between the two clones was FFC-1 having significantly higher sulfur than NM-6 at the Nutrien site (Figure 3.14). Other elements were not significantly different although some variation was seen in mean concentration for some elements.

Nearly all elements in vegetation from Nutrien and Ellerslie were below maximum tolerable levels set by the National Resource Council (2005) (Table 3.9). All but one element had considerably lower values than recommended for rodents or cattle. Boron in some woody samples was higher than recommended for rodents. All grass samples at Nutrien were below maximum tolerable levels for both rodents and cattle.

4.6. Isotopes In Vegetation

Radium-228 and thorium-228 were below detection in all of the vegetation samples (Table 3.10). Radium-226 was highest in grass vegetation and below detection in all tissues at Ellerslie. Lead-210 was in disequilibrium with radium-226. Ashed samples had lead-210 activity above NORM guidelines (0.3 Bq / g). Radium-226 in substrate was significantly negatively correlated with radium-226 in vegetation (Figure 3.15). The samples of leaves were below detection for radium-226 (< 0.01 Bq / g).

4.7. Radon Canisters

Radon concentration differed significantly with vegetation cover. Ellerslie canisters were below detection and lower than at Nutrien (Figure 3.16). Radon was significantly lower on bare PG than all other treatments at Nutrien. Ripped soil had significantly higher radon than all other treatments except grass capped soil. All four woody species did not differ significantly. All results were below the recommended 200 Bq / m^3 level set by Health Canada (2014).

5. DISCUSSION

5.1. Trace Element Effect On Soil And Vegetation On PG

Since results for all soil samples were below CCME soil guidelines there is no concern to the surrounding environment from the PG used in this study. Although PG affected element concentrations in the soil, all were below criteria. PG in this study generally had lower concentrations of impurities relative to other studies (Rutherford et al. 1994, Hallin 2010, AbouRizk 2015). Element concentrations in PG and soil mixes will depend on PG and soil ratios. Fluoride cannot be directly compared to CCME guidelines since only water soluble fluoride was assessed in this study and total fluoride is reported in criteria.

Lack of significant differences in tissue concentrations between Ellerslie and Nutrien, even though there were differences in total metal concentrations in soils, was likely due to metals in PG being in unavailable forms for plant uptake. This is supported by element concentrations in grass from ripped and capped soils. While ripped soils had mixed soil and PG with higher metal concentrations than capped soil, there were minor differences in tissue concentration in either grass treatment. A key influence on metal bioavailability in soil is pH (Cataldo and Wildung 1978, Tyler and Olsson 2001). At pH < 5 there are more metals in soluble form available to plants. PG pH in this study was near neutral, making mobility of metals low, and resulting in low uptake by plants. Plant contents were also low since metal concentrations in soil were low.

The few significant differences for *Populus balsamifera* clones and *Salix* species in this study contradict results from other studies (Landberg and Greger 1996, Sebastiani et al. 2004, Hermle et al. 2006). Laureysens et al. (2004a) found differences in *Populus* clones uptake on a more contaminated site over a similar study time. Although tissue concentrations varied within clones there were no differences in biomass for any clones suggesting *Populus* clones could tolerate higher concentrations of contaminants. Although no toxicity symptoms were seen for any of the trees at Nutrien, different *Populus* clones may respond differently. Pulford et al. (2002) found variable uptake and survival of 20 *Salix* species on a highly contaminated site and concluded that plant ability to partition elements in different organs altered growth success. Our study only had browse sampled so partitioning of elements cannot be addressed. Metal concentrations in vegetation reported by Pulford et al. (2002) were higher than some elements in our study and lead to reduced growth. *Salix* species in our study do not have large differences in survival, other than wildlife browsing, nor show signs of toxicity. Differences in uptake between woody species may be reduced since metals are likely not in a bioavailable form.

Despite very little difference in most element concentrations in vegetarian types, sodium concentrations in *Viminalis* were noticeably different from the rest of the data set. *Viminalis* had considerably higher concentrations of sodium than all other samples and it was the only species to have any value above detection at either location. This result has not been seen in other studies. Sodium is a plant micronutrient and is rarely discussed in literature regarding contaminant uptake and therefore the reason causing this is unknown. It is possible that this is an effect of differences in uptake in *Salix* species but it cannot be confirmed.

The lack of differences between unwashed and washed samples suggests that at the time of sampling, uptake and browse were similar. Browse was expected to differ from uptake since it would include particulate matter found on the outside of the tissue, particularly PG and soil from cultivation. This does not mean that browse and uptake would not differ at other sample dates. Precipitation can wash particulate material off vegetation, resulting in little difference between washed and unwashed samples. Rain could have rinsed particulate off in this study but is unlikely since there was little precipitation for nearly 2 weeks before sampling. Sampling vegetation at different dates throughout the growing season may reveal greater differences between browse quality and uptake. When vegetation management is no longer required on the site, browse quality should be similar to plant uptake throughout the growing season.

The vegetation tissue results in this study can be used for comparison to toxic concentrations found in other studies but cannot be used to assess risk to animals due to the complexity of interactions of elements in animal diets. Length of exposure, concentration in the food source, life stage of the animal, diet, mobility of the element in the animal, metabolism of an animal and interactions with other elements can all play a role in determining an impact to animal health (National Research Council 2005). Boron in some woody samples was higher than the recommended MTL for rodents but should not be an issue as it is unlikely that a rodent would eat woody material. Boron concentrations in PG are low and it is unknown why some woody samples have elevated values. Comparing data from Nutrien to toxology reference data, shows it is unlikely there is risk to animals consuming the vegetation. Data collected at different times of the year may yield different results since vegetation partitions elements differently throughout the year (Laureysens et al. 2004b). If animals ingest substantial quantities of soil while foraging, impact to animals must be reevaluated.

5.2. Isotope Activity In Soil And Vegetation On PG

The significant effects for radon emission in vegetation cover were unexpected. Bare PG had significantly lower radon than all other treatments at Nutrien despite having the least obstruction to the canister and no soil barrier present. Canisters used in this experiment have limited interpretation since they do not differentiate high or low fluxes of radon but give a gross estimate of radon emission over the sample period. Radon flux from soil can vary based on climate conditions and cultivation between tree rows would affect emission rates.

Richardson (1997) found presence of grass on bare PG reduced radon emission by half and placement of a 15 cm overburden cap halved the emission again. Bare PG in this study could have lowest observed radon for a few reasons. An adjacent stack used for the bare PG samples and was not made from the same ore as that used in the water holding pond (Nichol 2017). PG generated from different sources will have different levels of activity (Rutherford et al. 1996). Although possible, this is likely not the reason for low radon values. More likely the crust layer that forms on PG stacks resulted in little PG dust blowing off the surface and limited gas exchange (IAEA 2013). This hypothesis could be tested by taking cores of the PG stack to a depth of 1 to 2 m and determining lead-210 activity. Lead-210 is a relatively stable end product in the radium-226 decay chain and would accumulate near the surface as radon gas decays before it can reach the atmosphere. Assuming radon can move upward through the stack, accumulation at the surface would be expected relative to deeper samples (Duke 2017).

Variability from cover type was likely not due to variability in underlying PG or vegetation absorbing radon, but to differences in canopy structure of vegetation cover. Radon is heavier than air and will diffuse from the ground and collect on the soil surface. On windy days radon may be blown away from the detector which reduces the amount detected. Grass, at maturity, creates a windbreak around each canister protecting it from wind. Thus both grass covers would have higher radon than woody treatments. Since the canopy of a tree is higher than 56 cm, wind blowing under the canopy could blow radon off site and prevent it from reaching the detector. Wind could also be the reason for bare PG having lowest values at the Nutrien site because there was no vegetation to prevent radon from being blown off site. An alternative hypothesis for grass having highest radon is that grass roots create channels for gas to preferentially escape to the surface. The ripped treatment also breaks up the boundary layer between soil and PG allowing for greater gas diffusion, which could be why it had highest radon.

If the experiment had been set up to eliminate effects of wind it would also limit any effect of vegetation cover. This experiment was set up based on canister product recommendations adapted from use in a basement to use in the field. Ultimately this experiment was established to test the effect of vegetation cover on radon emission. An alternative approach to quantify radon gas emission variability in the stack would be to make small bore holes and hang the canisters under or at the soil surface (Duke 2017). This would reduce the negative effect of wind but limit interpretation to how vegetation cover impacts emission. Although using canisters to do this would work, there are instruments that can take real time radon measurements in the field.

Risk of radon inhalation to the public cannot be fully determined due to the way the experiment was designed. Based on data from this experiment there would be no risk, since values are below recommended criteria for basements and the site is not contained the likelihood of radon accumulating to a concentration warranting management changes would be low. Radium-226 activity in PG is above NORM guidelines and is therefore being generated so if radon was not being blown off site a risk may be present. Building permanent structures on top of the water holding pond is not recommended and would require special design to limit radon accumulation.

Radium-226 results for PG in this study are within a range of typical PG values in sedimentary ore (0.2 - 3 Bq / g) and below the mean calculated from many studies (IAEA 2013). All PG and highly mixed samples were above Canadian guidelines for NORM and therefore management of the material may require special consideration. Radium-228 and thorium-228 are lower in PG since they are part of the thorium-232 decay series which is uncommon in marine sediments used to make PG (Rutherford et al. 1994). Potassium-40 is typically high in soil since potassium is an abundant element in the earth's crust. Lead-210 is a daughter product of radium-226 decay and expected to be higher in PG than other soils. In future reclamation of PG stacks reducing the amount of PG incorporated into the topsoil to prevent radium-226 activity in topsoil becoming above NORM guidelines is recommended. By incorporating high amounts of PG into the top horizons the potential of radioactive isotopes being translocated into the surrounding environment is increased.

The correlation of radium-226 and potassium-40 in soil is a useful management tool for Nutrien and other companies dealing with NORM. Capping waste material is not always socially acceptable since it as viewed as covering up a problem. Applying PG to soil offers a beneficial use of the waste instead of covering stacks. Knowing the radiological properties of PG is essential to prevent over application of PG causing the soil to become NORM. This correlation cannot be directly used with other PG, since the source of the phosphate ore can alter PG characteristics. However, it can be used as a guide that can easily be replicated. With a better understanding of PG variability, a more sophisticated model can be used to determine safe levels of PG to add to soil.

All isotopes analyzed in fresh vegetation were below NORM guidelines and likely do not pose a risk to animals eating the vegetation. Vegetation activity at Nutrien and Ellerslie was similar. Health Canada NORM guidelines (2011) state that 71000 Bq of radium-226 can be safely ingested over a year without negative effects. A relativity high amount of radium-226 can be ingested since a low percentage is retained in the human body during digestion. Using the vegetation sample with highest activity, this equates to a human eating nearly 15 kg of dried vegetation every day for a year. Since humans would not eat this volume in a year, there is currently no risk to humans. This guideline is for humans so effects on grazing and browsing animals cannot be accurately assessed, but is likely low.

Radium-226 activity in plants was low for all samples. Vegetation was not washed prior to analysis and therefore plant uptake can only be estimated. Previous studies found low correlation with soil to plant transfer of radium-226, unless a soil was highly contaminated (Marple 1980, Gerzabek et al. 1998, Cerne et al. 2011). Radium-226 activity in soil was negatively correlated with plant tissue activity in this study. Uptake of radium-226 by plants can be affected by alkaline earth metal concentrations in soil (IAEA 2009, IAEA 2013). Soils with high calcium, which is very high in PG, can significantly suppress radium plant uptake due to calcium competing for exchange sites on roots (Gerzabek et al. 1998). At a neutral pH radium is not in a bioavailable form, resulting in low plant uptake (Hewamanna et al. 1988). Hypothetically plants at Nutrien are taking up little, if any, radium and the radium-226 detected in samples is from PG dust on vegetation. This would explain why grass showed highest activity since it has a greater surface area for PG to collect on and is closer to the soil surface than woody plants.

The imbalance between radium-226 and lead-210 in vegetation can be attributed to naturally occurring radon gas in the air decaying and being deposited on vegetation as fallout. Radon-222 decays into polonium-218 which attaches to debris in the air and settles onto vegetation where it eventually decays into lead-210. This is supported by the Ellerslie data since radium-226 is below detection but lead-210, a daughter product of radium-226, is present. Lead-210 in

Nutrien samples is not substantially higher than Ellerslie since radon from PG is likely blowing off site before it can decay since it has a half life of 3.8 days.

Ashing vegetation samples resulted in a concentrated sample above NORM guidelines for lead-210. Thus if biomass at Nutrien is burnt as a renewable fuel source the ash may require special disposal. The ash from an entire tree would likely have different activity since a larger portion of ash would be from woody material, unlike the browse ashed in this study. Analyzing leaves, buds and wood separately would allow for a better estimation of ash quality.

5.3. Suitability Of Phosphogypsum Stacks For Forest Production

Pure PG is likely a poor soil substitute to support trees but amendments can ameliorate it. PG is categorized as a poor topsoil and subsoil based on the Soil Quality Criteria Relative to Disturbance and Reclamation guidelines due to low organic carbon content (Soil Classification Working Group 1987). PG is good or fair for all other soil properties measured. In this study all other soils had good organic carbon due to topsoil. Incorporating PG and soil can be an effective way to improve organic carbon in soil (AbouRisk 2015), making it a suitable soil based on criteria. Trace elements and isotopes in PG will likely not effect tree growth.

PG is a good source of calcium, phosphate and sulfate. Nitrate in PG in this study was high and above typical values for PG (Jackson 2011, AbouRisk 2015). This elevated nitrate could be attributed to Sheritt, a nearby nickel and cobalt refinery that previously owned the site, disposing nutrient rich waste water into the stacks (Nichol 2017). PG pH, sodium adsorption ratio, saturation and texture in this study were categorized as good for topsoil and subsoil reclamation in the Alberta plains region. PG is considered fair due to its salinity and poor due to low organic carbon. Ripped soil and tree soil have different proportions of PG incorporated into the mix and raise organic carbon to a good level. The soil cap used in this study is fair in salinity, saturation percent and texture. In general, the soils mixed with soil and PG had the best soil quality.

Jackson et al. (2011) found that soil caps greater than 15 cm vegetated with grass on PG resulted in little water infiltration into stacks. No studies are available for the effect of soil cap depth with trees. Although it cannot be confirmed without soil water data beneath the PG boundary, it appears that grass and woody vegetation in this study may be limiting water infiltration. Water content at the soil barrier remained relatively constant unless large precipitation events occurred. If infiltration into the stack was occurring a decline in water content would be seen at the PG barrier over time, but this was not seen. Future work is needed to better understand water dynamics below the PG boundary. Lack of infiltration could also be attributed to low amounts of water in the soil. Precipitation over the study was rapidly used by vegetation and small precipitation events resulted in no change to soil water content.

Grass growth on PG stacks with different soil cap depths has been well researched with recommended depths between 8 to 15 cm (Hallin et al. 2010, Jackson et al. 2011, Christensen 2013). A potential limitation of long term success of forest production on PG stacks is the presence of the compacted PG barrier. *Populus* species have shallow spreading root systems that allocate 75 % of root biomass to horizonal roots in the upper 20 cm of the soil (USDA 2018). The remaining 25 % of roots penetrate deep (3 to 5 m) into the soil to reach water from capillary rise above the water table and for physical stability in the soil (USDA 2018, Zasada and Phipps 1990). During soil sampling some trees were seen to have thin roots going into PG. Woody species do not appear to be under water stress, but they are still young and the effect of a shallow soil profile in future is not known. *Salix* and *Populus* water content at a 10 cm depth dropped below permanent wilting point when precipitation was infrequent. Although water content at the PG barrier remained within plant available water at this time, as trees get larger and water requirements change water may become limiting. The PG barrier could restrict downward root growth resulting in a water deficit and poor tree stability as trees mature. Reduced growth is undesirable since that will increase the harvest cycle duration and ultimately reduce the economic returns of plantations.

5.4. Building A Suitable Soil For Forestry Plantations

The soil built at Nutrien for this experiment appears to be accomplishing reclamation goals. Traditional cap and cover reclamation have been successful for grass but a different soil profile may be more effective for trees. PG at Nutrien contains trace elements below criteria that likely do not pose any risk to vegetation or animals in the surrounding environment. However, radium-226 activity is above NORM guidelines and high surface mixing currently on the site increased radium-226 activity in topsoil making in NORM contaminated. PG offers nutrient and physical benefits that improve soil quality but lacks organic carbon for nutrient cycling and other biological processes of importance in soils.

An alternative soil to that which is being used on the PG stacks may provide conditions for more successful plant growth and development. The goal for this soil would be to create a gradient of PG and soil throughout the profile with pure PG at the bottom and pure topsoil on the surface. By incorporating PG and soil at different quantities at different depths the benefits of both materials would be utilized, the risk to the environment would be controlled and vegetation should perform better than with topsoil capping. The bottom layer would consist of pure PG to a depth necessary to hold the volume of PG needed for the stack. Above this would be a high percentage of PG with soil mixed as deep as possible given equipment available. This horizon would promote rooting deeper into the stack and would break up the PG boundary layer. The next layer would be 10 to 15 cm thick, comprised mostly of soil with some PG mixed throughout. This would be the area where most of the horizonal rooting would occur. A pure soil cap of 5 to 10 cm would be placed on the surface to act as a physical barrier in the same way a topsoil cap has been used for grass.

This alternative soil would provide the benefits of increasing the rooting zone to a greater depth, decreasing radon gas emission, reducing surface soil NORM contamination and reducing infiltration. This proposed soil would require approximately 25 cm of topsoil and would extend the predicted rooting zone deeper than currently possible. This approach would require more intensive equipment and capital to set up. A soil where trees can root deeper into PG should increase water available in soil and provide greater stability for trees throughout their life cycle. If implemented, this soil must be evaluated to meet reclamation objectives for PG stacks.

6. CONCLUSIONS

No elements in soil were above CCME soil guidelines for industrial land use. Aluminum, beryllium, chromium, copper, iron, magnesium, manganese, nickel and vanadium were significantly higher in soil at Nutrien and Ellerslie than in PG. Cadmium, calcium, fluoride, sodium and strontium were significantly higher in pure PG than in soil. Soils with PG and soil mixed in different proportions had values between pure soil and pure PG.

Plant tissue concentrations differed significantly with vegetation type but not within similar species. Grass typically had lower concentrations of elements than woody species. Washing tissue samples did not result in differences relative to unwashed. Despite differences in soil concentrations, tissue concentrations for most plant species were similar at Nutrien and Ellerslie, suggesting that elements with high concentrations in PG were not in a bioavailable form. Trace elements and isotopes in PG will likely not affect plant growth.

Variability in radon-222 was seen in different vegetation covers, likely due to canopy structure, not uptake or changes from plants. Pure PG and highly mixed PG were above NORM guidelines. Vegetation contained very low concentrations of radium-226 and disproportionately high lead-210 activity as a result of atmospheric fallout.

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Table 3.1. Seed mix composition of the mixes seeded at the water holding pond research site.

Species	Reclamation Mix	Naeth Mix
Elymus trachycaulus	20	15
Pascopyrum smithii	20	15
Poa sandbergii	25	-
Deschampsia cespitosa	20	-
Puccinella distans	10	10
Festuca saximontana	-	15
Agrostis scabra	-	15
Elymus canadensis	-	15
Bromus ciliatus	-	15

All units are percent.

Table 3.2. Mean soil chemical and physical properties.

Soil Property	Ripped Soil	Tree Soil	Capped Soil	PG
Calcium (mg / L)	768 (15.7) a	701.7 (40.7) a	327 (131.3) b	693 (51.3) a
Chloride (mg / L)	13.7 (6.4)	25.3 (26.6)	14.3 (7.5)	10 (0)
Magnesium (mg / L)	110 (13)	122.3 (68.5)	85.3 (29.2)	92 (19.5)
Phosphate (Available) (mg / kg)	58.8 (10.2)	85.5 (20.9)	30.6 (7.1)	69.6 (14.4)
Potassium (mg / L)	28 (5)	31.7 (3.5)	24.8 (4.6)	10 (8.7)
Sodium (mg / L)	9.3 (4)	22.3 (20.5)	8 (2.1)	20 (26)
Sulfate (mg / L)	1783.3 (76.4) a	1940 (260) a	602.3 (487.7) b	1690 (131.1) a
Ammonia (mg / L)	0.4 (0)	0.8 (0.3)	0.4 (0)	1.1 (0.7)
Ammonium (mg / kg)	6.3 (0.1)	7 (0.5)	2.8 (0.4)	6.3 (3.5)
Nitrate (mg / L)	1.4 (1.1)	38.8 (66)	1 (0.4)	72.5 (108.9)
Organic Nitrogen (%)	0.3 (0.03) ab	0.3 (0.02) b	0.3 (0.03) a	0.02 (0.01) c
Inorganic Carbon (%)	0.2 (0.1)	0.1 (0.02)	0.2 (0.03)	0.1 (0.1)
Organic Carbon (%)	3.4 (0.3) c	2.8 (0.2) a	3.9 (0.3) b	0.3 (0.1) b
Total Carbon (%)	3.6 (0.2) a	3 (0.2) b	4 (0.3) a	0.3 (0.2) c
Cation Exchange Capacity (meq / 100 g)	47.1 (23) ab	28 (5.3) b	73.7 (0) a	NA
Hydrogen Ions (pH)	6.8 (0.2)	7 (0.2)	6.8 (0.2)	6.2 (1.2)
Electrical Conductivity (ds / m)	3.1 (0.05)	3.2 (0.6)	1.8 (0.6)	3.1 (0.5)
Sodium Adsorption Ratio	0.1 (0.04)	0.2 (0.2)	0.1 (0.04)	0.2 (0.2)
Sand (%)	32 (1)	39.3 (3.1)	23.7 (1.2)	36.3 (4.2)
Silt (%)	46.4 (6.9)	51.9 (3.4)	39.5 (0.5)	55.6 (4.6)
Clay (%)	21.6 (7)	8.8 (4.3)	36.8 (0.7)	8.1 (0.5)
Texture	Loam	Silt Loam	Clay Loam	Silt Loam
Field Capacity (cm^3 / cm^3)	0.38 (0.004) c	0.43 (0.003) b	0.47 (0.008) a	0.36 (0.006) d
Permanent Wilting Point (cm^3 / cm^3)	0.12 (0.0007) c	0.17 (0.005) b	0.23 (0.006) a	0.02 (0.005) d

Means with the same letter in each row are not statistically different.

Table 3.3. Soil chemical and physical properties relative to soil quality criteria for disturbance and reclamation for the plains region.

Soil Property	Soil Layer	Ripped Soil	Capped Soil	Tree Soil	PG
Hydrogen Ions (pH)	Topsoil	Good	Good	Good	Good
	Subsoil	Good	Good	Good	Good
Electrical Conductivity	Topsoil	Fair	Good	Fair	Fair
	Subsoil	Fair	Fair	Fair	Fair
Sodium Adsorption Ratio	Topsoil	Good	Good	Good	Good
	Subsoil	Good	Good	Good	Good
Saturation	Topsoil	Fair	Fair	Good	Good
	Subsoil	Fair	Fair	Good	Good
Texture	Topsoil	Good	Fair	Good	Good
	Subsoil	Good	Fair	Good	Good
Organic Carbon	Topsoil	Good	Good	Good	Poor
	Subsoil	-	-	-	-
Inorganic Carbon	Topsoil	Good	Good	Good	Good
	Subsoil	-	-	-	-

Adapted from Soil Classification Working Group (1987).

Table 3.4. Summary table of soil elements not included in statistical comparison.

Element	Capped Soil	Ripped Soil	Soil PG Mix	PG	Ellerslie	Criteria
Antimony	0.3 (0.1)	0.3 (0)	0.2 (0)	0.2 (0)	0.2 (0)	40
Arsenic	7.1 (0.4)	6.7 (0.5)	4.9 (1.6)	1.7 (0.4)	6.5 (0.2)	12
Barium	177 (7.9)	162.7 (14)	105.7 (47.4)	46.1 (15.7)	171.3 (13.1)	2000
Bismuth	0.1 (0.1)	0.1 (0.1)	0.1 (0)	0.1 (0)	0.1 (0)	-
Boron	11.5 (3)	10.9 (0.8)	11.5 (1.7)	4 (2.7)	7.4 (0.5)	-
Cobalt	10.5 (0.2)	9.6 (0.4)	8 (2.5)	2.7 (0.8)	9.4 (1)	300
Lead	13.2 (2)	11.5 (0.6)	9.8 (2.2)	6.6 (0.5)	11.2 (1)	600
Lithium	18.3 (3.6)	15.7 (1.3)	12.2 (3.5)	2 (1.8)	16.5 (4.3)	-
Molybdenum	0.5 (0.2)	0.8 (0.3)	0.6 (0.1)	0.6 (0.2)	0.5 (0)	40
Phosphorus	808.3 (121.8)	1044.3 (146.8)	1678.7 (667.8)	2800 (447.1)	732 (154.6)	-
Potassium	2383.3 (110.2)	2266.7 (136.1)	2280 (298.7)	633.3 (319)	1340 (26.5)	-
Selenium	0.3 (0)	0.4 (0)	0.4 (0.1)	0.4 (0.2)	0.5 (0)	2.9
Silver	0.1 (0)	0.1 (0)	0.2 (0.1)	0.2 (0.1)	0.1 (0)	40
Sulfur	1866.7 (2367.1)	19033.3 (5762.2)	54700 (39020.9)	90933.3 (8555.9)	500 (0)	-
Thallium	0.2 (0)	0.2 (0)	0.2 (0)	0.1 (0)	0.1 (0)	1
Titanium	36.8 (7.8)	45.4 (2.4)	90.4 (39.1)	64.7 (17.7)	39.1 (11.3)	-
Uranium	1 (0.3)	1.3 (0.1)	2.8 (1.4)	4.9 (2.2)	2.4 (1.2)	300
Zinc	79.2 (0.1)	72.8 (5.6)	52.8 (21.8)	12.2 (6.3)	63.6 (0.2)	360
Zirconium	7.3 (0.8)	7.3 (0.4)	6.5 (0.7)	3.3 (1.5)	6 (0.9)	-

All units in mg / kg.
Standard error stated within brackets

Table 3.5. Mean element contents in each soil treatment for elements that showed variable levels.

Element	Capped Soil	Ripped Soil	Soil PG Mix	PG	Ellerslie	Criteria
Aluminum	16066.7 (1069.3) a	16233.3 (981.5) a	14433.3 (2565.8) a	3970 (2165.4) b	13000 (1562) a	-
Beryllium	0.9 (0.2) a	0.8 (0.0) a	0.6 (0.1) a	0.2 (0.1) b	0.7 (0.1) a	8
Cadmium	0.2 (0.0) c	0.3 (0.0) bc	0.4 (0.1) b	0.5 (0.1) a	0.2 (0.0) c	22
Calcium	12723.3 (4659.3) c	34000 (6794.9) bc	75500 (46155.3) ab	116666.7 (13796.1) a	9050 (1713.5) c	-
Chromium	22.4 (0.9) a	22 (1.7) a	19.5 (3.3) a	6.2 (2.5) b	17 (1.7) a	87
Copper	23.4 (1.1) a	21.2 (1.4) a	17.1 (5.3) a	6.9 (1.2) b	17.4 (1.4) a	91
Fluoride	7.5 (9.5) b	44 (1.5) a	47.4 (10.0) a	55.1 (14.1) a	0.5 (0.4) b	-
Iron	21733.3 (757.2) a	19666.7 (1497.8) ab	13983.3 (5410.7) b	3056.7 (1069.3) c	19500 (1253) ab	-
Magnesium	4886.7 (109.7) a	4240 (174.4) a	3103.3 (1117.4) a	843.3 (795.2) b	3706.7 (559) a	-
Manganese	354 (29.1) a	329.3 (36.6) ab	206.3 (92.9) b	43.2 (16.1) c	421.7 (37.9) a	-
Nickel	58.3 (13.3) a	47.2 (5.4) ab	46.6 (17.2) ab	11.3 (1.9) c	22 (1.5) bc	89
Sodium	58.3 (8.7) b	96 (18.0) b	138 (75.3) ab	291 (119) a	61.7 (3.2) b	-
Strontium	68.7 (16.9) c	122.3 (19.6) bc	222 (114.4) ab	312 (33.5) a	60.9 (16.0) c	-
Vanadium	39.8 (3.0) a	39.7 (2.5) a	35.4 (6.4) a	9.7 (4.8) b	31.1 (4.0) a	130

All units are in mg / kg.
Standard error indicated in brackets.
Means with the same letter in each row are not statistically different.
Criteria is from CCME soil guidelines.

Table 3.6. Mean isotope activity in soil.

Isotope	Low Soil Mixing	High Soil Mixing	Ripped Soil	Capped Soil	Pure PG
Radium-226	0.139 ± 0.001 c	0.375 ± 0.002 b	0.190 ± 0.002 ab	0.043 ± 0.001 c	0.690 ± 0.002 a
Radium-228	0.034 ± 0.002 a	0.023 ± 0.002 b	0.030 ± 0.002 ab	0.036 ± 0.002 a	0.009 ± 0.001 c
Thorium-228	0.034 ± 0.001 a	0.024 ± 0.001 b	0.032 ± 0.001 ab	0.036 ± 0.001 a	0.010 ± 0.001 c
Lead-210	0.134 ± 0.009 c	0.350 ± 0.014 b	0.172 ± 0.010 c	0.038 ± 0.007 c	0.629 ± 0.015 a
Potassium-40	0.523 ± 0.015 a	0.332 ± 0.013 b	0.476 ± 0.014 ab	0.563 ± 0.014 a	0.066 ± 0.006 c

All units are in Bq / g.
Numbers following the mean is the uncertainty on one standard deviation in measurement.
Means with the same letter in each row are not statistically significant

Table 3.7. Vegetation tissue concentrations of elements that had some samples below detection.

		Populus Balsamifera FFC1		*Populus Balsamifera* NM6		India		*Viminalis*		Grass	
	Location	Mean	BD	Mean	BD	Mean	BD	Mean	BD	Mean	BD
Aluminum	Nutrien	51.7 (16)	0	27.8 (11)	17	61.2 (17)	0	62.7 (26)	0	28.4 (9)	8
	Ellerslie	47.3 (11)	0	67 (10)	0	90.3 (74)	33	140.7 (3)	0	41.7 (8)	0
Boron	Nutrien	44.7 (12)	0	34.3 (8)	0	27.2 (1)	0	27.5 (2)	0	4.5 (2)	25
	Ellerslie	45.1 (6)	0	47.4 (7)	0	28.7 (24)	33	34.9 (1)	0	4.2 (2)	33
Cadmium	Nutrien	0.7 (0.2)	0	0.6 (0.1)	0	1.1 (0.4)	0	0.9 (0.1)	0	0.03 (0)	100
	Ellerslie	0.7 (0.3)	0	0.5 (0.2)	0	0.8 (0.7)	0	0.7 (0.2)	0	0.03 (0)	100
Cobalt	Nutrien	0.3 (0.2)	0	0.3 (0.1)	0	1.1 (0.4)	0	0.9 (0.3)	0	5.4 (2)	0
	Ellerslie	0.5 (0.1)	0	0.6 (0.1)	0	1.1 (0.9)	33	2.2 (0.3)	0	5.1 (1)	100
Chromium	Nutrien	2.2 (0.6)	67	2.3 (1)	50	2.8 (0.7)	0	2.7 (0.6)	0	0.6 (0.1)	0
	Ellerslie	0.4 (0.1)	0	0.3 (0.1)	0	0.5 (0.4)	33	0.5 (0.04)	0	0.08 (0)	0
Lead	Nutrien	0.04 (0)	100	0.04 (0)	100	0.07 (0.03)	50	0.07 (0.04)	50	0.04 (0)	100
	Ellerslie	0.04 (0)	100	0.04 (0)	100	0.11 (0.1)	33	0.2 (0.01)	0	0.04 (0)	100
Molybdenum	Nutrien	0.3 (0.2)	33	0.7 (0.4)	0	1.3 (0.2)	0	0.5 (0.1)	0	0.6 (0.3)	0
	Ellerslie	0.5 (0.2)	0	0.3 (0.1)	0	0.2 (0.1)	67	0.2 (0.03)	0	0.6 (0.2)	0
Selenium	Nutrien	0.2 (0)	100	0.2 (0)	100	0.2 (0)	100	0.2 (0)	100	0.2 (0)	100
	Ellerslie	0.2 (0)	100	0.2 (0)	100	0.2 (0)	100	0.6 (0.3)	33	0.2 (0)	100
Sodium	Nutrien	20 (0)	100	20 (0)	100	20 (0)	100	97.2 (21)	0	20 (0)	100
	Ellerslie	20 (0)	100	20 (0)	100	20 (0)	100	45 (4)	0	20 (0)	100
Tin	Nutrien	0.13 (0.1)	83	0.11 (0.1)	83	0.08 (0)	100	0.08 (0)	100	0.08 (0)	100
	Ellerslie	0.08 (0)	100	0.08 (0)	100	0.08 (0)	100	0.08 (0)	100	0.08 (0)	100
Titanium	Nutrien	0.7 (0.3)	67	0.6 (0.2)	83	1.6 (0.6)	17	1.4 (0.7)	17	0.6 (0.2)	83
	Ellerslie	1 (0.5)	33	1.6 (0.2)	0	2.4 (2)	33	3.8 (0.2)	0	0.9 (0.3)	33
Uranium	Nutrien	0.02 (0.02)	67	0.01 (0.01)	67	0.03 (0.03)	50	0.01 (0.01)	83	0.01 (0)	100
	Ellerslie	0.01 (0)	100	0.01 (0)	100	0.01 (0)	33	0.01 (0)	100	0.01 (0)	100
Vanadium	Nutrien	0.08 (0.04)	67	0.07 (0.03)	67	0.2 (0.04)	0	0.2 (0.1)	0	0.14 (0.04)	8
	Ellerslie	0.11 (0.1)	33	0.2 (0.02)	0	0.2 (0.2)	33	0.4 (0.03)	0	0.2 (0.04)	0

Standard deviation indicated in brackets. Mean units are mg / kg, BD is the percent of samples below detection.

Table 3.8. Tissue concentrations of vegetation sampled during 2016 and 2017 at Nutrien.

Element	Year	Grass	India	*Viminalis*	*Populus Balsamifera* (FFC-1)	*Populus Balsamifera* (NM-6)
Aluminum	2016	79.2	413.0	131.0	127.0	758.0
	2017	28.4 (6.2)	61.2 (8.8)	62.7 (8.8)	51.7 (8.8)	27.8 (8.8)
Barium	2016	11.9	3.0	2.2	2.2	5.7
	2017	19.7 (2.2)	2.9 (3.1)	1.8 (3.1)	2.5 (3.1)	2.1 (3.1)
Boron	2016	5.5	18.0	31.0	39.0	28.0
	2017	4.5 (2.2)	27.2 (3.1)	27.5 (3.1)	44.7 (3.1)	34.3 (3.1)
Cadmium	2016	0.03	0.6	1.1	0.8	0.5
	2017	0.03 (0.1)	1.1 (0.1)	0.9 (0.1)	0.7 (0.1)	0.6 (0.1)
Calcium	2016	2556.7	12700.0	13700.0	15600.0	12200.0
	2017	4044.2 (890.5)	15300 (1259.4)	9191.7 (1259.4)	18200 (1259.4)	13076.7 (1259.4)
Chromium	2016	2.3	2.3	1.6	1.2	5.5
	2017	5.4 (0.3)	1.1 (0.4)	0.9 (0.4)	0.3 (0.4)	0.3 (0.4)
Cobalt	2016	0.2	1.6	2.2	1.6	2.2
	2017	0.6 (0.2)	2.8 (0.2)	2.7 (0.2)	2.2 (0.2)	2.3 (0.2)
Fluoride	2016	2.8	24.0	38.5	12.5	10.5
	2017	9.7 (1.2)	8.7 (1.7)	13.3 (1.7)	12.2 (1.7)	8.1 (1.7)
Magnesium	2016	1032.7	3280.0	3980.0	3290.0	3210.0
	2017	1014 (224.2)	4256.7 (317.1)	3421.7 (317.1)	4800 (317.1)	4301.7 (317.1)
Potassium	2016	10213.3	16200.0	15700.0	14800.0	12900.0
	2017	5611.7 (746.7)	13950 (1056)	15750 (1056)	14900 (1056)	13200 (1056)
Sodium	2016	51.5	85.0	779.0	39.0	97.0
	2017	20 (2.1)	20 (3)	97.2 (3)	20 (3)	20 (3)
Strontium	2016	17.0	61.7	71.0	96.6	66.5
	2017	33.8 (6.5)	100.3 (9.2)	53.7 (9.2)	121 (9.2)	96.8 (9.2)
Zinc	2016	11.5	58.8	57.7	41.9	35.5
	2017	10.7 (7.2)	55.1 (10.2)	40.1 (10.2)	60.3 (10.2)	33.9 (10.2)

2016 results are leaf samples with no replication, 2017 samples are browse with standard deviation indicated in brackets.
All units are in mg / kg.

Table 3.9. Vegetation concentration from the highest Nutrien sample relative to maximum tolerable levels for rodents and cattle.

Element	Highest Value From Nutrien	Rodent Maximum Tolerable Level	Cattle Maximum Tolerable Level
Aluminum	99	200	1000
Antimony	0.1	70	-
Arsenic	0.1	30	30
Barium	33	250	-
Bismuth	0.3	500	-
Boron	62	15	150
Cobalt	3	25	25
Copper	6	500	40
Fluoride	21	150	40
Iron	203	500	500
Lead	0.1	10	100
Lithium	2	25	25
Manganese	165	2000	2000
Molybdenum	2	7	5
Nickel	32	50	100
Selenium	0.2	5	5
Strontium	160	1000	2000
Tin	0.4	100	100
Uranium	0.1	100	-
Vanadium	0.3	-	50
Zinc	67	500	500

All units ug / mg.

Adapted from National Research Council (2005).

Table 3.10. Radioactive isotope activity vegetation samples.

Sample	Sample Matrix	Mass (g)	Count Time (s)	Radium-226	Radium-228	Thorium-228	Lead-210	Potassium-40
Grass Capped Soil	Ground Vegetation	12.646	230000	0.009 ± 0.001	< 0.007	< 0.003	0.023 ± 0.005	0.17 ± 0.01
Grass Ripped Soil	Ground Vegetation	14.191	250000	0.013 ± 0.001	< 0.006	< 0.003	0.044 ± 0.006	0.25 ± 0.01
Poplar	Ground Vegetation	25.644	235000	< 0.005	< 0.005	< 0.003	0.016 ± 0.003	0.38 ± 0.01
Willow *	Ground Vegetation	51.64	300000	0.004 ± 0.0003	< 0.01	< 0.01	0.035 ± 0.001	0.343 ± 0.003
Willow *	Ground Vegetation	40.44	250000	0.004 ± 0.0003	< 0.01	< 0.01	0.024 ± 0.001	0.630 ± 0.005
Ellerslie Willow	Ground Vegetation	35.792	300000	< 0.01	< 0.01	< 0.01	0.034 ± 0.003	0.4 ± 0.01
Willow	Ashed Vegetation	3.7401	300000	0.06 ± 0.004	< 0.01	< 0.01	0.49 ± 0.02	4.73 ± 0.04
Willow	Ashed Vegetation	2.6506	250000	0.062 ± 0.005	< 0.01	< 0.01	0.35 ± 0.02	9.62 ± 0.07

*Calculated value.
Units of activity in Bq / g.
Uncertainty ± one standard deviation; 68 % confidence limit.

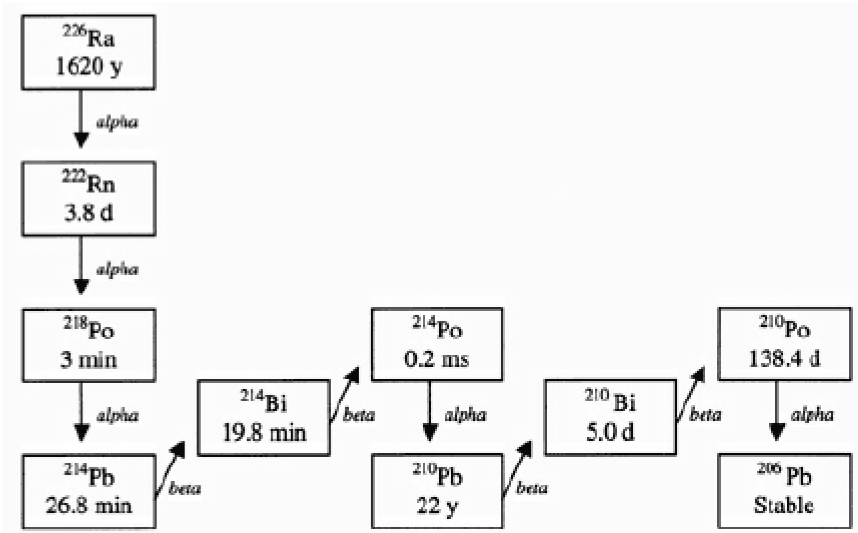

Figure 3.1. Radium-226 decay series. (ENHS n.d.)

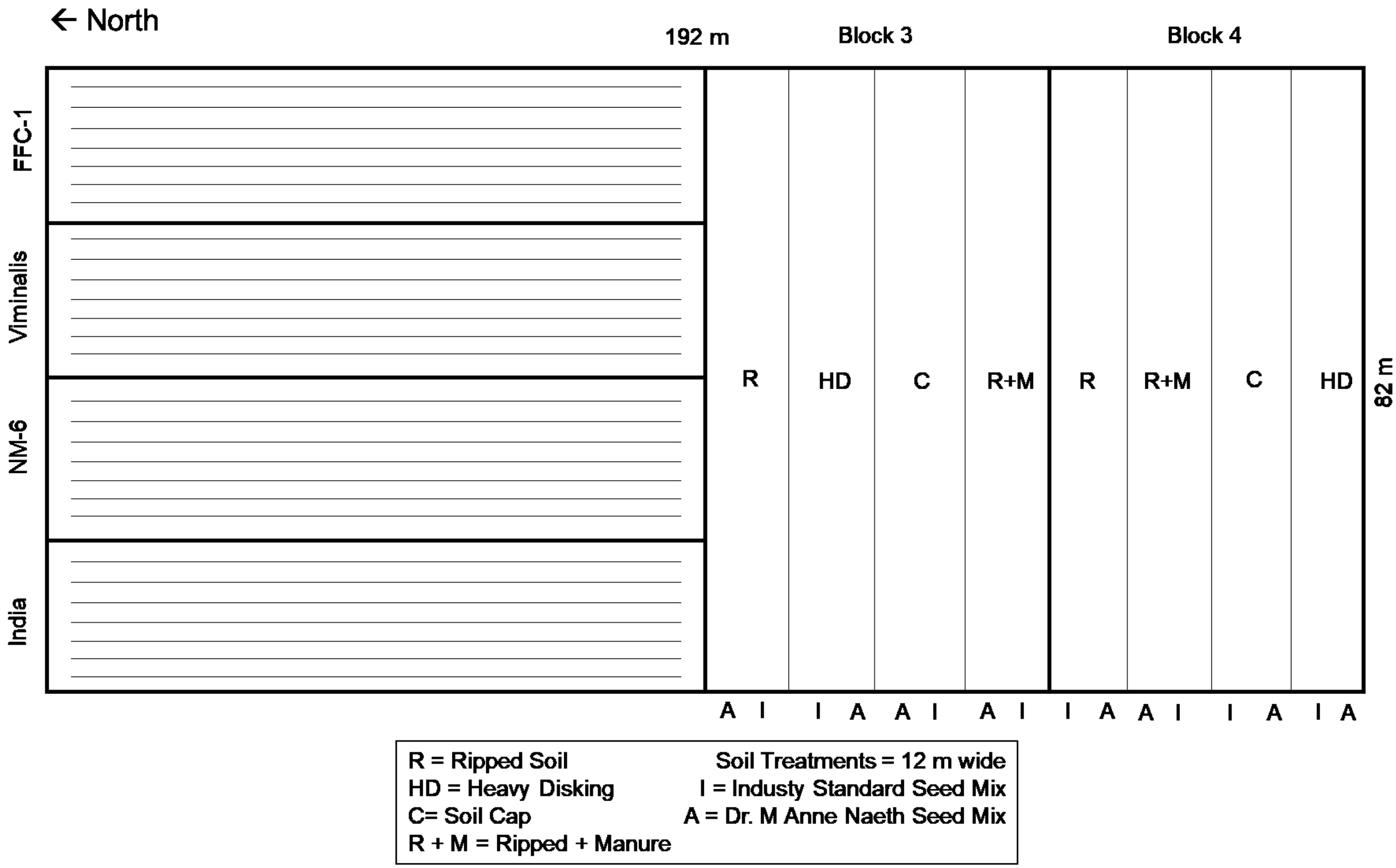

Figure 3.2. Map of vegetation and soil treatments on the water holding pond.

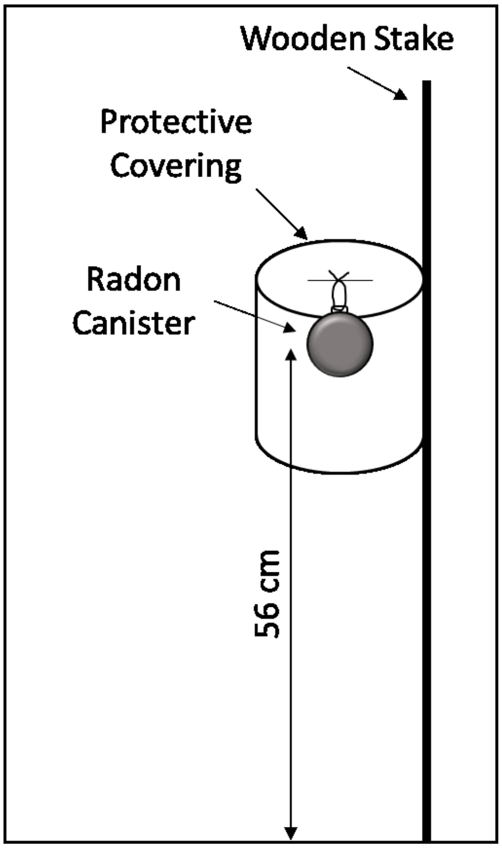

Figure 3.3. Radon canister set up schematic.

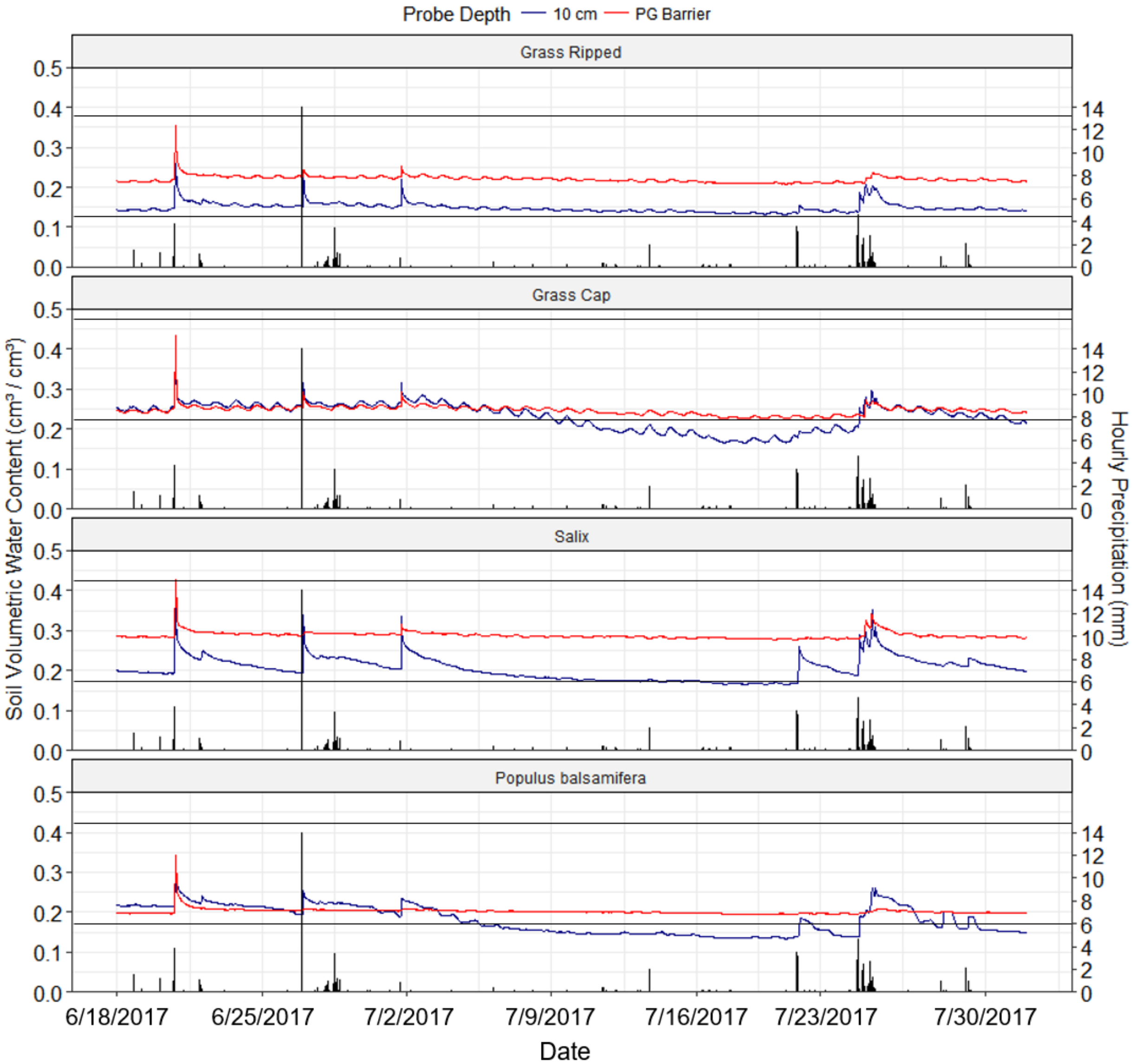

Figure 3.4. Soil water content at two depths in different vegetation covers. Upper and lower horizontal lines indicate field capacity and wilting point respectively.

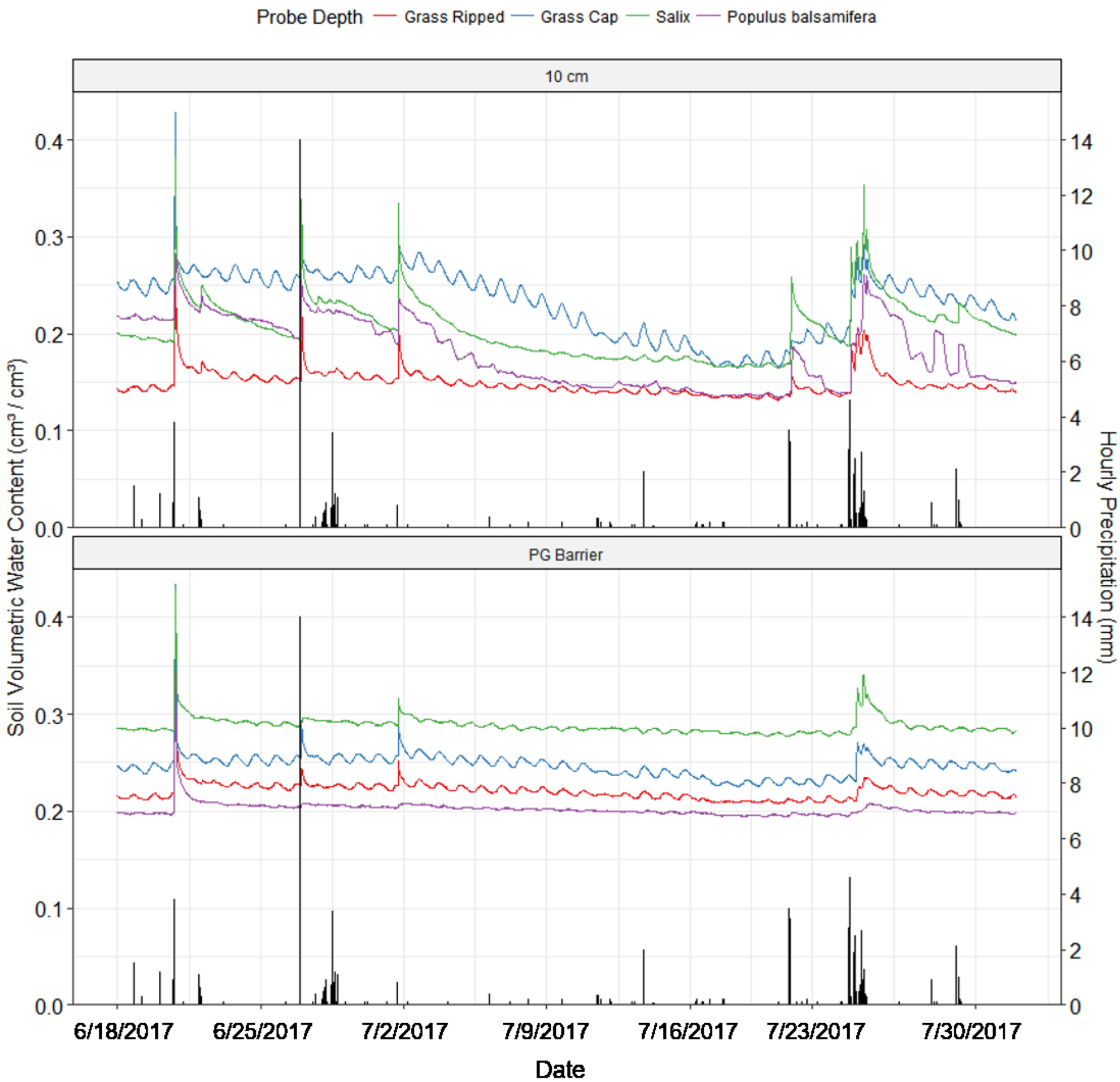

Figure 3.5. Soil water content under different vegetation covers for two soil probe depths.

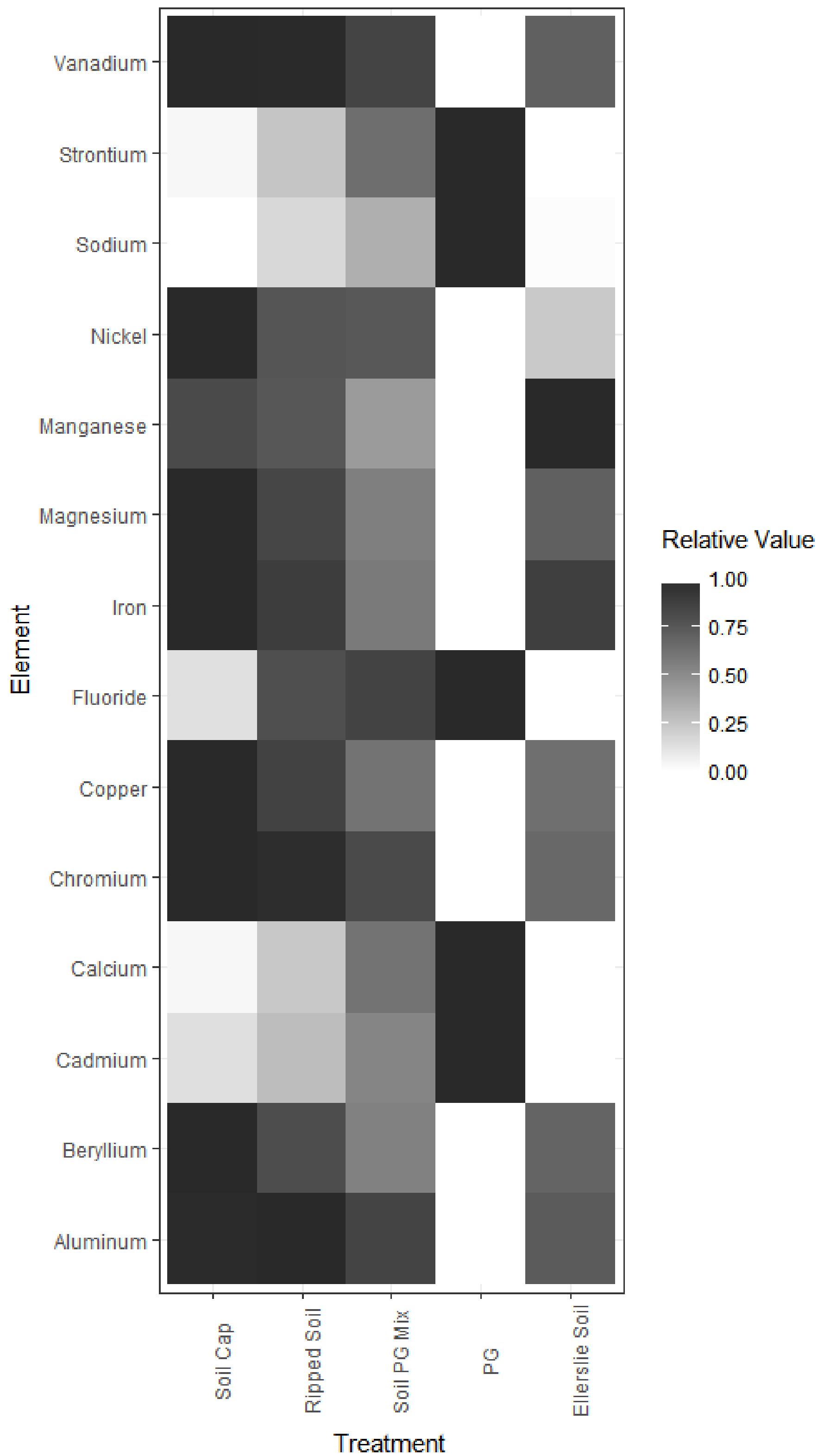

Figure 3.6. Soil heat map of elements chosen for comparison in each soil treatment. The darker the value the higher the mean value in that treatment relative to other treatments.

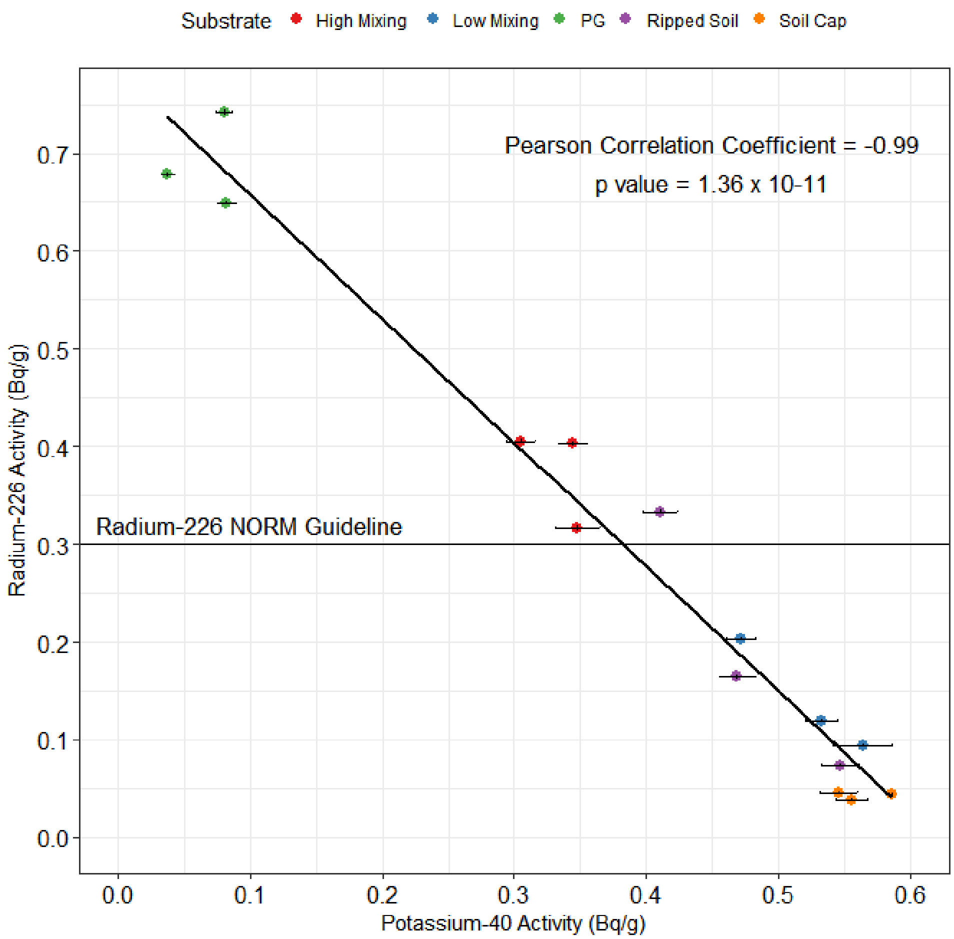

Figure 3.7. Radium-226 and potassium-40 activity in soil correlation.

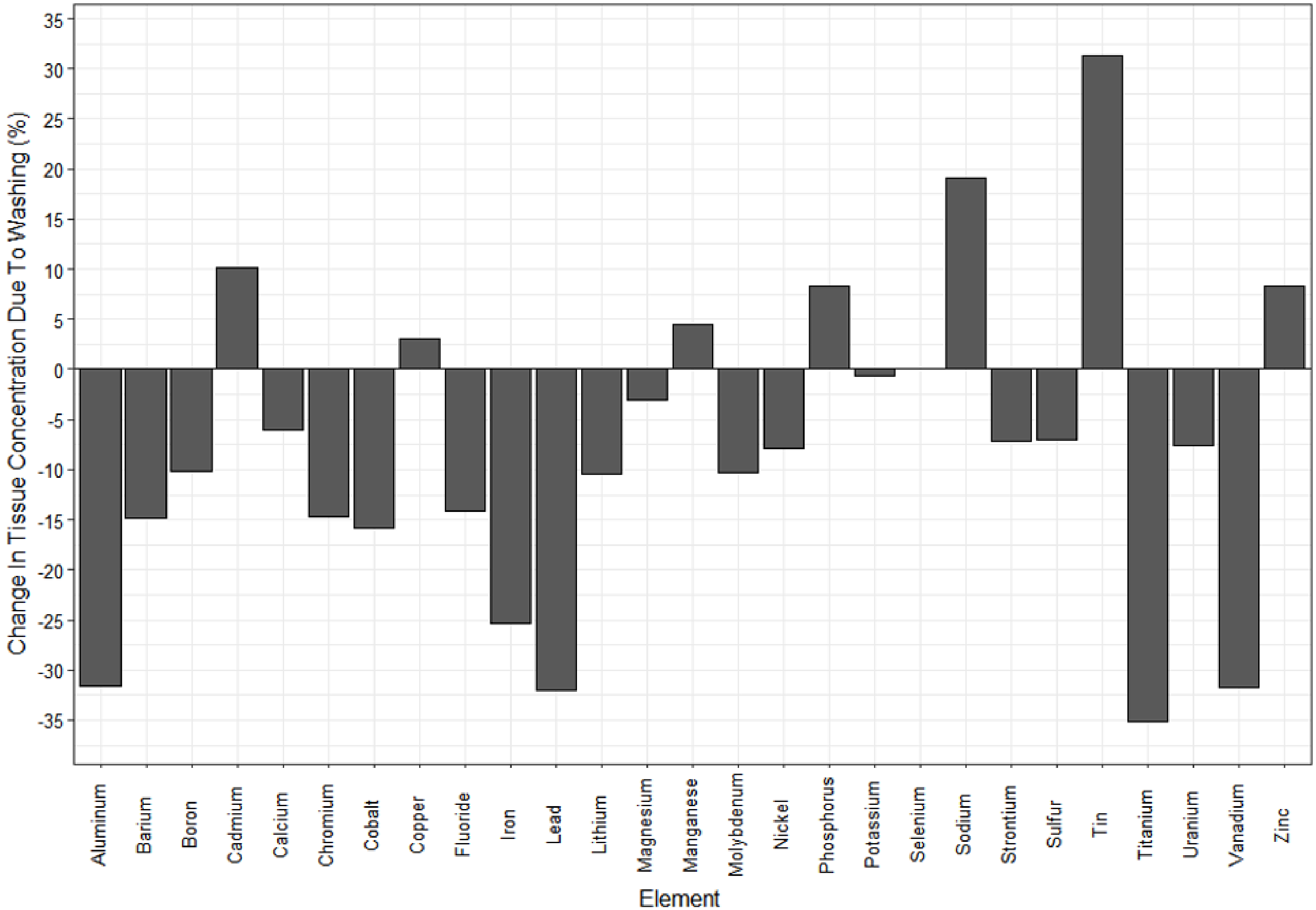

Figure 3.8. Change in analysis results due to washing vegetation. Percent calculated by taking washed-unwashed/unwashed.

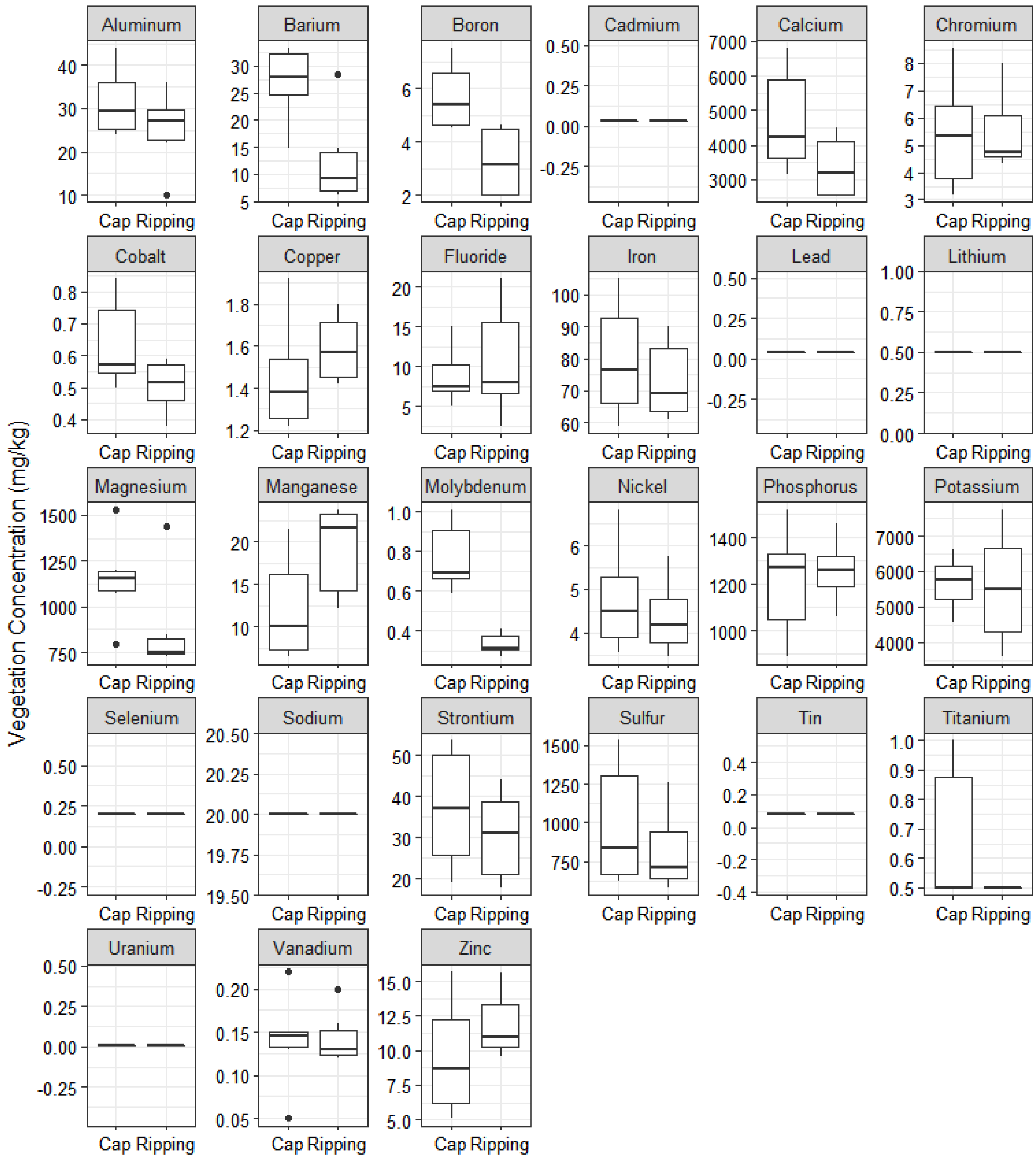

Figure 3.9. Boxplots of elements comparing effects of ripped soil and capped soil treatments on grass tissue concentration.

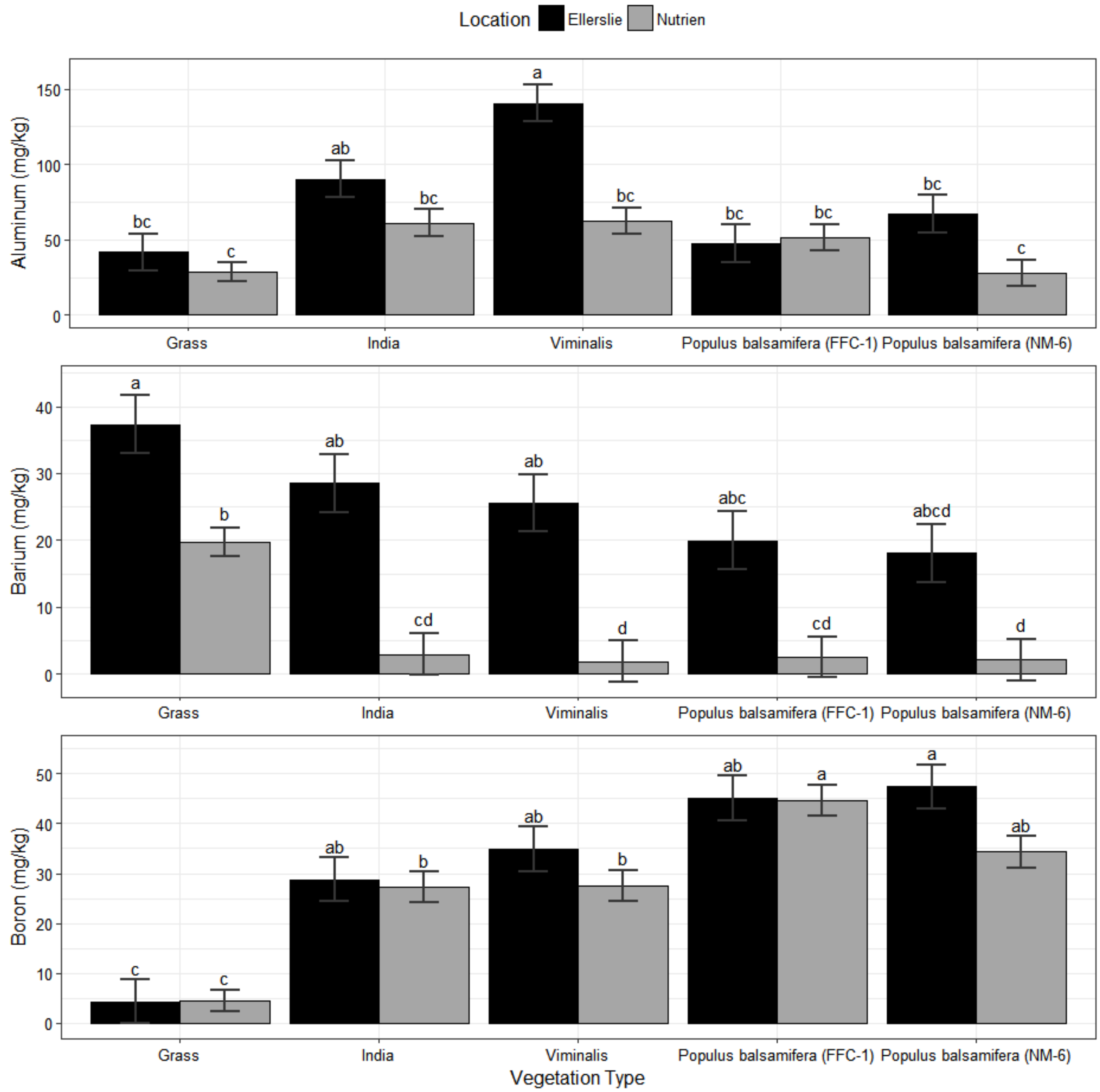

Figure 3.10. Element concentrations for aluminum, barium and boron in vegetation from Nutrien and Ellerslie. Standard error indicated with error bars. Bars with the same letter are not statistically significant from each other.

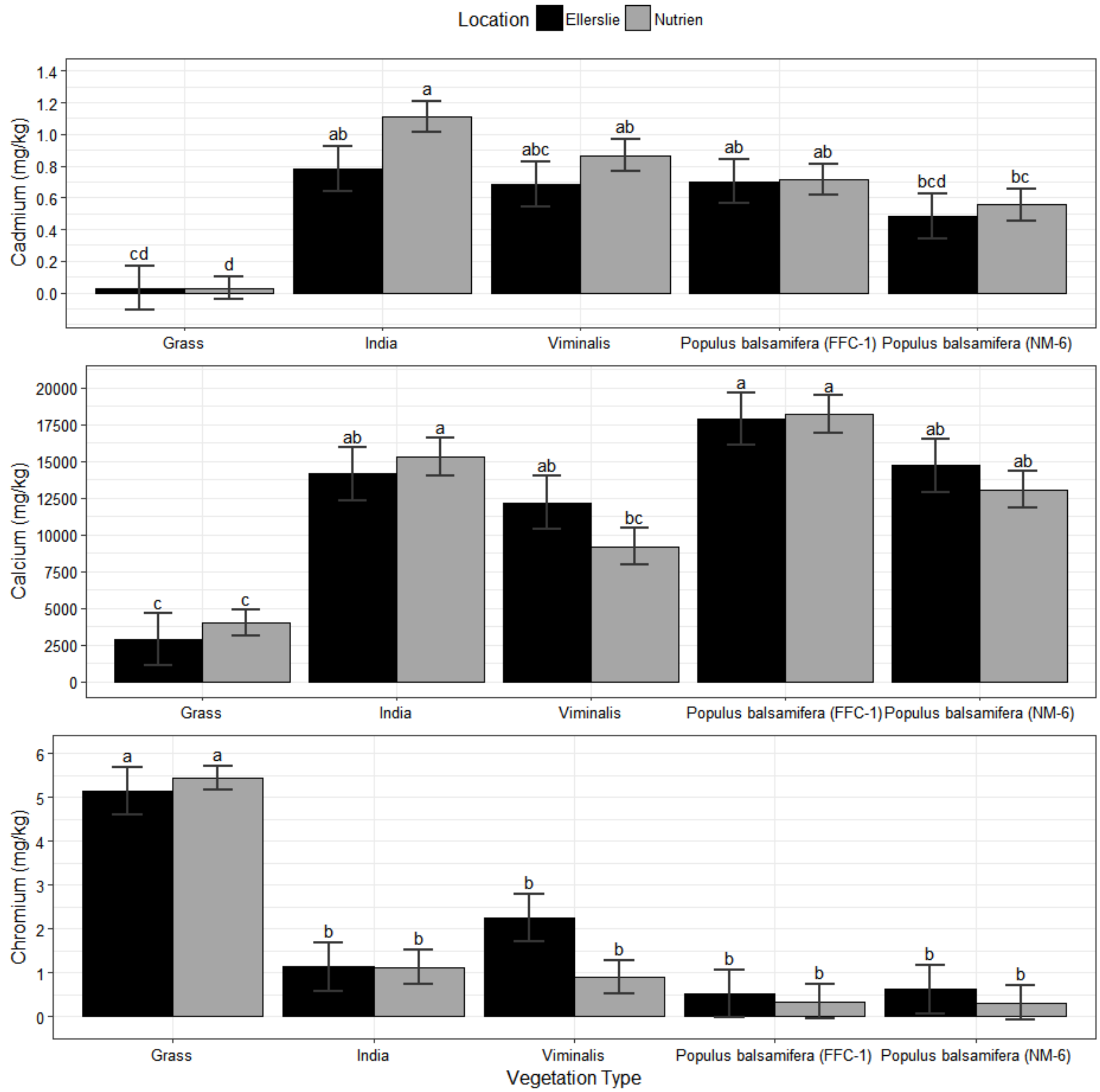

Figure 3.11. Element concentrations for cadmium, calcium and chromium in vegetation from Nutrien and Ellerslie. Standard error indicated with error bars. Bars with the same letter are not statistically significant from each other.

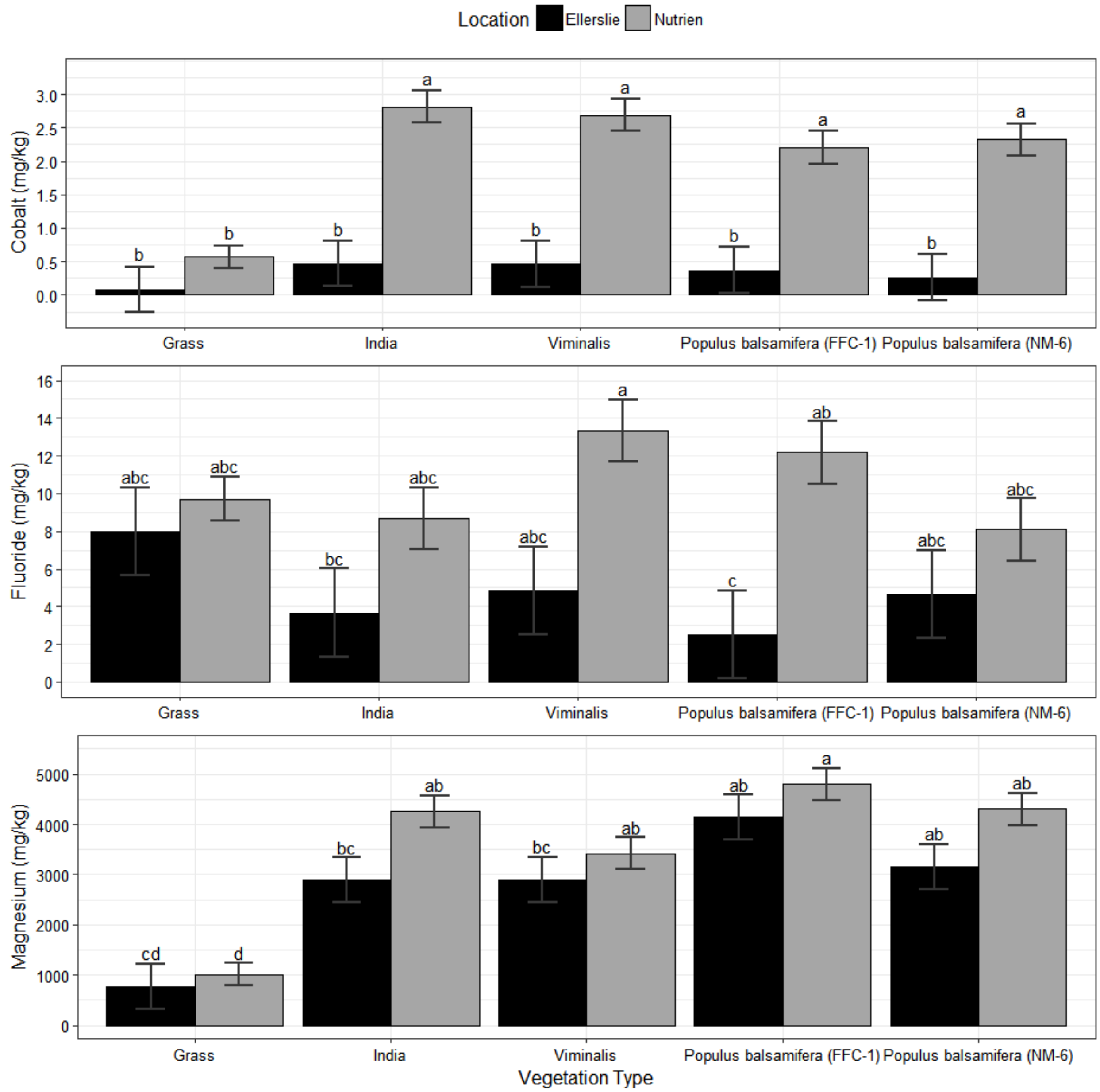

Figure 3.12. Element concentrations for cobalt, fluoride and magnesium in vegetation from Nutrien and Ellerslie. Standard error indicated with error bars. Bars with the same letter are not statistically significant from each other.

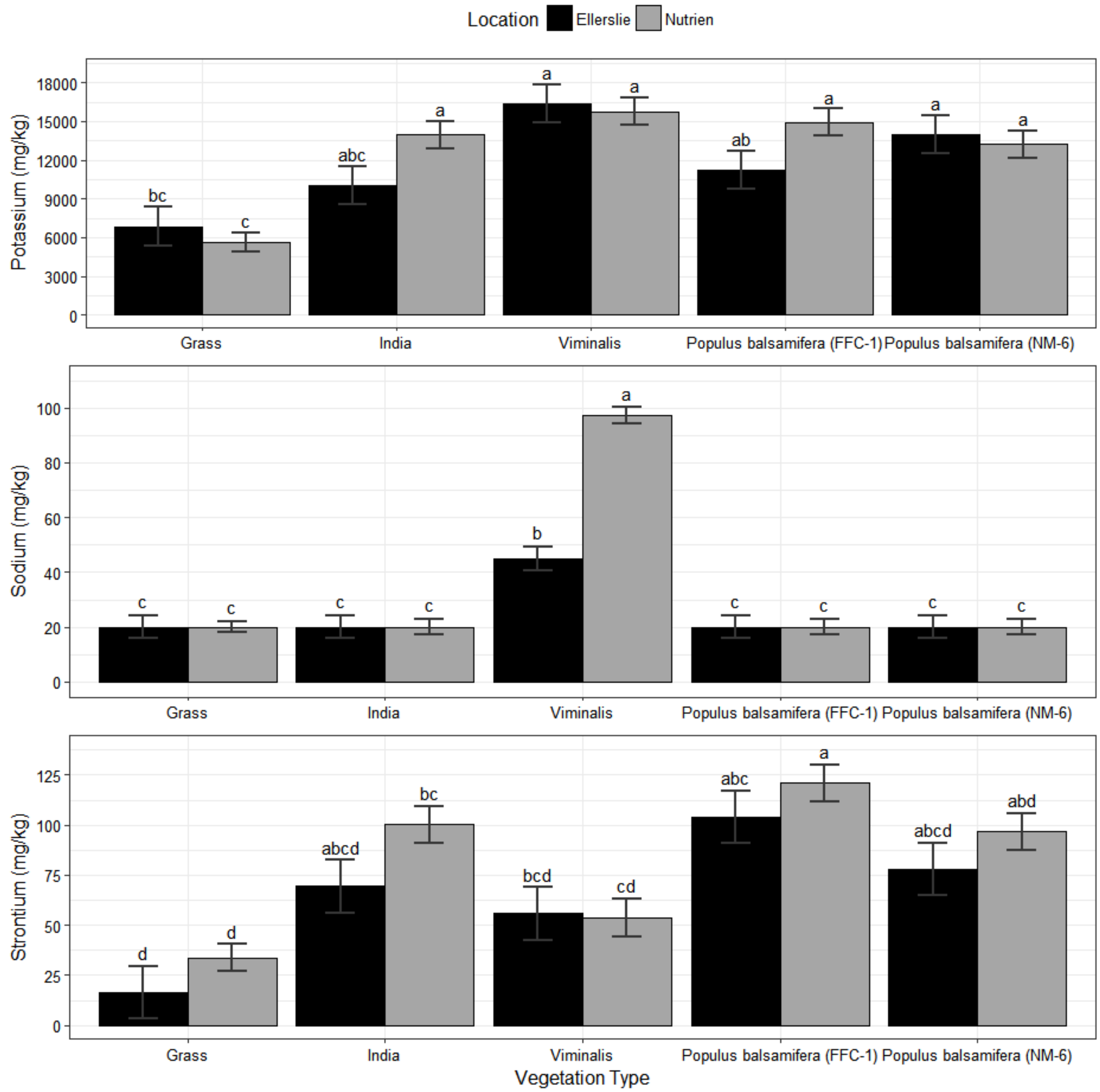

Figure 3.13. Element concentrations for potassium, sodium and strontium in vegetation from Nutrien and Ellerslie. Standard error indicated with error bars. Bars with the same letter are not statistically significant from each other.

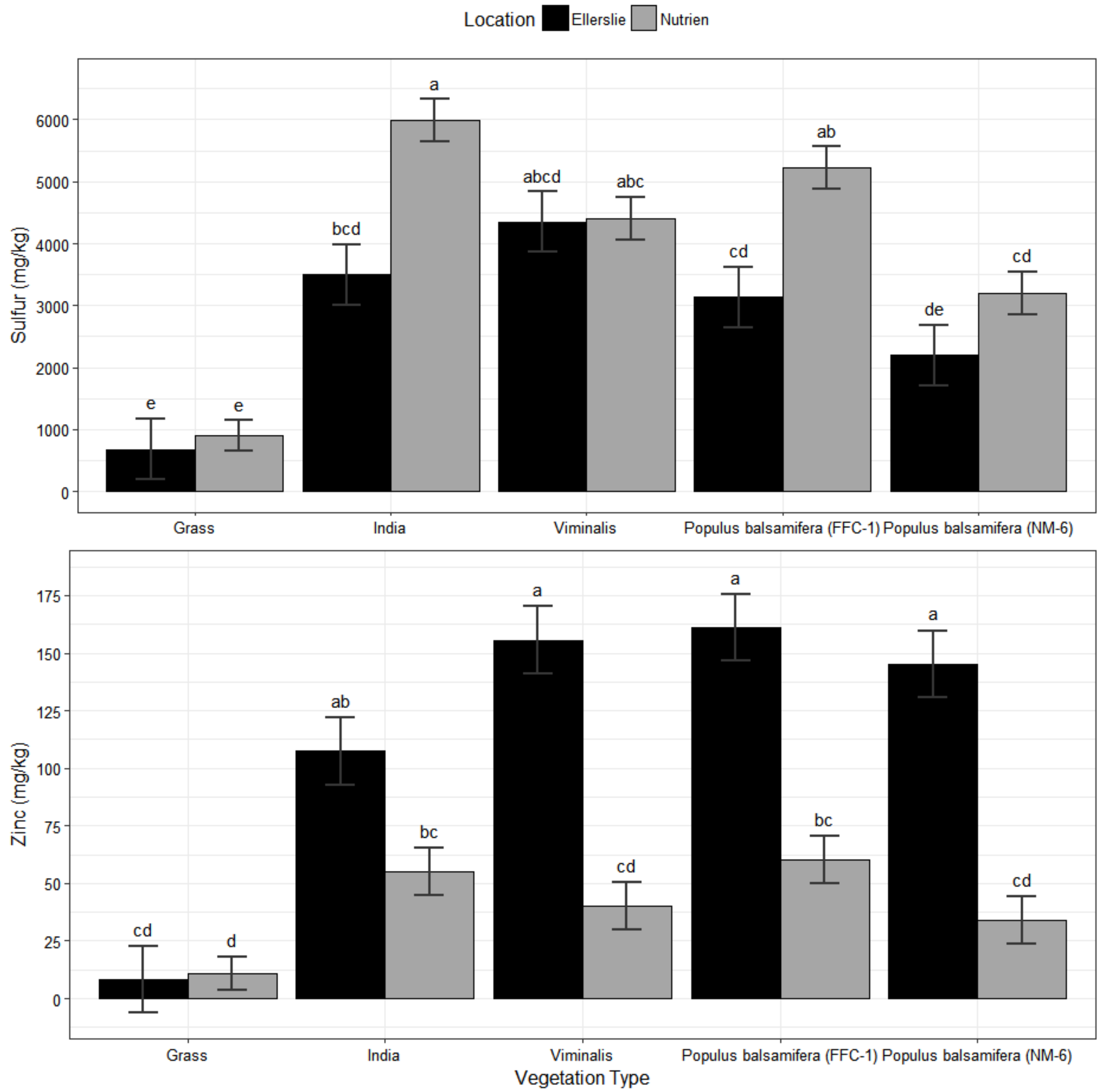

Figure 3.14. Element concentrations for sulfur and zinc in vegetation from Nutrien and Ellerslie. Standard error indicated with error bars. Bars with the same letter are not statistically significant from each other.

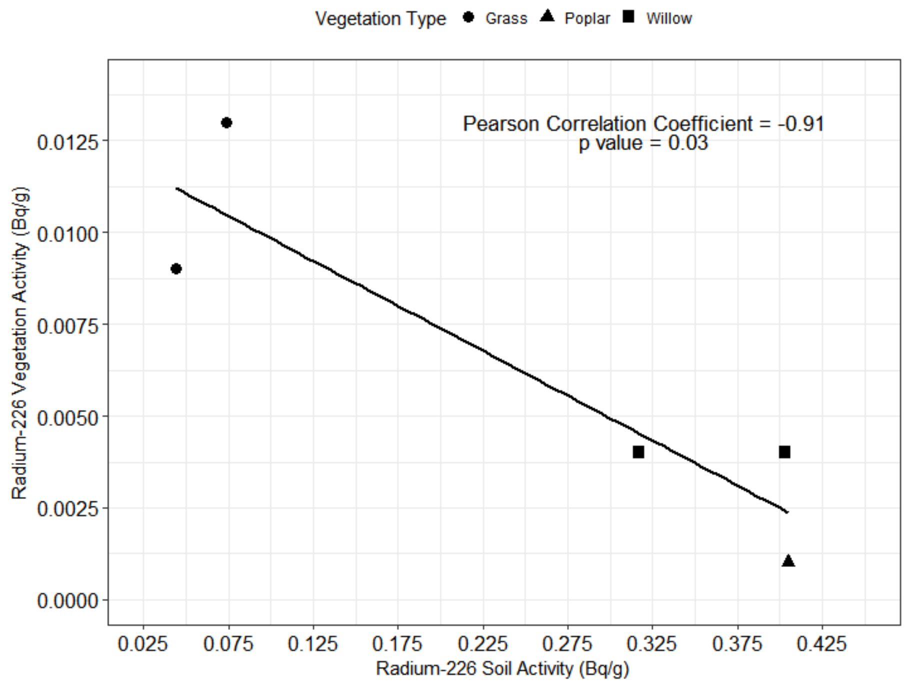

Figure 3.15. Radium-226 activity in soil and vegetation correlation for plant samples at Nutrien.

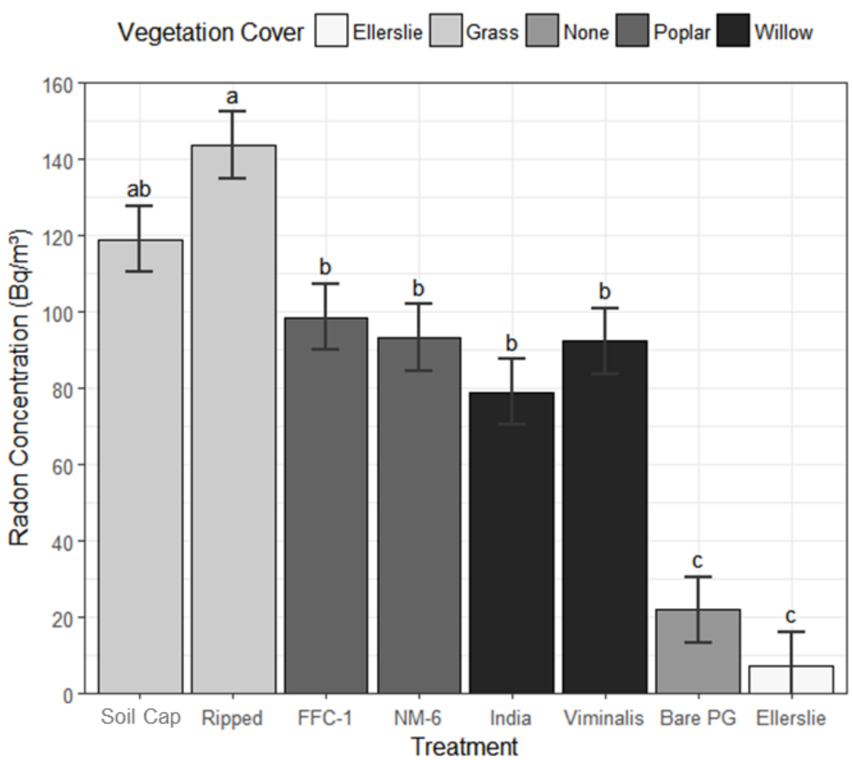

Figure 3.16. Radon-222 activity in different vegetation covers from Nutrien and Ellerslie.

IV. RESEARCH SYNTHESIS

1. RESEARCH SUMMARY

This research showed good potential for phosphogypsum (PG) stack reclamation using short rotational forestry plantations. Pure PG is likely not suitable as a soil to support trees on its own because of low organic carbon; however, by using topsoil and amendments limitations of PG can be ameliorated. Using data from this study it appears that forestry plantations are meeting most reclamation objectives for PG stacks in a similar way to traditional grass cover methods.

Five soil amendments were used in a field experiment to determine effects on *Picea glauca* Voss. (white spruce) and *Populus balsamifera* L. (balsam poplar) growth. After two years amendments had little effect on either species for the parameters measured. The only significant effect of treatment was the control having significantly healthier *Populus balsamifera* trees. In general, the healthiest and most successful trees of both species were from the control treatment. Survival of all trees was very low relative to other studies with significant mortality of both species over the 2016 to 2017 winter in all treatments. At the end of the study *Picea glauca* had higher survival than *Populus balsamifera* for all treatments. Nutrient rich compost and manure increased soil nutrients whereas mycorrhizal fungi and the high carbon material, Black Earth, had no effect on soil properties. Despite efforts to control unwanted vegetation such as weeds and competitive grass, the use of amendments may have inadvertently benefited competing vegetation more than the planted trees.

Pure soil, PG and mixtures of both were analyzed for chemical composition and no elements were above guidelines for an industrial end land use. Aluminum, beryllium, chromium, copper, iron, magnesium, manganese, nickel and vanadium concentrations were higher in soil than in PG. Cadmium, calcium, fluoride, sodium and strontium concentrations were significantly higher in PG than in soil. Soils with PG and soil mixed in different proportions had values between pure soil and PG. Pure PG and soils containing high amounts of PG were above guidelines for naturally occurring radioactive materials for radium-226 and lead-210. A strong correlation was found between radium-226 and potassium-40 in soil and PG mixes.

Measurements of radon-222 gas, a daughter product of radium-226, were higher at Nutrien than at the Ellerslie control site. Radon-222 emissions from the PG stack may have been underestimated due to wind and other environmental factors. Variability in radon-222 emissions were seen in the different vegetation covers, likely due to canopy structure, not uptake or changes from plants growing on the site.

Element concentrations of different types of vegetation growing on PG were compared to the same species growing at the Ellerslie control site. Plant tissue concentrations differed significantly with vegetation type but not within similar species. Grass typically had lower concentrations of elements than woody species. Washing tissue samples resulted in few differences relative to unwashed. Despite differences in soil concentration, tissue concentrations for most plant species were similar at Nutrien and Ellerslie, suggesting that elements with high concentrations in PG were not in a bioavailable form. Element concentrations in tissue were below tolerable levels for most animals and likely have no negative impact. Vegetation contained very low concentrations of radium-226 and disproportionately high lead-210 activity, likely as a result of atmospheric fallout. Trace elements and isotopes in PG will likely not negatively impact plant productivity.

2. RESEARCH APPLICATIONS

The results of the amendment experiment can be used by other industries looking to reclaim waste materials using forestry plantations or that want to use soil amendments in conjunction with the use of topsoil. Although there were no effects of amendments on trees, some problems that arose during this study may help in planning future studies. This study is an example of how forest plantations can be successful on waste materials and by-products that may otherwise be capped with grass.

Analysis of PG in this study shows that it has benefits making it useful as a soil amendment. PG can be applied to soils deficient in calcium, phosphate and sulfate. No trace elements in PG appear to be a concern for PG application. Radionuclides in PG may limit application but if activity concentrations are known, it can be applied appropriately. Using the correlation of radium-226 in PG and potassium-40 in soil built from this study, environmental practitioners can determine suitable application rates of PG in agriculture and reclamation.

Methods used in this study are transferable to other PG stacks, but the results may be different on other PG stacks. PG from different sources contains different impurities and the age of the PG stack may affect mobility and bioavailability of some elements. This study was completed on a flat stack whereas most PG stacks have sloped perimeters that may influence soil water content, which could affect tree growth.

3. STUDY LIMITATIONS

The short time over which this study was conducted is one of its major limitations. Plant establishment on a new site is a dynamic process that will shift over time as plants mature and a plant community develops. Trees are slow growing species that may not respond to amendments within two years but could respond later in their life cycle. *Picea glauca* trees used in this experiment had variable morphology and vigour when planted which may limit results. Competing vegetation around trees may have reduced water, sunlight and nutrients to trees, resulting in early mortality which prevented an effect of amendments from being detected.

There was replication in this study, but it was still within a localized area of the entire stack. Trees in the control treatment had significantly poorer growth than other unamended trees on the stack that were not part of this study. It is possible that the portion of the stack that this study was established contained soil or PG that effected soil water dynamics or unmeasured soil properties which led to poor growth in all treatments. Field experiments offer more realistic growing conditions relative to greenhouse studies; however, uncontrollable weather and site variability can strongly influence results.

Vegetation analyses was only conducted on samples collected on a single date during the growing season and on only new growth of plants. The interpretation of overall risk from the results is limited since individual plant species can allocate different elements in different locations throughout the year and throughout their life cycle.

Radon gas flux from soil is variable based on weather and atmospheric conditions. Radon canisters only give an average of radon emissions over the sampling time. This limitation prevents a detailed assessment of high and low fluxes which could affect risk determination. Canisters used in this study are likely not in a position to accurately detect radon emissions since radon is heavier than air and may not reach a detector above the soil surface.

4. RECOMMENDATIONS FOR FUTURE RESEARCH

Although short rotation forest plantations were successful on PG in the first years of development, it is a relatively new approach from traditional grass cover. The main objective for traditional PG cover systems is to isolate PG from the surrounding environment. This study has investigated components of soil and vegetation, however, there are still other aspects to assess before informed recommendations can be made. Forestry plantations are established to maximize tree production and therefore more research into different species and soil building techniques may benefit maximization of capital returns.

Altering proportions of amendments and changing soil depths in future studies may result in a greater effect of amendments. Utilizing waste materials from other industries as amendments is beneficial since they are typically less expensive than pure topsoil caps. Adjusting the depth of the surface soil cap may be beneficial as trees get larger and water requirements change. Results from this study suggest that grass may not be the best erosion control approach for forest plantations since it can provide unnecessary competition to trees. Effective vegetation control directly around each tree is necessary, particularly when trees are young and are competing for resources. Effectiveness and cost of chemical and mechanical controls of vegetation should be compared in future studies.

This study was not able to clearly determine the effect of trees on soil water dynamics with the same detail that has been done for grass cover in previous studies. Water content in the root zone and at different depths in the PG stack should be measured to determine if reclamation objectives are met. Bare soil between tree rows may affect runoff quality and should be assessed in future studies.

Radon-222 assessment on PG stacks could be conducted with real time measuring devices that can be repeated throughout the growing season. This would allow for better detail on fluxes of radon from soil and PG to make a more informed determination on total radon emission and risk. Vegetation around the site could be analyzed for lead-210 activity to determine if radon-222 is being emitted from PG and blowing off site, decaying into lead-210, and being deposited as fallout. Uptake of isotopes from PG by vegetation could be more effectivity assessed in a controlled greenhouse or growth chamber experiment. Solar and atmospheric contamination of field samples make it unclear where the activity detected in this study originates.

Ultimately, long term monitoring of this study is necessary to determine if trees will continue to be successful on PG stacks since no long term studies are available. Comparing growth of trees in this study to natural sites may be an effective way to determine how successful forest plantations on PG are at offsetting fertilizer production costs. A cost benefit analysis would be an effective way to compare different reclamation approaches for PG stacks. Costs of different site establishment techniques, vegetation management, plant species used and soil inputs can be compared for their benefit in productivity over time as a stand matures.

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