ULTRAMAFIC TECHNOSOLS: METAL MOBILITY AND CARBON SEQUESTRATION IN RECLAIMED MINE TAILINGS

by

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Abstract

Ultramafic settings have garnered increased attention as sources of nickel (Ni), a critical component of sustainable infrastructure and computer hardware. Interest in ultramafic minerals is exemplified by FPX Nickel's Baptiste project, a large operation located outside of Fort St. James, BC. Ultramafic rock has the potential to be carbonated when exposed to sufficient CO_2, as the silicate anion within the mineral lattice is replaced with a carbonate group. This creates the exciting possibility of carbon sequestration through enhanced rock weathering. However, extraction from these settings leads to the generation of ultramafic mine tailings, a nutrient poor and heavy metal rich parent material. Its currently unclear what the optimal reclamation strategy is for these settings, and how reclamation might affect carbon sequestration. To test these questions, two randomized complete block design experiments were conducted. One experiment featured a modified leaching column design, which tested for trace metal mobility and plant growth response to reclamation treatments. The other experiment looked at how reclamation strategies affect the sequestration of carbon, with particular attention given to organic carbon occlusion and mineral carbonation. It was found that adding 12.5% compost by mass to the tailings significantly improved plant growth, and lead to the formation of mineral-associated organic carbon (MAOC) within one year. Chromium was the only trace metal that leached in quantities above the CCME guidelines for irrigation water, signalling a potential risk that requires further investigation. These findings will contribute to develop a reclamation protocol for ultramafic tailings across the world and potentially abet the Baptiste Project's goal of carbon neutrality.

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ANOVA	Analysis of Variance
BEC	Biogeoclimatic Ecosystem Classification
CCME	Canadian Council of Minsters of the Environment
CEC	Cation Exchange Capacity
CSSC	Canadian System of Soil Classification
DMF	Dry mass fraction
DOC	Dissolved organic carbon
EFL	Enhanced Forestry Laboratory
ERW	Enhanced rock weathering
ICP-OES	Inductively Coupled Optical Emission Spectroscopy
LMWOA	Low molecular weight organic acids
MAOC	Mineral-associated organic carbon
MAOM	Mineral-associated organic matter
MOAs	Mineral-organic associations
PCA	Principal Component Analysis
POC	Particulate organic carbon
POM	Particulate organic matter
RCBD	Randomized complete block design
RMF	Root mass fraction
SMF	Shoot mass fraction
SOM	Soil Organic Matter
TB	Total biomass
TC	Total Carbon
TIC	Total inorganic carbon
TN	Total nitrogen
TOC	Total organic carbon

UT	Unamended Tailings
WASI	Wet aggregate stability indices
WEOM	Water-extractable Organic Matter

Acknowledgement

Despite its length, the words on the following pages do not even begin to capture the amount of time, energy and spirit that went into this project. It required many hands, many minds, and many trips to the Home Depot.

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Finally, I want to dedicate this work to my parents – David and Esther DuBovis. Mom and Dad, thank you so much for encouraging me to be *me*. It is through that encouragement, that love and support, that I have the privilege of writing this acknowledgement. I am currently typing this while I hear you both happily retired in the next room, and I can't help but smile. I am so proud to be your son.

With all that out of the way, let's start the show.

Chapter 1

Introduction

Mining for strategic minerals for sustainable infrastructure represents a double bind. Material demands of renewable technologies and computer hardware necessitate the extraction of rare earth elements and trace metals (Calvo and Valero 2022). However, mining also exacerbates extant negative externalities, including habitat loss, waste rock generation, and carbon emissions. It's possible to mitigate these externalities through mine reclamation, but reclamation procedures vary depending on the context. This thesis will begin to address the reclamation of ultramafic mine tailings as generated by FPX Nickel's Baptiste project in British Columbia, Canada. While the Baptiste project is in early stages of implementation, the ultramafic minerals contain potential risks, as well as potential benefits, that require investigation into the suitability of reclamation strategies.

1.1. Context – The Baptiste Project

The Baptiste Project resides in the Decar Nickel District, a 24,700 hectare property located 90 km northwest of Fort St. James, BC (Grandillo et al. 2020; Figure 1). The area lies within five kilometers of Ruby Rock Provincial Park, situated on the unceded territory of the Tl'azt'en and Binche First Nations. Eight different Biogeoclimatic Ecosystem Classification (BEC) units (to variant) are found within the Baptiste Project, with Subboreal Spruce (SBS) and Engelmann Spruce-Subalpline fir (ESSF) zones making up the majority (Allnorth 2012a). The site initially garnered attention 1940s, but did not receive development until nickel (Ni) was noted at the turn of the 21st century (Grandillo et al. 2020).

The project area lies atop a section of a larger ophiolite sequence, known as the "Trembleur" ultramafite unit (Grandillo et al. 2020). Ophiolite sequences form from the uplifting oceanic crust, and often include ultramafic strata (Cannings et al. 2011). Ultramafic rocks in the area host awaruite [Ni_3Fe], which forms through the serpentinization of nickeliferous olivine (Grandillo et al. 2020). This alloy is the chief mineral of economic interest at the site. Awaruite's high density and strong magnetic character allows for extraction via repeated pulverization and magnetism, foregoing the use of chemical processing (Grandillo et al. 2020). In addition to awaruite, the serpentinization of pyroxene and olivine within ophiolite sequences creates mineral serpentine and magnetite (Grandillo et al. 2020). Thus, after the awaruite is extracted, the tailings contains high amounts of these minerals, which imparts a high Fe and Mg content (Power et al. 2019).

The provincial government of British Columbia requires mineral property holders to return the site to "prior capability" (BC MEMPR 2010). Per these regulations, FPX Nickel intends to keep the tailings behind a dam on site (*Figure 2*), with the deposition beginning twenty-two years after extraction begins (Grandillo et al. 2020). While the dam keeps the tailings contained, the ultramafic minerals create soils of low fertility (2012b). Furthermore, the extant soil at the Baptiste project display arsenic (As), cobalt (Co), chromium (Cr), copper (Cu), and Ni in exceedance of CCME Park guidelines (see Table 1-1). Thus, returning the site to prior capability requires introducing plant available nutrients and mitigating the effects of toxic heavy metal abundance.

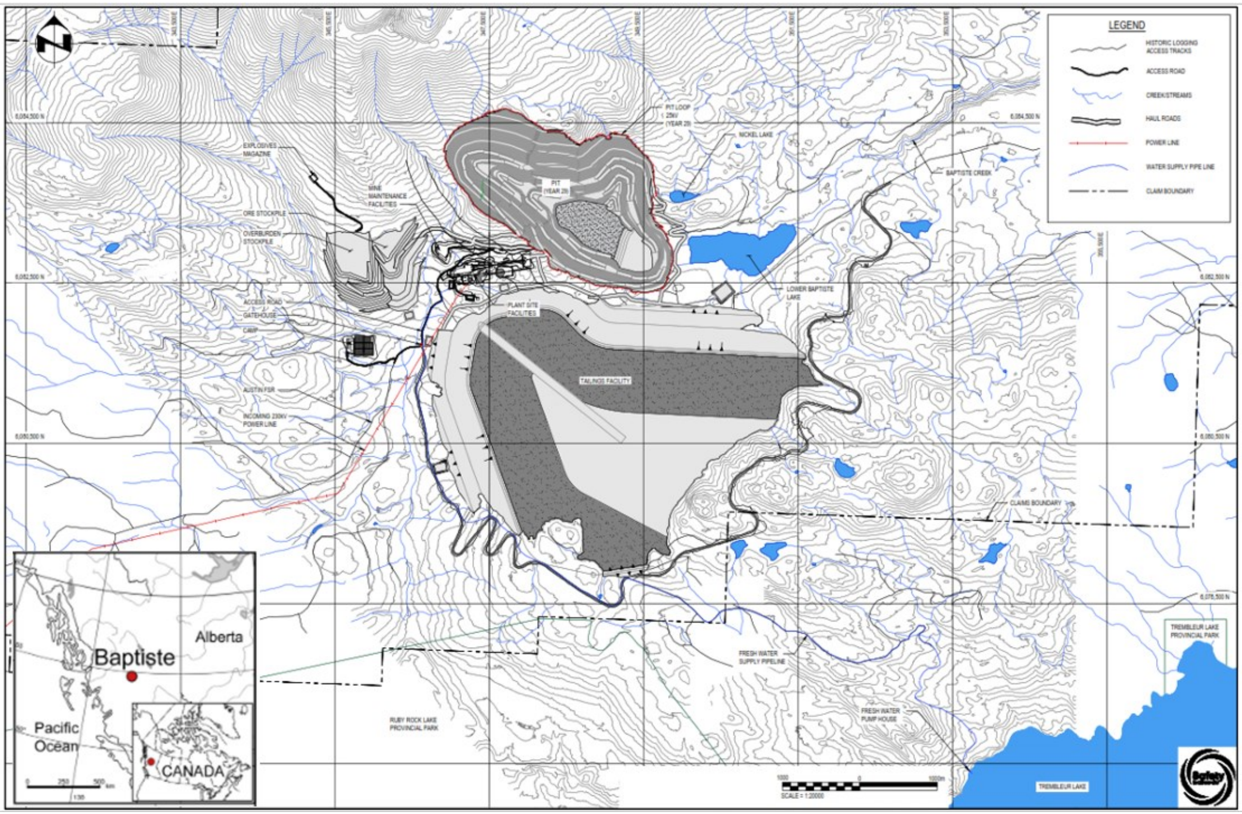

Figure 1. The site plan and infrastructure at the Baptiste Project. The tailing facility is below the pit and shows the ultimate extent of the facility. Image from Ausenco Engineering Canada Inc. 2023.

Table 1. Number of soil samples in exceedance of CCME park guidelines at the Baptiste project, adapted from Allnorth (2012b). 18 sites were chosen for metal analysis, sampled at the three layers contained within the labeled columns.

Metal	Criteria (mg/kg)	LFH	0-20 cm	20-40 cm
As	12	0	2	4
Cr	64	1	16	17
Co	50	0	3	5
Cu	63	0	1	4
Ni	50	1	15	18

1.2. Mine Reclamation

Mine tailings host many negative externalities for environmental and human health, including heavy metal toxicity, methane generation, and acid drainage (Sun et al. 2018; Xu et al. 2019). The extensive legacy of these externalities has led to increased pressure on mining operations to be proactive in their risk mitigation. Thus, a large body of literature has been amassed to assess strategies to address these risks. Most of the mine reclamation research has focused on remediating metalliferous pyritic tailings (whose oxidation leads to acid mine drainage), lignite deposits, and bauxite waste rock (Foght et al. 2017; Sun et al. 2018). The focus on these substrates reflects their extensive historical legacy and severe hazards they impose. By contrast, ultramafic (or 'serpentine') deposits have, until recently, been limited in their economic utility, leading to a dearth of literature on their reclamation. As exemplified by the Baptiste Project, ultramafic deposits are rapidly becoming areas of interest, as they host a wealth of strategic heavy metals such as nickel (Ni) and chromium (Cr) (Power et al. 2019). It is projected that utilization of ultramafic deposits will lead to the generation of approximately 419 Mt of tailings annually, creating an urgent need for research into their reclamation (Power et al. 2019). Thus, reclamation researchers are left to address a growing risk embodied by ultramafic tailings with techniques developed for tailings of vastly different mineralogy.

The ubiquitous risk of heavy metal contamination from mine tailings has made it a subject of immense scrutiny within the literature. Capping the tailings with salvaged soil has been the primary means of addressing the risk of metal uptake by native vegetation (Sun et al. 2018). While this practice abets the return of native plants, the high cost of collecting and storing the soil makes the practice rather prohibitive (Sun et al. 2018). Soils from the Baptiste site are fairly young in age, constituting Brunisols dominated by the translocation of dissolved organic matter with iron and aluminum (Bergh 2012b). The relatively young age of these soils might pose challenges for their salvage, as their lack of weathering makes them more sensitive to changes incurred through the salvage process. To avoid the potential costs and pitfalls of the soil salvage approach, I propose the Baptiste project can be returned to prior capability through the construction of soil from the tailings – a *technosol.*

1.2.1. Technosols

Recently, researchers in reclamation have called for increased emphasis to restoring soil development on mine tailings (Li and Huang 2015). Under this approach, organic amendments mixed with tailings constitute a novel parent material, which weathers through the action of biota and climate to create a novel soil type (Huot et al. 2013). Blending these two artifacts together satisfies the International Union of Soil Scientists' definition of a *technosol* – a soil derived from industrial or artisanal artifacts (Huot et al. 2015). Technosols have been noted to share similar pedogenic transformations as those of in-natura soils, but their extremely heterogeneous composition leads to the co-production of transformations that are rarely observed in their natural counterparts (Leguédois et al. 2016). Within the technosol framework, vegetation becomes an agent of transformation within the soil system, rather than the end-goal of reclamation. My study seeks to apply this system approach to understanding the effects of organic amendments and revegetation to the pedogenesis of ultramafic mine tailings.

Restoring the soil functioning of the ultramafic tailings will require application with organic amendments. Many of these amendments are derived from other waste streams, with the most common examples being compost, biosolids, pulp and paper sludge, and biochar (Larney and Angers 2012). In addition to enhancing the nutrient content of other oligotrophic minerals, organic amendments have been shown to stabilize the release of heavy metals through the amelioration of substrate pH and adsorption of metal cations to organic ligands (Kumpiene et al. 2008). However, organic amendments vary in their efficacy to address all these constraints presented by tailings and pose a risk of secondary pollution depending on the process responsible for their production (Hargreaves et al. 2008). Both compost and biosolids, two of the most applied organic amendments, have been shown to release heavy metals of their own when overapplied (Hargreaves et al. 2008; Asemaninejad et al. 2021). Boiler and fly ash often contain extreme alkalinity and toxic heavy metals depending on the source material and combustion process (Nunes et al. 2016). Thus, discerning the optimal application rate of organic amendments to tailings is important for minimizing the risk of secondary pollution, while maximizing the benefits conferred by amendment application.

In addition to organic amendment application, technosols at the Baptiste site would benefit from active revegetation. Like with organic amendment application, revegetation poses potential benefits and risks for returning prior capability. Revegetating with native plants offers the opportunity to rapidly return ecosystems services to the site, a long sought after goal of mine reclamation and restoration (Bell 2001). In addition, the action of plant roots and microorganisms within the soil are integral for soil structure formation, which can help to reduce erosion and improve infiltration of water to deeper horizons (Bronick and Lal 2005; Poirier et al. 2018). However, the action of plant roots and soil microorganisms might have a contradictory effect on mineral weathering – potentially posing risks for technosol weathering. Root and microbial exudates drive mineral weathering (hereafter, bio-weathering), which has been shown to denude primary minerals of their heavy metal cations (Burghelea et al. 2018; Fang et al. 2023). In ultramafic contexts, certain plant species are Ni-hyperaccumulators, which allows them to absorb high amounts of Ni into their tissues (Chardot-Jacques et al. 2013). While the amount of Ni is greater in plant tissue than in the soil solution, the amount of Ni released in planted treatments tends to be greater than that released from abiotic weathering. So, while some advocate that plant roots can stabilize metal contaminants within the soil, the action of their roots can equally as likely lead to their more rapid dissolution into the soil solution. Thus, one of the primary objectives of this study will be to assess the impacts of organic amendments and revegetation on technosol formation.

1.2.2. Carbon Sequestration

Carbon cycling is integral to the biogeochemistry of soils, and the pooling of carbon within technosols is an important component of their legacy. In the case of inorganic carbon, the Baptiste tailings have been extensively observed for their potential to be artificially carbonated (Power et al. 2019). The recalcitrance to volatilization of inorganic carbon has garnered interest as a climate mitigation tool (Sanna et al. 2014). Enhanced rock weathering (ERW) refers to a suite of technologies that seek to transform labile primary minerals into carbonated varieties, with the goal of enhancing the carbon sequestration potential of soils (Cong et al. 2024). Ultramafic minerals, such as brucite and serpentine, have been observed to carbonate when exposed to CO_2, making them interesting prospects for minerals to be used in ERW (Power et al. 2017). However, the carbonation of these minerals has only been observed in highly controlled conditions, making it difficult to discern their carbonation potential when deposited in-situ. Furthermore, plant-driven bioweathering has not been observed in concert with mineral carbonation, which means it’s difficult to discern the stability of derived carbonates when exposed to plant root exudates. While microorganisms have been shown to abet mineral carbonation, these are again only observed in highly controlled circumstances (McCutcheon et al. 2016). Thus, understanding how ultramafic mineral carbonates fair in more microbially heterogeneous settings is an unknown within the literature. Both plants and microorganism release CO_2 within the soil as a part of their respiration, so it is just as likely that this CO_2 might react with the labile minerals within ultramafics to create Mg-carbonates (Power et al. 2021).

Considering that mineral carbonation stands as one of the shared transformations between technosols and natural soils, measuring the formation of pedogenic carbonates is integral to understanding the pedogenesis of ultramafic technosols (Leguédois et al. 2016). In addition to deepening our understanding of technosol pedogenesis, measuring inorganic carbon offers an important means of offsetting the carbon emissions of the Baptiste project.

In addition to inorganic carbon sequestration, ultramafic technosols have the potential to sequester organic carbon by providing conditions for biomass accumulation, accumulating particulate organic matter (POM), and forming mineral-organic associations (MOAs) and macroaggregates. MOAs (also referred to mineral-associated organic matter or MAOM) refers to polar organic ligands that bind to reactive Fe-Al oxides and clay particles in the soil (Kleber et al. 2015). Recent advancements in organic matter dynamics have shown that the rate at which organic matter oxidizes to CO_2 is determined by accessibility of residues and biopolymers to microorganisms – a view dubbed the 'Soil Continuum Model' (Lehmann and Kleber 2015a). Within this framework, adsorption of biopolymers to mineral surfaces and aggregates constitutes the primary means of occlusion from microorganisms (Lehmann and Kleber 2015a). This mechanism recognizes that there are two types of organic matter within the soil: particulate organic matter (POM), which is free available for microbial consumption, and mineral-associated organic matter (MAOM), that which is adsorbed to mineral surfaces (Cotrufo et al. 2019). While certain microbial and plant exudates have demonstrated the ability to liberate MAOM, this process requires more energy than decomposing more labile POM, meaning that enhancing organic carbon sequestration within the soil should maximize the amount of mineral surface area available to ligand adsorption (Li et al. 2021). MAOM formation differs depending on the degree of chemical weathering and pH of the soil; more acidic and highly weathered soils demonstrate MAOM formation in conjunction with Fe/Al oxides (Kleber et al. 2015). By contrast, circumneutral and alkaline conditions, like those observed in newly deposited ultramafic minerals, rely on polyvalent cations (such as Mg^{2+} and Ca^{2+}) to form 'cation bridges' between organic ligands (Rowley et al. 2018a). While specific studies observing POM/MAOM fractions within ultramafic soils are limited, evidence suggests that Mg-bearing minerals within ultramafic soils form weak outer-sphere complexes with organic ligands (Falsone et al. 2016). Over time, the primary minerals found within ultramafic parent materials weather to become phyllosilicates and metal oxides such as smectite and goethite, which have a much greater capacity for mineral adsorption (Alexander 2020; Ruiz et al. 2023). Currently, there are no available materials on the POM/MAOM fractions found within amended ultramafic mine tailings, meaning that reclamation projects in these settings cannot accurately account for organic carbon sequestration potential.

Mineral and organic matter dynamics at the microscale, coupled with the action of plant roots, lead to the formation of aggregates at the more macroscale (Weil and Brady 2016). Aggregates are the principal components of soil structure, and their shape and size determine many aspects of soil functioning. Regarding organic carbon occlusion, aggregates create sites of anoxia within the soil, leading to slowed decomposition of organic matter (Keiluweit et al. 2017). Thus, aggregate stability, the resistance of aggregates to wind and water erosion, holds great importance for understanding soil carbon dynamics. Few studies have observed aggregate stability in technosols, and even fewer still in ultramafic contexts.

The coupling of inorganic carbon and organic carbon within the Baptiste technosols has the potential to greatly offset the lifecycle emissions of the mine. FPX Nickel has accounted for the operating emissions of the mine through the use of hydroelectric power and lower-carbon haulage technology (Power et al. 2019; FPX Nickel 2021) These measures already significantly reduce the emissions associated with the Ni extracted from the Baptiste Project. Demonstrating further sequestration potential through the construction of technosols has the potential to make the Baptiste Project the first carbon-neutral mine in the world.

1.3. Mine Reclamation

Taken together, this project seeks to observe the interplay between amendments, vegetation and ultramafic minerals, and how that interaction mobilizes metals and sequester carbon. Achieving this requires the application of a systems thinking approach, which helps to understand how each extant component interacts to create an emergent entity (Arnold and Wade 2015). The emergent entity, in this case, is the technosol the components of interest are vegetation, microbes, organic matter, and ultramafic minerals. I developed two experiments to answer four different research questions developed through this framework.

The first question looks at the effects of organisms on mineral and organic matter weathering. *How does bio-weathering change mineral dissolution within the ultramafic technosol system?* As previously described, plants and microbes can denude metals from their mineral lattice. Thus, I hypothesize that revegetation will lead to the disproportionate mineral dissolution, which will lead to a change in metal concentrations within the soil solution. Conversely, *how does the rate of organic matter application change plant biomass generation?* Given that organic amendments tend to introduce plant nutrients into oligotrophic tailings, I hypothesize that a change in organic matter application rate will lead to a change in plant biomass generation compared to unamended tailings. These questions were tested through a modified leaching column experiment, whose results are described in Chapter 2. The findings of this experiment will discern optimal organic amendment application, which inform implementation costs of technosol construction. Furthermore, these findings will help reclamation planners understand the benefits and risks associated with different approaches to technosol construction.

The second experiment details the change in soil carbon within ultramafic technosols. More specifically, *how does the rate of organic amendment application affect mineral carbonation rates?* Given that mineral carbonation relies on mineral exposure to CO_2, I hypothesized that a change in organic amendment application rate will a change in the rate of mineral carbonation. Lastly, *how does the rate of organic amendment application affect organic carbon occlusion?* MAOM formation and aggregation rely on the mixture of minerals and organic matter, but the optimal proportions that these processes require are highly context specific. I hypothesize that a change in organic amendment application will lead to a change in carbon occlusion within the ultramafic technosols, as discerned through aggregate stability and MAOM formation. Both these questions will inform stakeholders of the long-term carbon sequestration potential of chosen reclamation strategies, and details how to most optimally maximize both pools of carbon. If mineral carbonation within the tailings is coupled successfully with organic carbon occlusion, this has the potential to make the Baptiste project a carbon-neutral, or even carbon negative, mine. I tested these hypotheses in an outdoor mesocosm experiment, which is detailed in *Chapter 3.*

The construction of ultramafic technosols represents a viable alternative to more traditional storing and capping approaches. These novel soils have the potential to support vegetation, stabilize toxic heavy metals, and sequester carbon in various forms, making them a multifunctional solution to problems posed by unamended tailings. Creating these novel soils can satisfy regulatory requirements for prior capability, while providing the added value of carbon sequestration. Outside the context of the Baptiste project, this study will address the knowledge gaps associated with ultramafic mine tailing reclamation, which will help to address the externalities associated of an ever-expanding industrial byproduct.

Chapter 2

Plant growth performance and mineral dissolution on ultramafic technosols

1. Introduction

The *Health, Safety and Reclamation Code* for mines in BC stipulates that the owner of a mineral deposit must ensure that land capability of is restored to conditions comparable pre-extraction. While capping with the pre-existing topsoil has been a common method for restoration of prior capability, there has been increased interest in the development of 'technosols' - soils created through the mixture of different industrial artifacts (Leguédois et al. 2016; Watkinson et al. 2017; Jayapal et al. 2022). While this method shows promise for alleviating logistical and infrastructural constraints for reclamation, there are still unanswered questions regarding implementation and risk assessment. The objectives of the following study will focus on addressing these risks and logistics. More specifically, within the context of the Baptiste project, this study will assess:

1) What is the optimal organic amendment application rate for plant growth on ultramafic tailings?
2) Does plant-driven bio-weathering lead to a change in metal mobility within ultramafic technosols?

For the first question, I hypothesize that the proportion of organic matter will change the growth of plants in association with ultramafic tailings. Technosol construction relies on finding the application rate of the chosen organic amendment that both maximizes plant health and minimizes implementation costs. However, results about application rate are conflicting, with some studies reporting greater plant biomass with increasing proportion of organic amendments, while in others, the effect appears minimal (Sarathchandra et al. 2022; Ball 2023). Findings from this research will provide an estimate of the target amendment application for optimal revegetation. Municipal compost and bio-ash were chosen due to their scale of production, widespread usage, and contrasting characteristics (Larney and Angers 2012; Jayapal et al. 2022). In preliminary trials, ryegrass (*Lolium penne*) demonstrated greater biomass when sown in ultramafic tailings mixed with compost compared to biosolids and peat moss (Ball 2023). However, bio-ash was not tested in this trial. Incorporating ash into this trial will offer the opportunity to observe both how application rate (as described by proportion) and amendment characteristics interact to change plant growth.

For the second question, I hypothesize that the presence of plants will lead to a change in mineral dissolution rates, which will lead to a change in metal mobility. Technosols are constructed with the purpose of improving revegetation of mine tailings sites. However, plants and their associated micro-organisms play an important role in primary mineral weathering, and have been shown to denude trace metals from mineral lattices (Burghelea et al. 2018). In the case of ultramafic tailings, weathering of these deposits could lead to the leaching and accumulation of toxic heavy metals such as As, Cr, Cu and Ni (Palich and Geo 2012a). While common primary minerals have been observed, few studies have observed serpentine minerals. A study that observed chrysotile dissolution noted that plants accumulated more Ni than was leached, but the leached quantity in the vegetated samples were greater than the abiotic controls (Chardot-Jacques et al. 2013) Thus, attempts to introduce vegetation measures might exacerbate metal mobility within these settings.

To answer both the aforementioned questions, I developed a novel, modified leaching column experiment. Leaching columns are a standard method for assessing metal mobility and mineral dissolution in controlled conditions. They typically involve the placement of a substance within a column, which then undergoes elution with a solvent. (Thom et al. 2013; Sangiumsak and Punrattanasin 2014; Mitchell et al. 2018). My study departs from typical leaching column studies in that the target substance (in this case, the technosol) does not receive constant elution with a solvent. Instead, the technosols receive consistent watering, akin to that employed in typical container studies. In addition, this study further modifies the conventional method by assessing the effect of plant-driven bio-weathering on a suite of elements, rather than targeting one element or mineral (Chardot-Jacques et al. 2013; Yu et al. 2021). These specific changes are meant to simulate conditions closer to those of natural weathering. This study marks the first attempt to model incipient mineral weathering and plant growth on an ultramafic technosol.

2. Methods

2.1. Experimental Design

Ultramafic tailings were obtained from the Baptiste Project near Fort St. James, British Columbia, which lies atop the Trembleur ultramafite deposit. Municipal compost, containing primarily yard waste, was sourced from the Municipality of Prince George, British Columbia. Bio-ash was received from the CanFor Pulp and Paper Mill, Prince George, British Columbia, and contained a mixture of boiler and fly ash. Leaching columns (see section 2.1.1. for column details) were filled with a mixture of organic amendments and tailings. Organic amendments were mixed with the tailings in five mass to mass ratios (*Figure 2-1*). In addition, I included a negative control containing no amendments (hereafter, unamended tailings, or UT) and a positive control containing the amendment alone. To provide for a contrast between vegetated (biotic) and unvegetated (abiotic) treatments, half the treatments were seeded with *Vicia villosa* (“hairy vetch”). Columns were arranged within a randomized complete block design (RCBD) to control for potential differences temperature and light within the greenhouse space provided by the Enhanced Forestry Laboratory (EFL) at the University of Northern British Columbia (UNBC).

The greenhouse pod was kept at 25°C on a 12-hour photoperiod, from 6 AM to 6 PM. Hereafter, treatments will be referred to by their constitute organic amendment and technosols (e.g. compost technosols).

The experiment ran for three months, with leachate being collected at the start (week 1), midpoint (week 4), and conclusion (week 8) of the trial. At the end of the experiment, the columns were disassembled, with subsequent vegetation and bulk soil samples separated for analysis. Each treatment was replicated five times to account for stochasticity and statistical power. With two amendment treatments, two plant treatments, seven mass-to-mass ratios ratios, and five replicates, the total amount of columns constructed was 140 (2 X 2 X 7 X 5). However, both the amendment treatments share the same negative control, which brings the value down to 135 units.

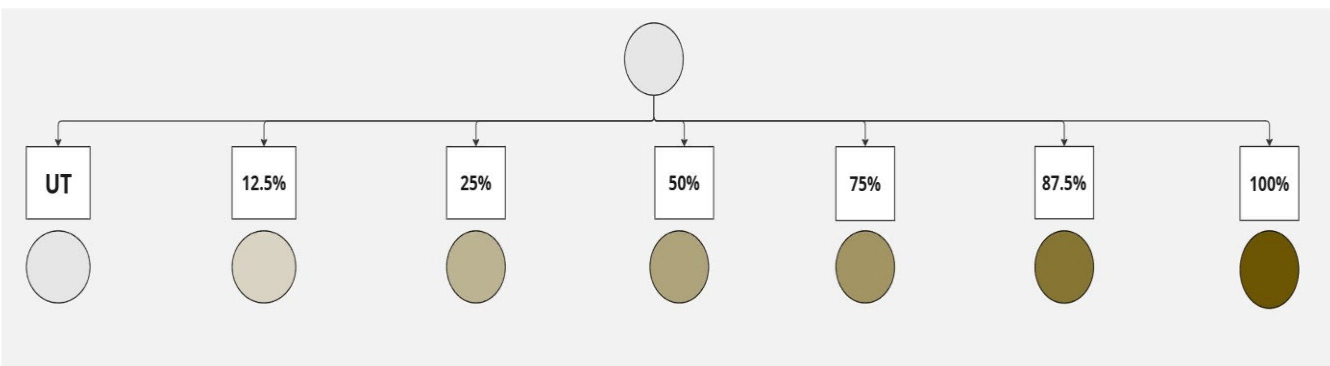

Figure 2. A schematic of the proportions of amendment to tailings assessed within the leaching column experiment. Ratios Percentages are read as amendment to tailings, with UT representing the unamended control.

2.1.1. Leaching Column Design

Columns were constructed using a polyvinyl chloride (PVC) pipe and a coupling (*Figure 3*). PVC is an inert substance that is employed in typical columns (OECD 2004); the white color reflects insolation, thereby reducing temperature flux. Together, pipe and coupling were 30 cm in height, with an internal diameter of 7.6 cm, giving a total volume of 1368.1 cm^3 (~1.37 L). A polypropylene weed barrier was fitted between the tube and the coupling to ensure that no coarse fragments drain within the leachate. The coupling was filled with rubber mulch to support the contents within the tube. Rubber mulch instead of the typical sand or gravel, as the latter run can potentially leach excess dissolved silica, confounding mineral weathering results. Each coupling had a hole drilled into its side and a propylene tube was inserted, through which the leachate was extracted using a plastic syringe. Finally, each column was set onto a catchment disk where any excess leachate would be collected.

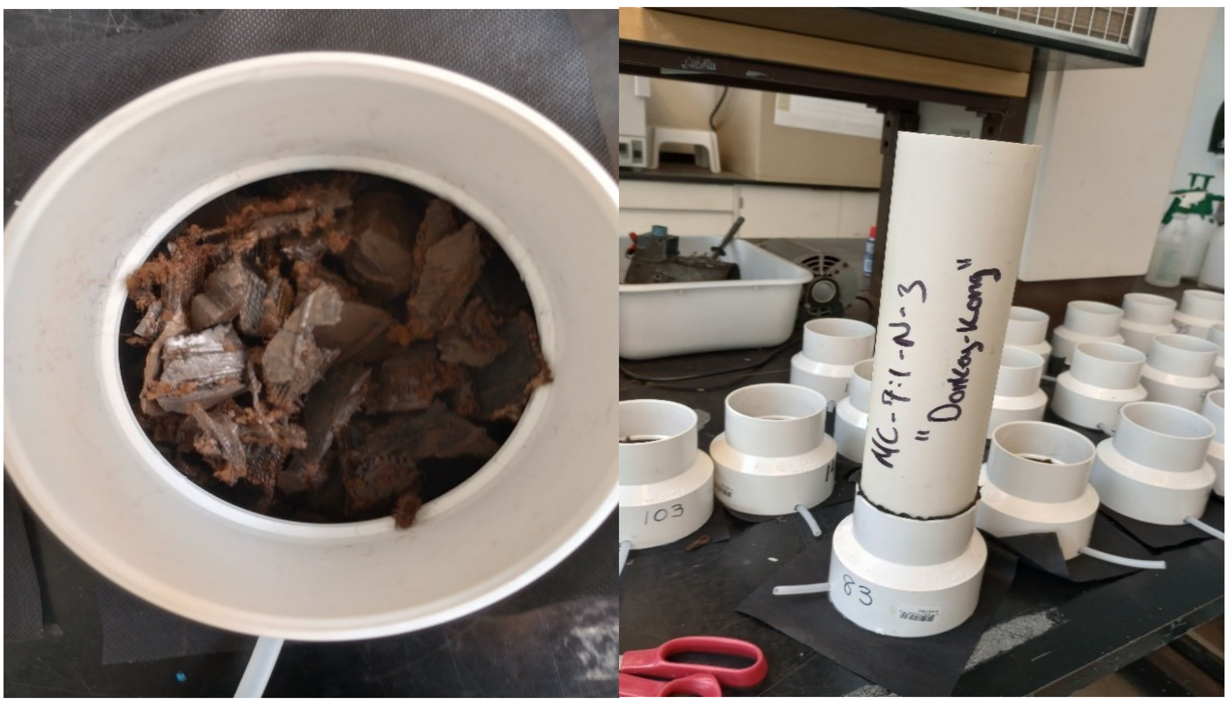

Figure 3. Assembly of the leaching columns. The left image shows the base of the column, formed by a PVC coupling, filled with rubber mulch. The right image shows the full column prior to substrate deposition.

After assembly, each column was filled with the predetermined amendment to tailings ratios on a *dry* mass to mass basis. To compensate for the different densities of the tailings and organic amendments, each treatment group had its own predetermined weight of substrate. Mixing of the technosols involved mixing the two "wet" components, whose respective masses were added based on the corresponding dry weight. This ensured that the mass-to-mass ratios were true to the dry weight of materials and excluded mass of water within the components. To determine the appropriate amount of wet component, I derived a dry mass fraction. Dry mass fraction (DMF) was determined using the following equation:

$$DMF = 1 - \frac{wet\ weight\ (g) - dry\ weight\ (g)}{wet\ weight\ (g)}$$

The DMF allowed me to determine how much wet mass to add to each treatment to achieve the desired mass to mass ratio. The precise mass of each component can be found in Appendix Table 1. Once the mass of each component was determined, they were manually mixed using an electric power drill fitted with a cement mixing bit and deposited into the column.

2.1.2. Plant selection and care

Half the replicates were seeded with *V. villosa,* a nitrogen-fixing pioneer species that has an endemic counterpart at the site of the Baptiste project (Palich and Geo 2012b). Its rapid growth and nitrogen fixing capabilities ensured reliable growth within a timeline fitting a short-term experiment. Columns assigned to the biotic treatment were seeded with four vetch seeds and then assessed for germination two weeks into the trial start.


Figure 4. Three vetch treatments in full inflorescence. This image was taken in August of 2023, within the last month of the experiment, and shows that the vetch had reached full maturity close to experiment end.

Columns were watered every other day with 200 ml of water. This struck the best balance between maintaining consistent moisture across columns without being overly cumbersome to apply. When leachate was to be extracted, the amount of water was increased to 500 ml to ensure there would be adequate leachate to collect, with collection occurring immediately after watering. Leachate samples were then refridgerated after collection before analysis (see *Section*

2.2.2. for more details). To reduce the variation introduced by seasonal insolation temperature swings, the columns received twelve hours of artificial sunlight provided by overhead grow lamps, and the temperature of the greenhouse was held at a constant 25°C.

To control spatial variability within the greenhouse, the columns were arranged within a randomized complete block design (RCBD) along an east-west gradient. Each block contained one replicate of each treatment unit, with each block replicated five times. The position of each unit varied randomly between blocks.

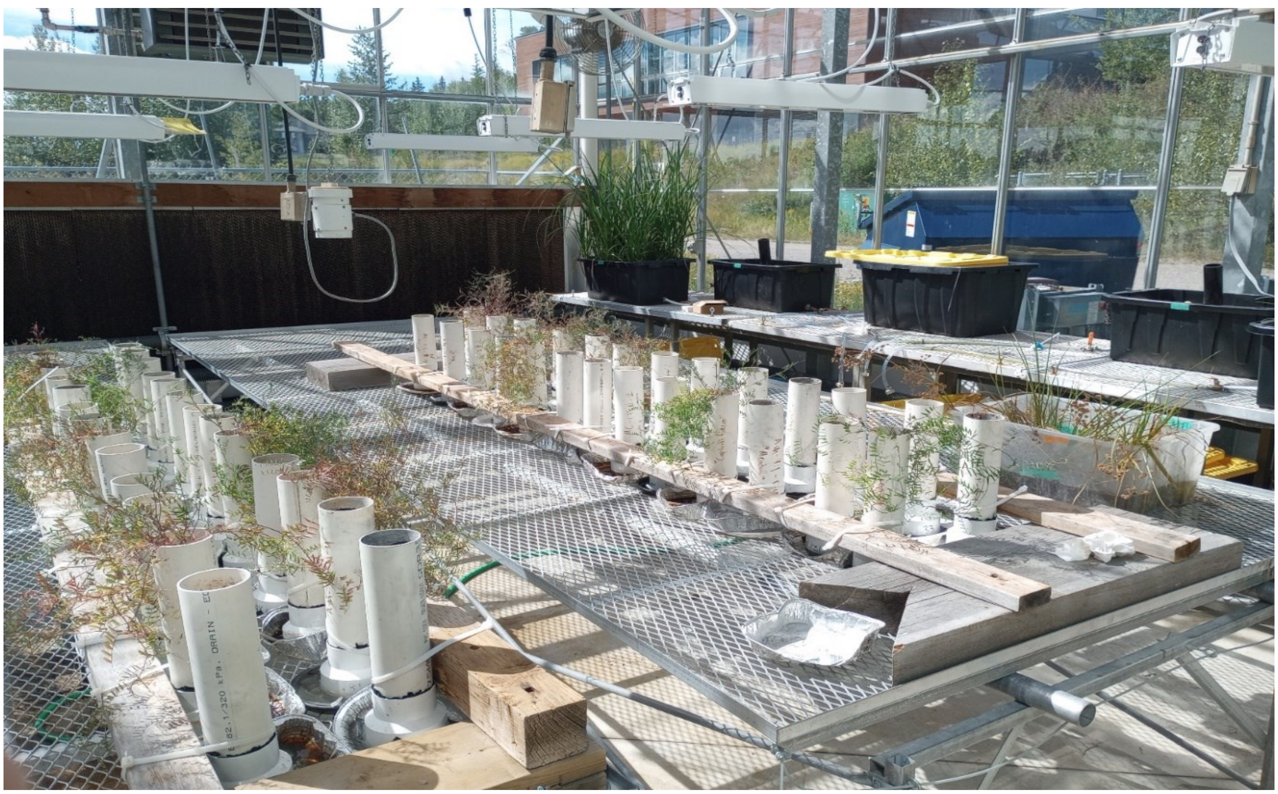

Figure 5. The randomized complete block design within the Enhanced Forestry Lab at the University of Northern British Columbia. Each block contained 27 columns, each containing a treatment group from the 27 assessed.

2.2. Data Collection

2.2.1. Plant metrics

Germination rate was determined by dividing the number of successes by four and then multiplying by 100. Once removed from the columns, shoots and roots were separated and subjected to separate preparations. Roots were separated from the substrate through washing, and then oven-dried at 60° C for 48 hours. Shoot samples were also oven-dried to a constant mass and oven-dried at 60° C for 48 hours. Dry biomass samples were placed into a weight boat with a predetermined mass. The mass of the boat was subtracted from the combined weight of boat and dry biomass, yielding the weight of the dry biomass.

Total biomass was determined through the combined mass of root and shoot. Shoot mass fraction (SMF) was calculated by dividing the shoot mass by the total biomass, and the root mass fraction (RMF) was determined by dividing the root biomass by the total biomass.

2.2.2. Leachate sampling

Three rounds of leachate were collected from the columns during the experiment: the initial (week 1), the midpoint (week 4) and the final (week 8). This provided for clear distinct points for the trial period, and provided discrete intervals to discern whether the plant growth affected elemental concentrations.

When it came to sampling the leachate, columns received 500 ml of reverse osmosis water sourced from the EFL. The water was allowed to drain through the column and then collected from a tube coming out the side of the column with a plastic syringe. Leachate was transferred from the syringe into a 50mL propylene Falcon tube and was promptly refrigerated at 4° C until further analysis could be conducted. Since all five replicates of each treatment were sampled three times, the total number of leachate samples collected amounted to 405 (135 X 3).

2.2.3. *Elemental analysis and pH*

Shortly after collection, pH was measured for all five replicates within each treatment. Each leachate sample was vortexed, and pH was determined with an Orion pH/ISE 420A probe (ThermoFisher Scientific, Waltham, MA). The probe was calibrated using controlled buffers (pH 4, 7 and 9) provided by Fisher Scientific.

Inductively coupled plasma emission spectroscopy (ICP-OES; Agilent ICP-OES 5100, Malaysia) was used to observe total concentrations of 26 elements (Al, As, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sd, Se, Si, Sn, U, V, and Zn). Total F^-, Cl^-, Br^-, NO_3^-, NO_2^-, PO_4^{-3}, and SO_4^{-2} were measured using anion chromatography (Dionex ICS-6000, Mexico/USA). ICP-OES was chosen based on unit cost per sample, but poses constraints on the resolution of trace elements. Mg and Si are common ions used to assess mineral dissolution within ultramafic studies, so Si was specifically included to approximate this process (Power et al. 2020). To prepare the samples for analysis, three leachate samples were randomly selected from each treatment and diluted by 500 times. Dilutions were made in acid-washed 50 mL Falcon tubes and delivered to the Northern Analytical Laboratory Services (NALS) at UNBC. The same three replicates were sent for ICP-OES and anion chromatography to ensure continuity between each round and minimize the risk of false correlations.

Cation exchange capacity was approximated by converting the total concentration of all metal cations within each leachate replicate from ppm to cmol/kg. Base saturation and the specific cation saturation indices were derived by dividing the cmol/kg of each base cation (Mg, Ca, Na and K) by the total CEC. CEC and base saturation were used to observe elemental flux at cation exchange sites, and estimate the degree of mineral weathering at each point of the trial where leachate was collected.

2.3. Statistical Analysis

All data collected from plants and leachate was subjected to Shapiro-Wilks tests for normality and Levene's test for homoscedasticity in the 'rstatix' package (Kassambara 2019). Where the data conformed to normality and homoscedasticity, factorial ANOVA was conducted, followed by Tukey's Post-hoc. For plant biomass data, a two-factor ANOVA was employed, with amendment and proportion used as fixed-effects. ANOVAs and Tukey Post-Hoc tests were conducted with the base functions in R 4.3.1. (R Core Team 2023). In the case of the leachate elemental fluxes, amendment, proportion, plant presence (biotic/abiotic), were treated as the fixed effects. The leachate data required a repeated-measures ANOVA, as time was also treated as a random-effect within the model. Repeated measures ANOVA and Tukey's post hoc tests were carried out with the 'emmeans' package within R (Lenth 2017).

Where data did not meet the assumptions of normality and homoscedasticity, one-factor Kruskal-Wallis tests were conducted followed by a Dunn's test. These tests were done with one factor (proportion, amendment, plant) per variable, so no interactions could be observed. The Kruskal-Wallis test was done using the base function in R, and the Dunn's test was conducted using the R package 'dunn.test' (Dinno 2014).

To discern the elements driving plant growth across treatments, elemental concentrations from each point were summed and then averaged across replicates. This data was then subjected to the same tests for normality and homoscedasticity as described above. The average sum of each element concentration was incorporated into a correlation matrix with shoot and root metrics; the correlations were assessed by deriving Pearson's coefficients in the 'rstatix' package in R (Kassambara 2019). This same data was then analyzed with Principal Component Analysis

(PCA) in R packages ‘factoextra’ and ‘FactoMineR’ (Husson et al. 2006; Kassambara and Mundt 2016). PCA allowed for further corroboration of trends observed in the correlation matrix and discern the clustering of units where applicable.

While all fixed effects were initially tested together, the amendments presented such contrasting characteristics that they often warranted their own independent testing. In such cases, all the previously described tests were conducted again, but amendment was removed as a fixed effect.

3. Results

3.1. Plant Growth

3.1.1. Germination

Compost technosols averaged 84 ± 3.88% germination success, with the unamended tailings averaging roughly the same (85 ± 6.11%), whereas the ash technosols averaged only 20 ± 5.67% success (*Fig. 6*). Ash technosols demonstrated significantly less germination success (X^2 = 36.378, $p < 0.001$) compared to compost technosols and the unamended tailings. Ash technosols were the only group that showed a significant interaction with proportion, with a significant decrease noted with increasing ash application (X^2 = 22.135, $p < 0.05$).

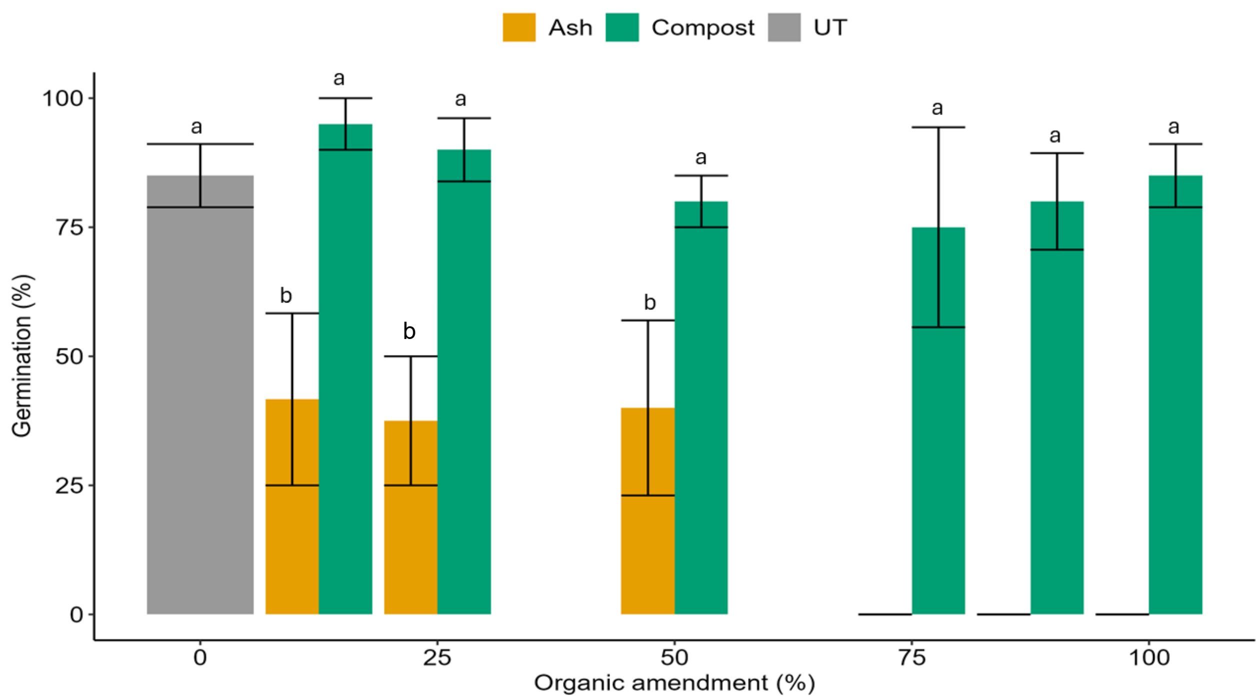

Figure 6. The germination success across organic amendment applications and amendments. Error bars represent ± SE, n = 3. UT connotes "unamended tailings" meaning these treatments did not receive any organic amendments.

3.1.2. Biomass

Amendment type was the only significant factor affecting total biomass generation, with the ash and unamended tailings demonstrating significantly less total biomass compared to the compost ($X^2 = 45.574$, $p < 0.05$). Total biomass of vetch specimens grown in compost technosols averaged fifty times greater than the unamended tailings and ash technosols (*Fig. 7*). Amendment proportion did not significantly interact with amendment type.

It became clear that ash was not an effective amendment for revegetation, so the following results will solely focus on the compost treatments. Within the compost technosols, shoot biomass was significantly greater than unamended tailings ($F_{1,33} = 16.133$, $p < 0.001$), but proportion of compost did not show any significant interactions. Within the compost technosols, shoot biomass peaked at 25% compost by mass (11.85 ± 0.94g). SMF did not significantly vary after reaching 25% compost by mass (81.1 ±0.03 %), with only a moderate increase at 75% compost by mass (*Fig. 8*). Root biomass did not vary significantly beyond the initial application of compost. However, RMF follows a trend that inverts that of shoot biomass, with the unamended tailings demonstrating the greatest RMF (*Fig. 8*).

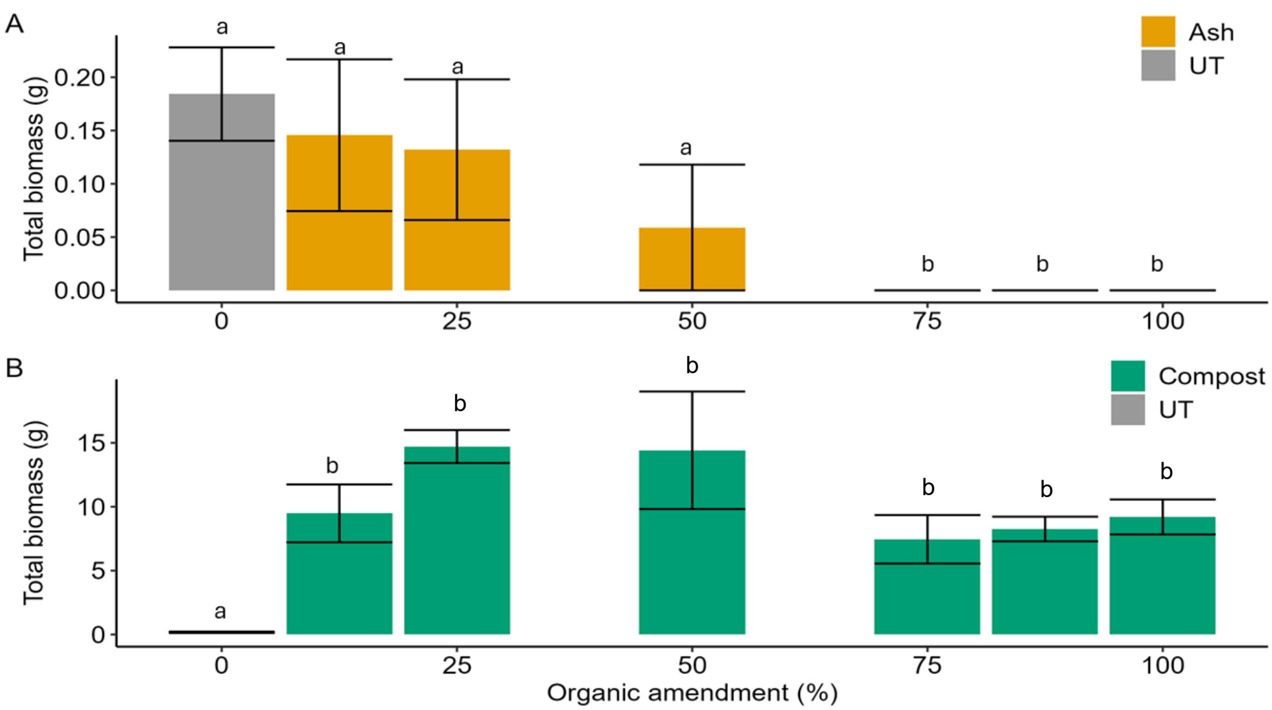

Figure 7. The variations in total biomass (root + shoot biomass) across amendment and proportion treatments. Error bars represent ± SE, n = 5. Plot A contains the total biomass of ash proportions, and Plot B contains the compost proportions.

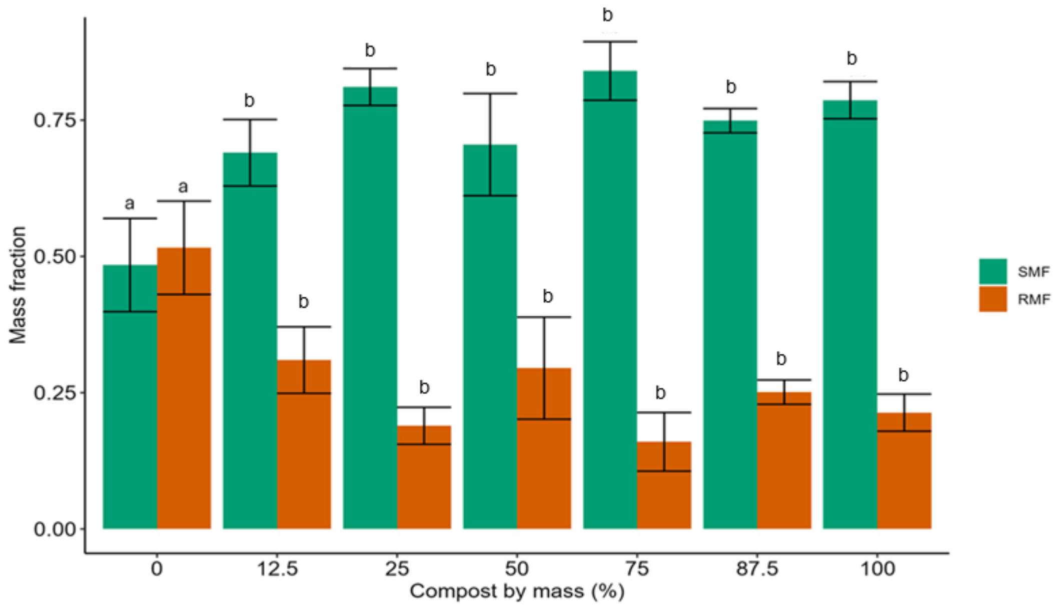

Figure 8. Differences in SMF and RMF across compost proportion. Error bars represent ± SE, n = 3.

3.1.3. Elemental concentration and plant growth

Since ash treatments did not demonstrate consistent plant growth, the results discussed in this section will only focus on the compost technosols. Total shoot biomass and SMF most significantly negatively correlated with the pH recorded at the last round of collection (R^2 = -0.65, $p < 0.05$ and R^2 = -0.63, $p < 0.05$, respectively). Total leached potassium and nitrate was positively correlated with total shoot biomass (R^2 = 0.43, $p < 0.05$ and R^2 = 0.44, $p < 0.05$, respectively). SMF was positively correlated with nitrate and phosphate concentration in the leachate (R^2 = 0.48, $p < 0.05$, and R^2 = 0.46, $p < 0.05$, respectively). SMF was significantly decreased with Mg leachate concentration (R^2 = -0.60, $p < 0.05$).

Proportion of compost by mass also negatively correlated with final solution pH (R^2 = -0.5, $p < 0.05$). Nitrate, phosphate and K showed strong positive correlations with compost application rate (R^2 = 0.69, $p < 0.05$, (R^2 = 0.95, $p < 0.05$). and R^2 = 0.94, $p < 0.050$, respectively). Mg concentration was negatively correlated with compost proportion ($R^2 = -0.70$, $p < 0.05$).

Total root biomass significantly correlated with total leached K ($R^2 = 0.47$, $p < 0.05$) and total leached sulfate ($R^2 = 0.59$, $p < 0.05$). This is in stark contrast with total shoot biomass, which demonstrated no significant correlation with sulphate at all (*Fig. 9*). Unlike other noted nutrients, total leached sulphate did not vary significantly between proportion of compost. RMF demonstrated the same relationships as SMF, but in the opposite direction (*Fig. 10*).

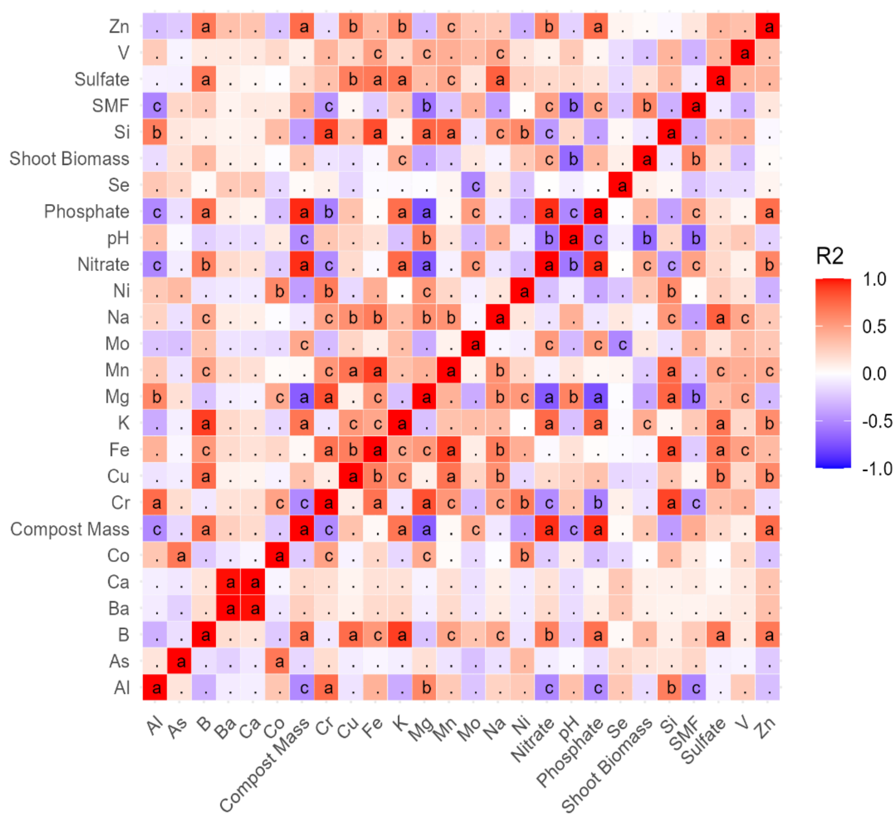

Figure 9. A heat map showing the correlations between total leached elements examined through the leaching column experiment and their relationship to shoot biomass and SMF. Letters denote significance of the relationship (*a* < 0.001, *b* < 0.01, *c* < 0.05, with anything greater than 0.1 marked with a ".").

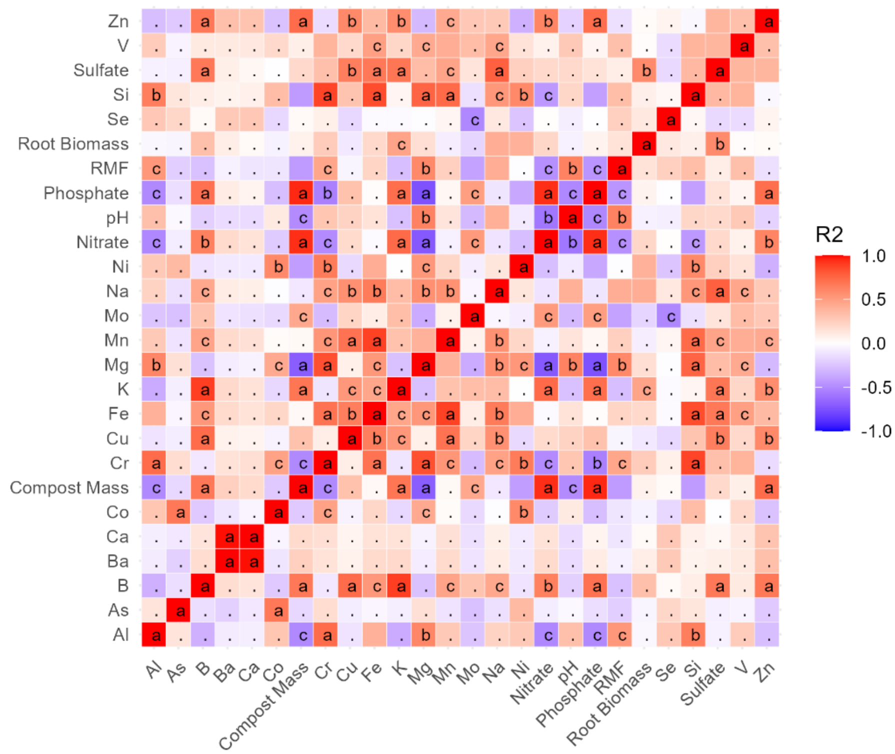

Figure 10. A heat map showing the correlations between total leached elements examined through the leaching column experiment and their relationship to root biomass and RMF. Letters denote significance of the relationship (*a* < 0.001, *b* < 0.01, *c* < 0.05, with anything greater than 0.1 marked with a ".").

The principal component analysis (PCA) revealed similar trends observed in the correlation matrices. Both dimensions of the PCA contributed roughly the same to the overall variation, with Dim1 accounting for 28.4% and Dim2 affecting 23.7% *(Fig. 11).* Plant nutrients (such as N, P, and K) increased with greater compost proportion, and contributed to about 26% of the variability within the model (~8% for nitrate and phosphate, and ~10.5% for K). Their increase corresponds to the clustering of technosols receiving greater than 25% compost by mass *(Fig. 5).* Both total biomass and SMF increased in tandem with the aforementioned nutrients. By contrast, base cations (mainly Na and Mg), trace metals (Cr and Ni), and other mineral components (Al and Si) were greatest in the lowest compost proportions and the unamended tailings *(Fig. 11).* RMF also strongly covaried with these groupings.

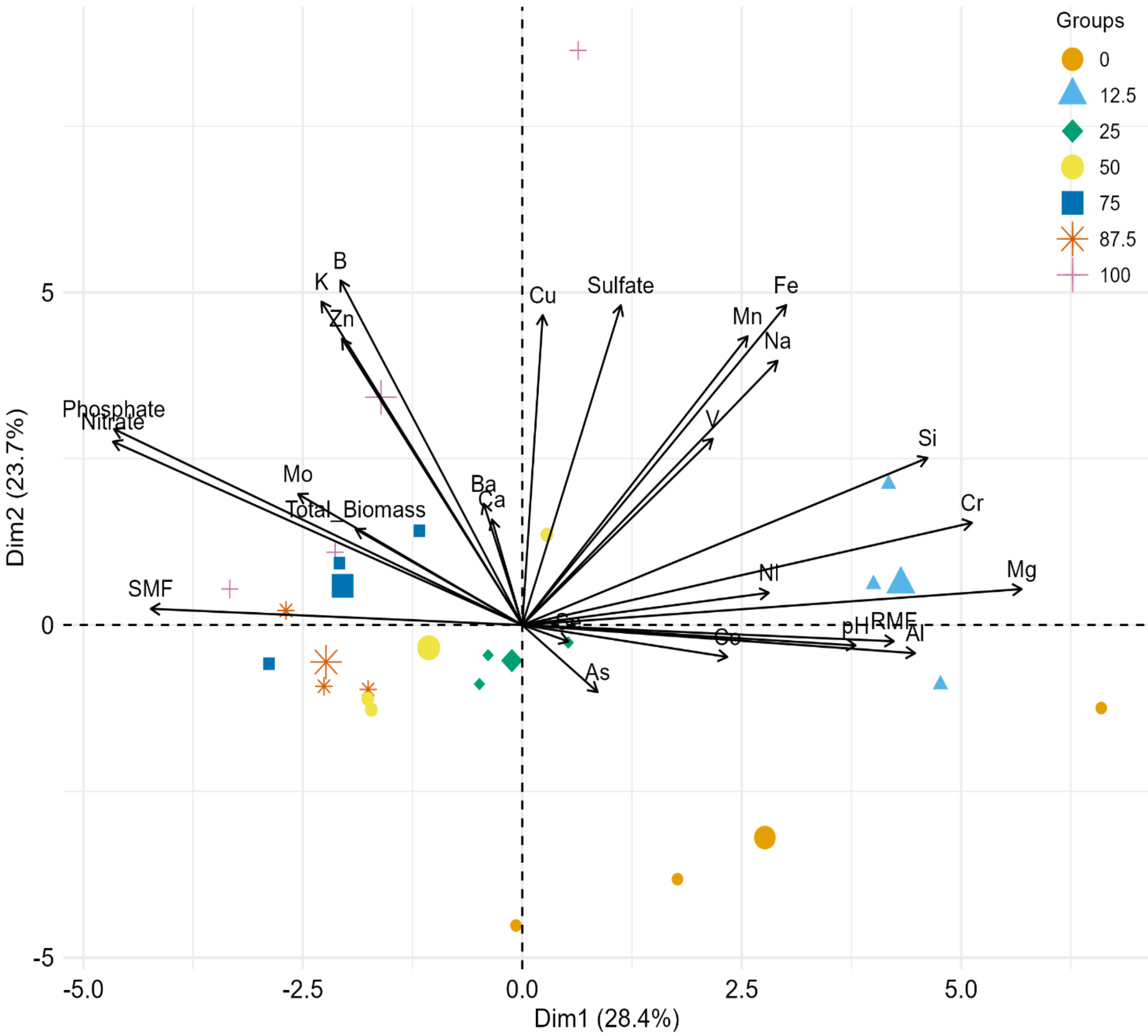

Figure 11. A biplot displays the PCA of the elemental data and the plant growth metrics. Elements and biomass data appear as eigen vectors whose length indicates their contribution to the total variation found within the data.

3.2. Mineral Dissolution

3.2.1. pH

Amendment proved to be the only significant treatment dictating pH variation. Ash technosols were significantly elevated compared to compost and unamended tailings across time ($F_{4,224} = 48.15$, $p < 0.0001$). However, ash technosols displayed consistently significant decreases over time ($F_{2,104} = 288.08$, $p < 0.0001$). Ash pH values were 13.42 at the start of the trial and then decreased significantly to 10.81 by trial completion. Compost technosols showed no significant variation over time, with their values being circumneutral through the course of the trial *(Fig. 12, Plot A).* Vetch was found to exert a significant influence on leachate pH in the unamended tailings themselves ($F_{1,11} = 33.78$, $p < 0.05$). Within the unamended tailings, pH significantly increased between week 1 and week 8 ($F_{2,11} = 27.05$, $p < 0.05$), being true for both biotic and abiotic technosols (*Fig. 12, Plot B*).

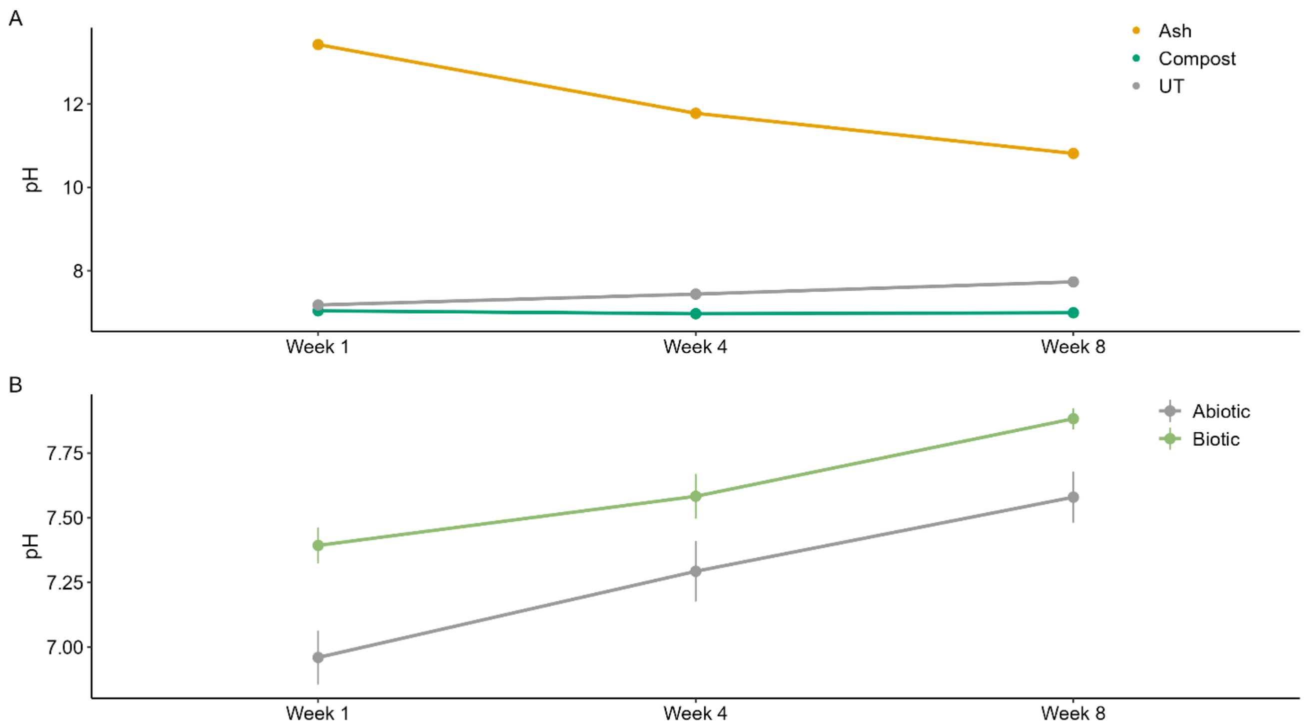

Figure 12. Change in pH for amendment treatments over time (Plot A). Error bars connote with ± SE, n = 36 for amendments, n = 6 for unamended controls. UT connotes with unamended tailings. Plot B shows the unamend tailing treatments. Error bars correspond to ±SE, n = 3.

3.2.2. Common mineral ions and cation exchange

Cation exchange capacity (CEC) showed distinct patterns for each amendment treatment over time. Amendment type proved to be the most significant factor, with ash technosols showing consistently greater CEC compared to the compost technosols and the unamended tailings ($F_{2,231} = 784.2$, $p < 0.001$). At the start of the trial, ash technosols' CEC were 13 times higher than compost technosols and close to 70 times higher than the unamended tailings, but these differences disappear by trial completion (*Fig. 13*). Compost technosols displayed CEC values that were significantly greater than unamended tailings across time ($F_{6,104} = 2.755$, $p < 0.05$), but proportion displayed no significant interactions. In addition, compost technosols CECs significantly increased between week 1 and week 4 ($F_{2,104} = 60.935$, $p < 0.001$), and continued to increase between week 4 and 8 but not significantly (*Fig. 13*). The unamended tailings also demonstrated significant increases between weeks 1 and 4 ($F_{10.62} = 9.667$, $p < 0.001$), and week 4 to week 8 ($F_{10.62} = 11.693$, $p < 0.001$).

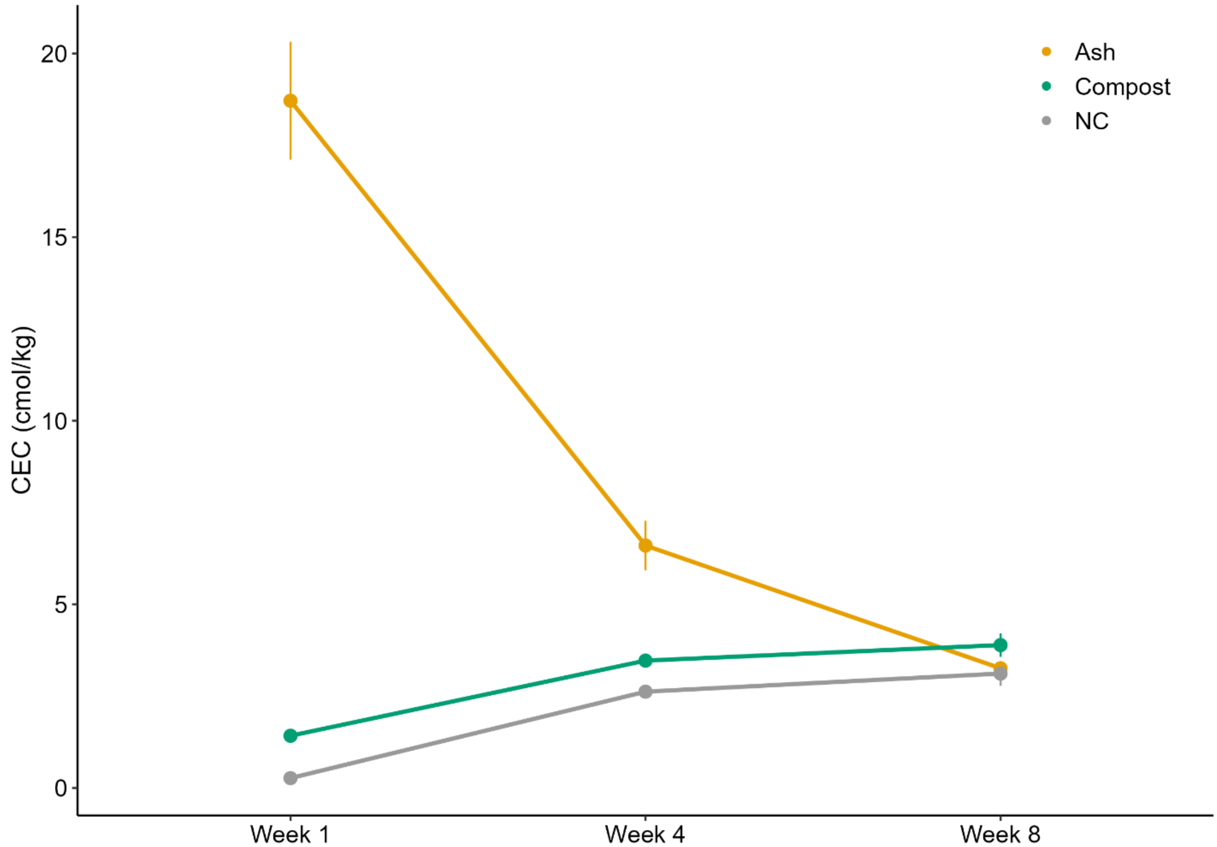

Figure 13. The changes in CEC over time across amendment treatments. Error bars represent ± SE (n = 36 for amendments, n = 6 for unamended tailings).

Calcium concentrations showed significant variations between amendment treatment ($X^2 = 19.1422$, df = 2, $p < 0.05$) over time ($X^2 = 161.5276$, df = 2, $p < 0.05$). Proportion and vetch treatment did not significantly affect calcium concentrations. Both amendment treatments showed elevated calcium concentrations compared to the unamended tailings at all points in time. All three amendment groups increased most significantly between week 1 and week 4, with calcium concentrations increasing by a factor of ten (*Fig. 14*). The increase between week 4 and week 8 was still significant, but the increase was more most compared to the change in week 1 and week 4.

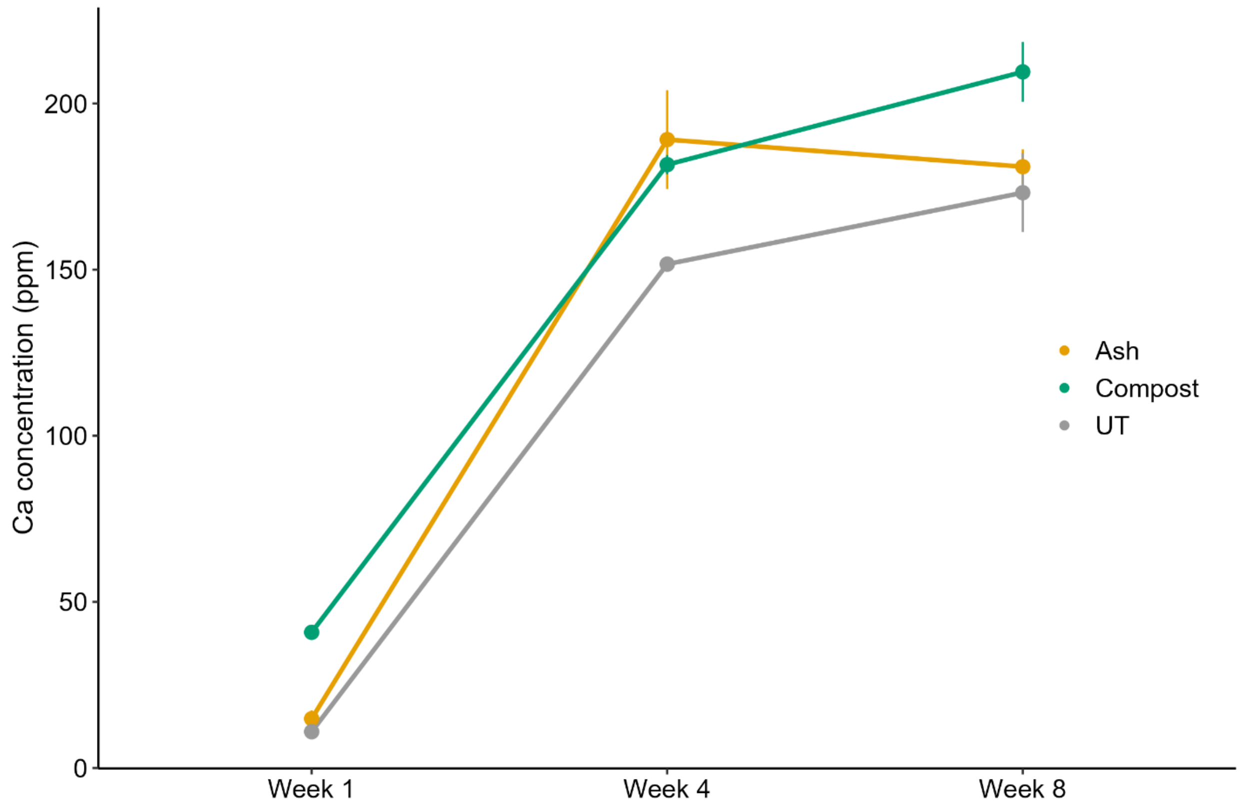

Figure 14. The changes in Ca concentration over time across amendment treatments. Error bars represent ± SE (n = 36 for amendments, n = 6).

Amendment proved to be the only factor dictating the concentration of Na within the technosols (X^2 = 155.6, df = 2, p < 0.05), with ash treatments showing significantly greater concentrations throughout the trial (*Fig. 15*). These differences are most noticeable in week 1, where ash treatments were nearly 50-70x greater than the unamended tailings and the compost technosols. However, the differences between the ash treatments and the other amendment treatments disappear by week 8. This highlights the role of time in the ash technosols; Na concentrations within the ash technosols significantly declined from week 1 to week 4 ($F_{2,123}$ = 48.11, p < 0.001). The Na concentrations within the ash technosols dropped by as much of a third between week 1 and week 4 (*Fig X.*). The drop between week 4 and week 8 was not significant.

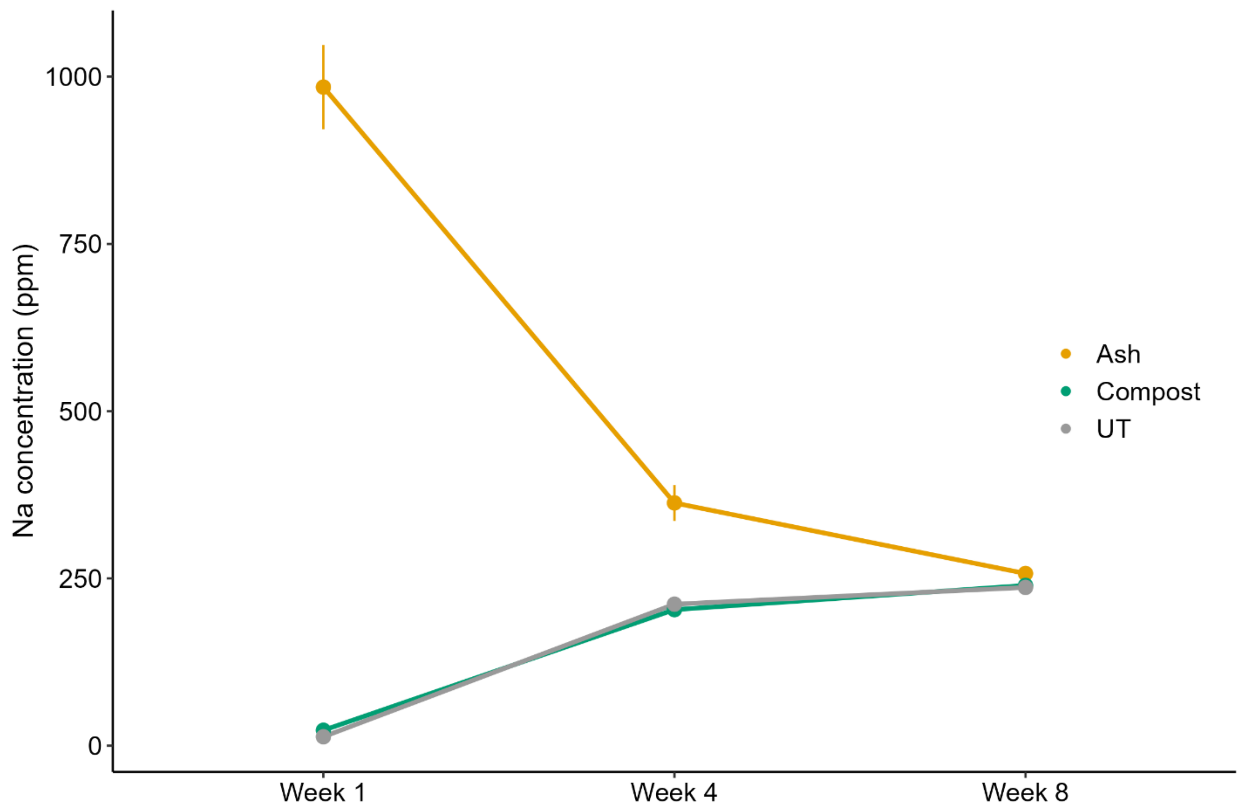

Figure 15. The changes in Na concentration over time across amendment treatments. Error bars represent ± SE (n = 36 for amendments, n = 6).

K concentrations varied significantly by amendment ($X^2 = 81.5379$, df = 2, $p < 0.05$), over time ($X^2 = 69.34$, df = 2, $p < 0.05$), with both amendments exhibiting greater K concentrations than the unamended tailings (*Fig 16.*). Ash technosols displayed K concentrations 17x those of compost technosols and 320x those of the unamended tailings at week 1. However, much like with Na concentrations, K concentrations reach the same levels as the compost technosols at week 8. K concentrations within the compost technosols remain largely the same, with a slight decline between week 4 and week 8. Proportion and plant showed no effect on K concentrations.

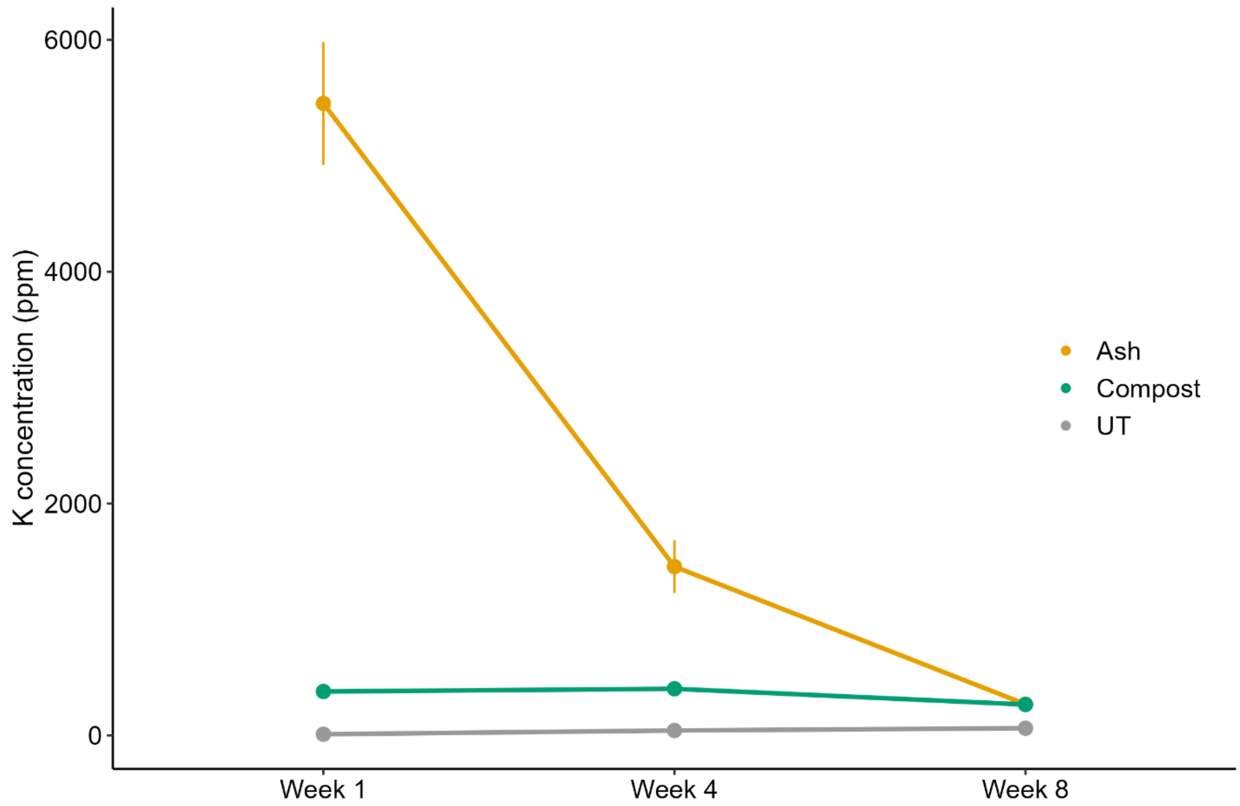

Figure 16. The changes in K concentration over time across amendment treatments. Error bars represent ± SE (n = 36 for amendments, n = 6).

Magnesium (Mg) concentration showed a significant interaction between amendment type and proportion over time ($F_{10,194} = 2.4$, $p < 0.05$). The unamended tailings showed the greatest Mg concentrations across time but only showed significant differences between the ash technosols. (*Fig 17., Plot A*). Both the unamended tailings and the compost demonstrated Mg concentrations 2x that of the ash technosols. Compost technosols were the only amendment treatment that demonstrated significant differences Mg concentrations based on proportion ($X^2 = 17.26$, df = 6, $p < 0.05$), with the 12.5% compost technosols showing elevated concentrations compared to the unamended tailings and the other proportions (*Fig. 17, Plot B*). This elevation remained consistent over time; the 12.5% treatments were 1.5x higher than the unamended tailings, and over double the concentrations of the other treatments (*Fig. 17, Plot B*). Ash technosols did not show this kind of variation between proportions.

It’s worth noting that vetch presence showed a significant effect on Mg concentrations within the unamended tailings over time ($F_{2,11} = 6.24$, $p < 0.05$). Both treatments showed significant increases over time ($F_{2,11} = 18.48$, $p < 0.05$), but starting at week 4, tailings receiving the vetch treatment exhibited 1.5x the concentration of the abiotic controls (*Fig 18*).

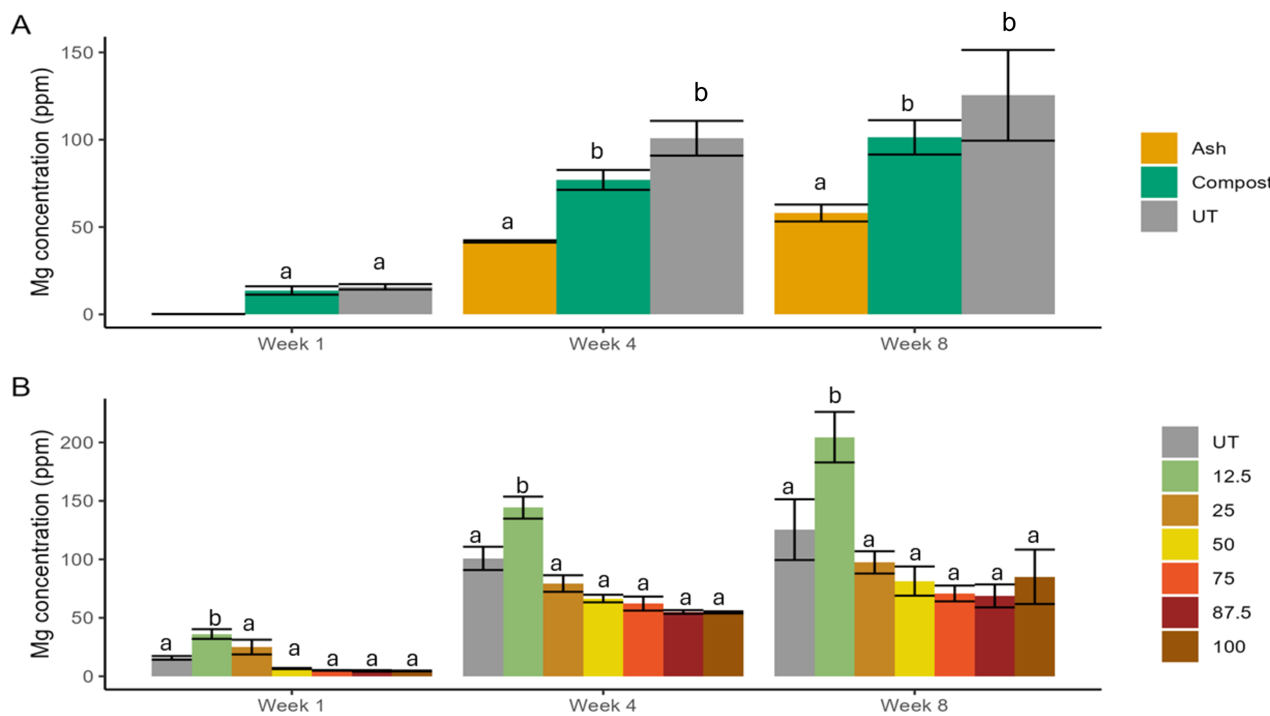

Figure 17. Mg concentrations based on amendment type and proportion over time. *Plot A* contains the Mg concentration based on amendment over time; error bars represent ± SE (n =36 for amendments, n = 6 for UT). *Plot B* shows the differences between compost proportion over time. Error bars represent ± SE (n = 6).

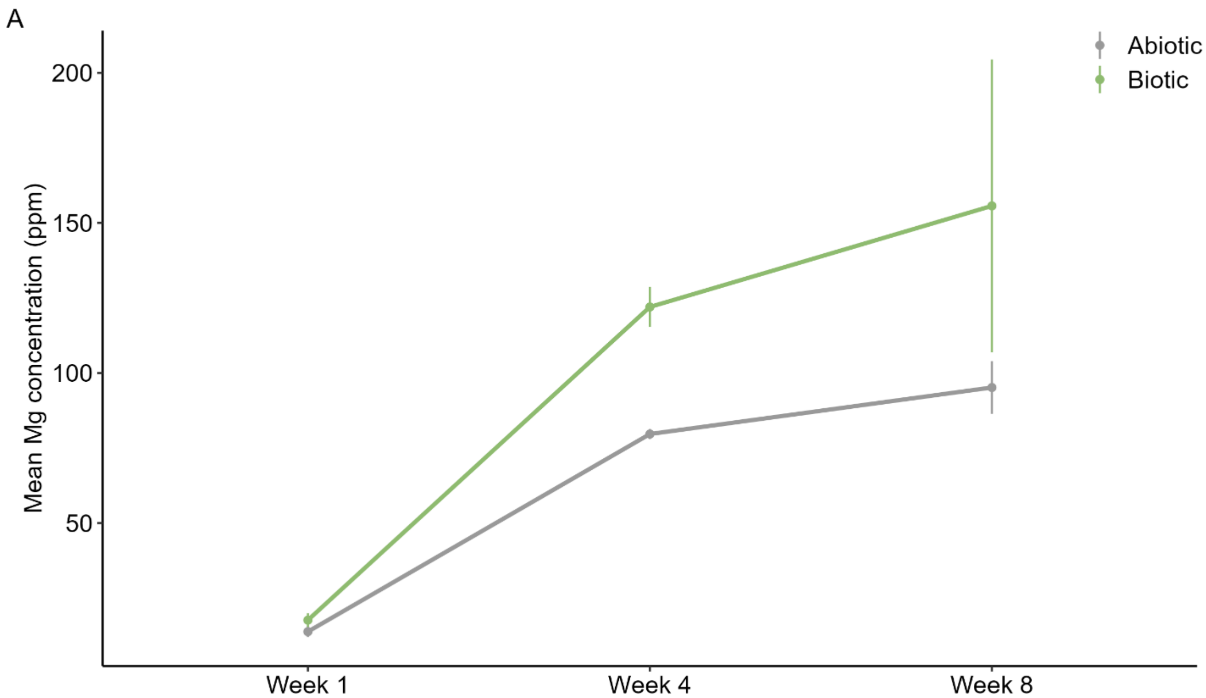

Figure 18. Changes in mean Mg concentration within the unamended tailings over time. Error bars represent ± SE, n = 3.

Relative saturations of base cations between amendment treatments showed that K was the most variable between the treatments (*Figure 19*). Within the unamended tailings, K occupied the lowest proportion of cation exchange sites, and Mg, Na, and Ca showed relative equal saturation. By contrast, both amendment treatments showed elevated K at time 1 compared to the unamended tailings (*Fig 19, Plots B and C*). However, while the overall K saturation across time was higher in both amendment treatments, both amendments also showed a rapid decline in K concentration observable within the timespan of the trial. This drop in K saturation was followed by an increase in the relative saturation of Mg, Na, and Ca. By week 8, K came to occupy a similar proportion of exchanges sites as the other three base cations within the compost and ash technosols.

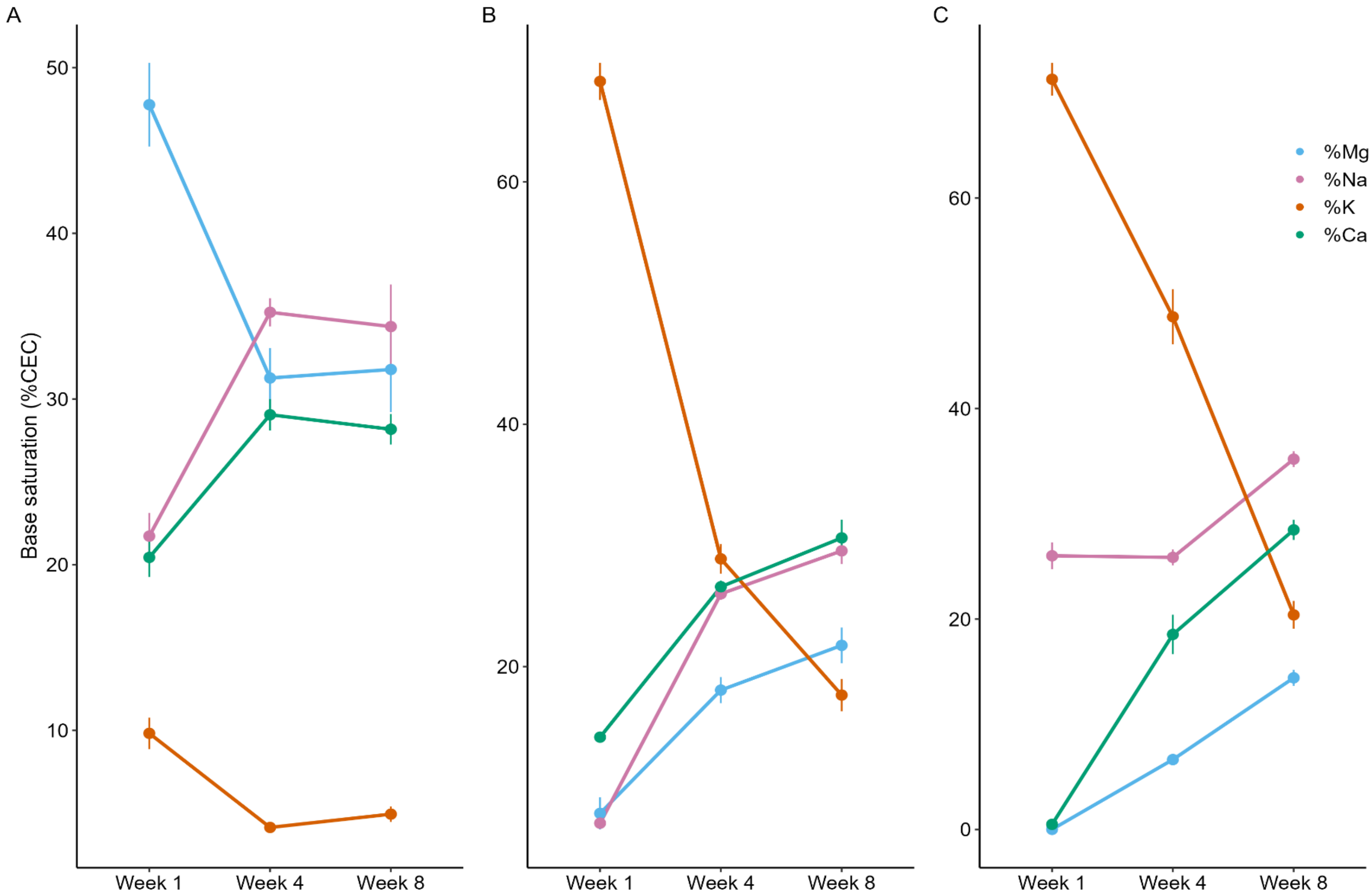

Figure 19. The relative saturation of base cations within the amendment treatments. *Plot A* shows the relative saturation within the unamended tailings, *Plot B* shows that of the compost treatments, and *Plot C* shows the ash treatments. Error bars represent ± SE (n = 36 for amendments, n = 6).

Within all technosols, Fe concentration varied around the detection limit (0.0003 ppm), making it difficult to discern any effects of the proportion or plant treatments. Amendment and time showed a significant interaction ($X^2 = 103.41$, df = 2, $p < 0.001$), with the compost technosols demonstrating the highest concentration over time (*Fig*. 20, *Plot A*). By contrast, ash technosols showed significantly greater values in week 8 compared to week 1 ($X^2 = 7.90$, df = 2, $p < 0.05$). Compost applications corresponded with significantly elevated Fe concentrations within week 1 ($X^2 = 35.61$, df = 6, $p < 0.05$) and week 4 ($X^2 = 35.61$, df = 6, $p < 0.05$), but not at week 8. Fe concentrations were significantly higher in the compost X plant treatments at week 8 ($X^2 = 6.53$, df = 1, $p < 0.05$), regardless of proportion (*Fig. 20, Plot B*). Biotic treatments averaged 2.77 times greater than their abiotic counter parts at week 8.

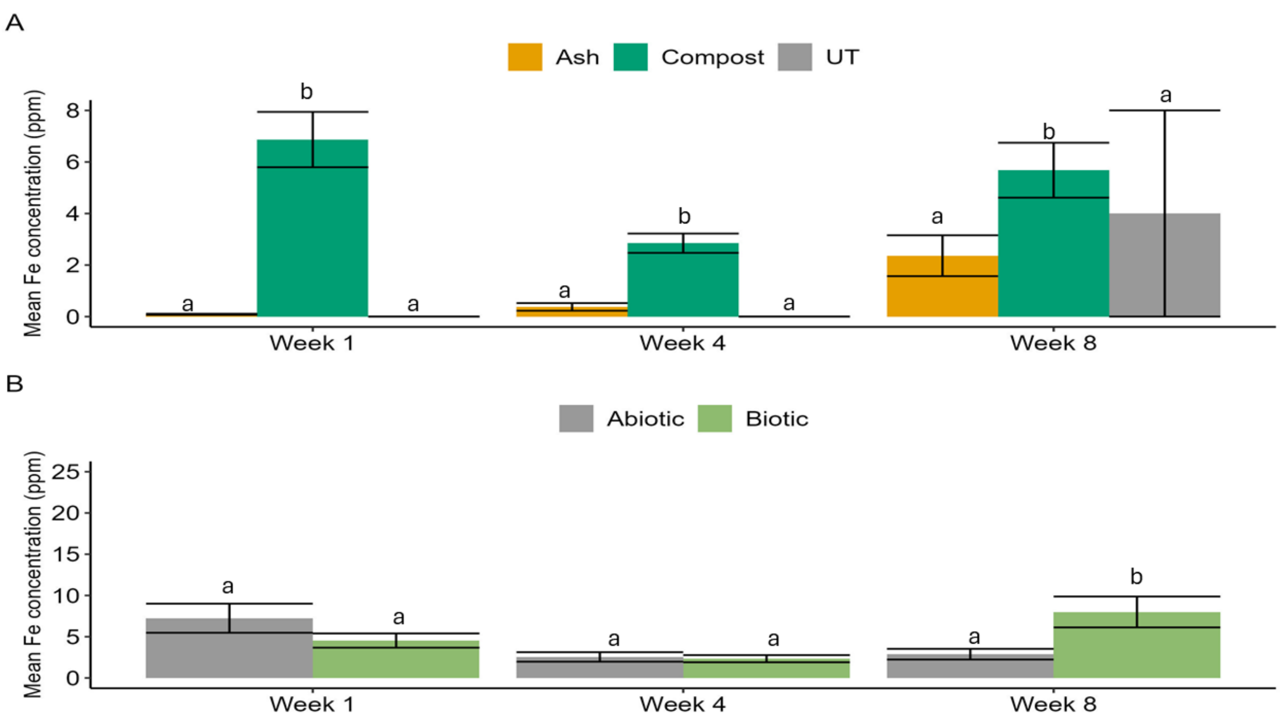

Figure 20. Fe fluctuations across technosols over time. Plot A shows the fluctuations for ash technosols (Error bars represent ± SE, n = 6) and Plot B shows those for the compost technosols (Error bars represent ± SE, n = 3).

Time was the only significant factor affecting silica concentration within the leachate (*Fig. 21*). Within the negative control group, vetch was not found to be significant across time. However, the technosols receiving vetch showed elevated dissolved silica at week 8 (*Fig. 21, Plot A*). Within compost technosols, all proportion groups showed consistently significant increases across time ($F_{2,104}$= 171.24, $p < 0.0001$). In ash technosols, dissolved silica significantly increased between each observation of the leachate within the ash technosols ($F_{2,104}$= 138.52, $p < 0.0001$). Proportions showed variation across time, but these did not remain consistent. Plants did not show any correlation with silica concentrations in either amended technosol.

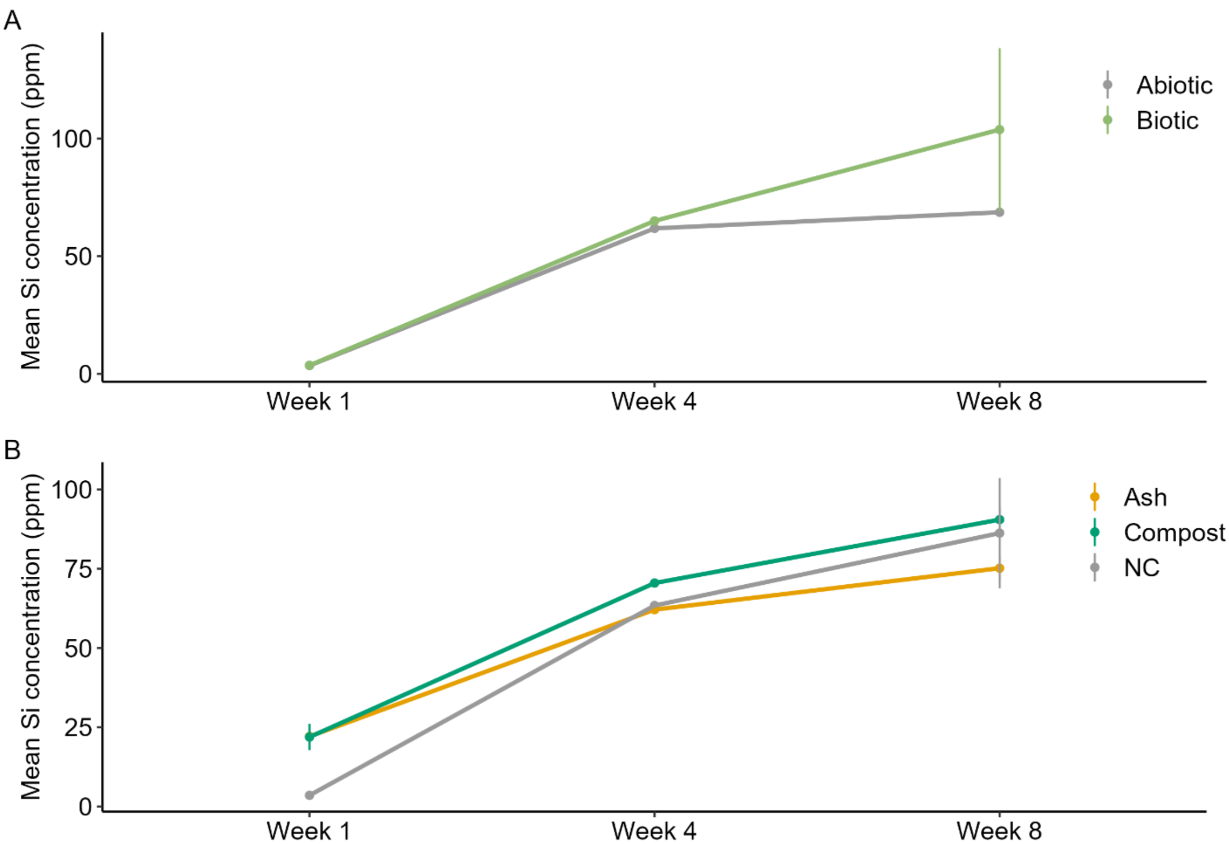

Figure 21. Si fluctuations across technosols over time. Plot A shows the fluctuations for unamended tailings (Error bars represent ± SE, n = 3) and Plot B shows those for all amendments (Error bars represent ± SE, n = 6 for NC, n = 36 for amendments).

Finally, aluminum concentrations were elevated in week 1 but then became largely undetectable (DL = 0.02 ppm) thereafter. Where aluminum did appear, it was never consistently linked with organic amendment or plant treatment.

3.2.3. Trace Metals

As, Cd, Co, Pb, Sb, and V all fell below detection after initial leachate testing at week 1 (*Fig. 22*). None of these elements appeared in concentrations above their CCME standards for protection of irrigation water. Of the trace metals assessed, Cr, Cu, Ni, and Zn appeared in sufficient quantities above detection to warrant further analysis.

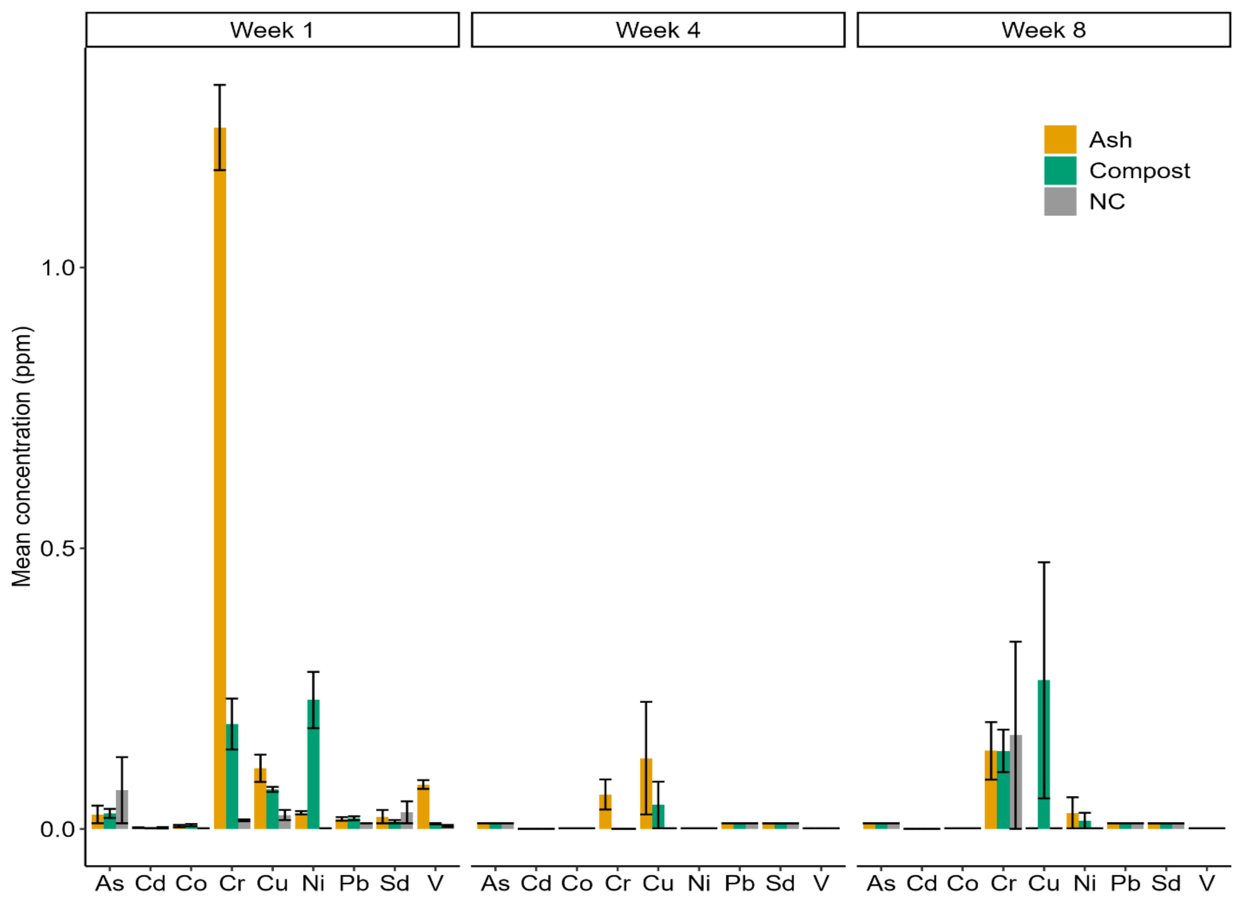

Figure 22. Trace metal fluctuations across amendment technosols over time. Error bars represent ± SE, n = 6 for NC, n = 36 for amendments.

Amendment ($X^2 = 7.88$, df = 3, $p < 0.05$) and time ($X^2 = 137.69$, df = 3, $p < 0.05$) showed significant influence on the concentration of Cr within the leachate. The highest Cr concentration occurring in the ash technosols at week 1, which was nearly 7x greater than the other two treatments (*Fig. 22*). Across amendment treatments, Cr concentrations showed a significant decrease between week 1 and week 8 ($X^2 = 137.69$, df = 2, $p < 0.05$). However, week 4 demonstrated the lowest concentrations of Cr for all treatments and then increased at week 8. Proportion did not introduce any consistent effects through time. Chromium leaching exceeded the CCME guidelines trivalent chromium for Protection of Irrigation Water and Protection of Aquatic Life at all three points in time within the ash technosols (*Fig. 22*). Chromium concentrations at week 1 and week 8 were in exceedance of CCME water guidelines for trivalent chromium by as much as two-hundred times.

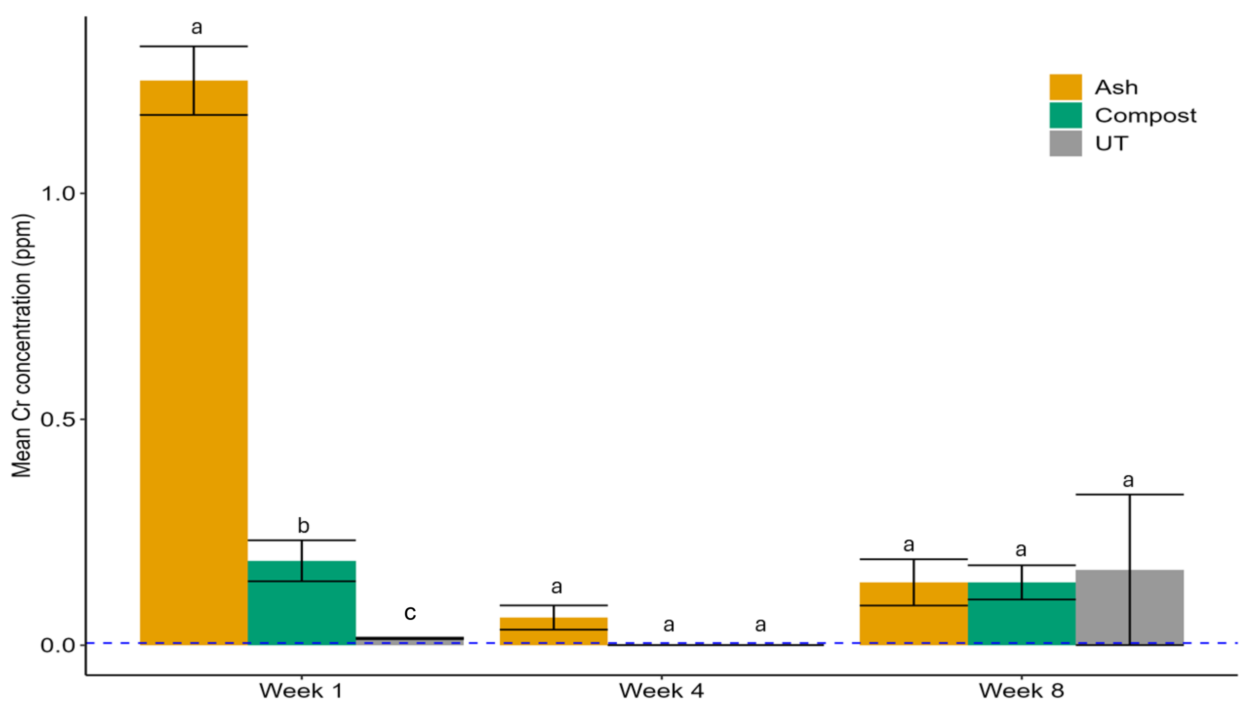

Figure 23. Cr fluctuations across amendment treatments over time. Error bars connote with ± SE, n = 36 for amendments, n = 6 for unamended controls. UT are represented by unamended tailings. CCME guidelines for Cr (III) in irrigation water are noted with the dotted blue line (4.9 µg/L).

Cu concentrations did not vary significantly between amendment technosols and the unamended tailings. Time proved to be the only significant factor, with values at week 1 being significantly elevated compared to week 4 and 8 ($X^2 = 23.44$, df = 6, $p < 0.05$). Thereafter, Cu concentrations within the leachate rarely went above detection after the start of the trial (*Fig. 24*). When they did exceed detection, there was no clear trend that followed proportion or plant presence. Cu concentrations did not exceed irrigation water guidelines at the start of the trial but occasionally did in weeks 4 and 8. These spikes were observed in the amended treatments (*Fig. 25*).

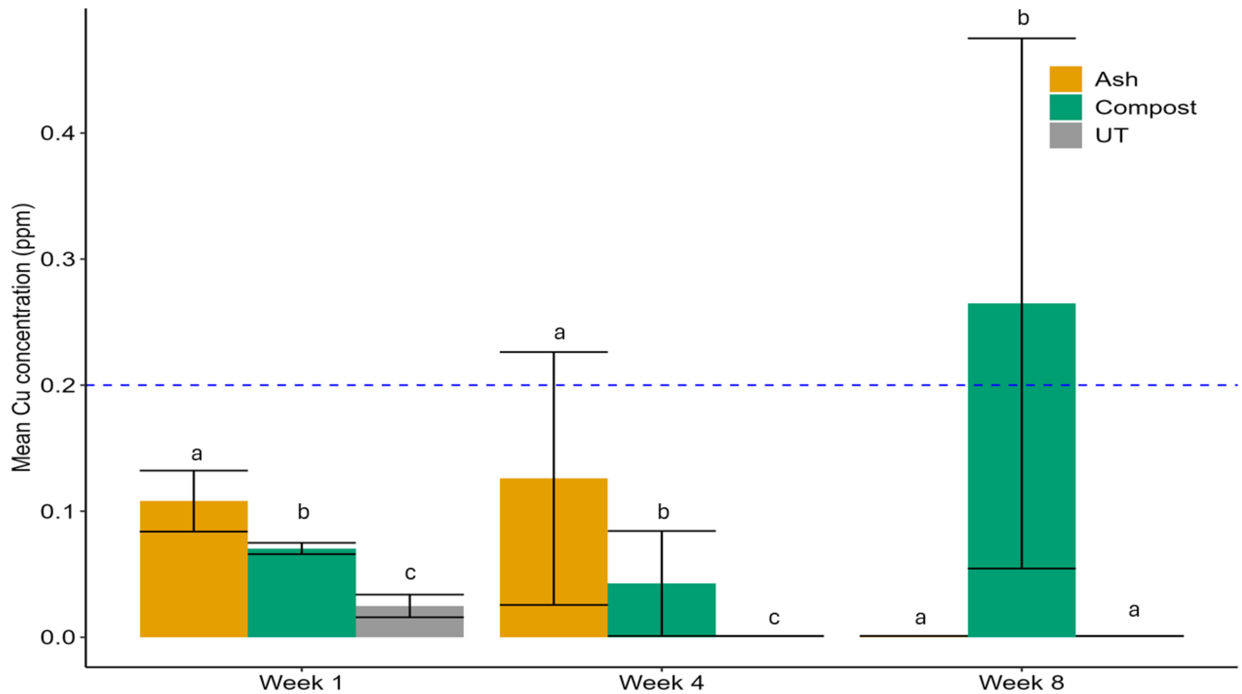

Figure 24. Cu fluctuations across amendment treatments over time. Error bars connote with ± SE, n = 36 for amendments, n = 6 for unamended controls. CCME guidelines for Irrigation Water for cereal crops is denoted with the dotted blue line (200 µg/L).

Both amendment ($X^2 = 8.26$, df = 2, $p < 0.05$) and time ($X^2 = 1575.35$, df = 2, $p < 0.001$) demonstrated significant effects on Ni concentration within the leachates. Proportion nor vetch did not introduce consistent significant differences to the Ni leached from the columns. Compost technosols at week 1 demonstrated the greatest concentration of Ni, being 8x that of the next highest in the ash treatments (*Fig. 25*). Thereafter, the concentration becomes too variable to be fairly compared. The unamended tailings demonstrated the lowest values of the three amendment treatments, and rarely went above detection. Values for nickel never surpassed the CCME guidelines for Protection of Irrigation Water (*Fig. 25*).

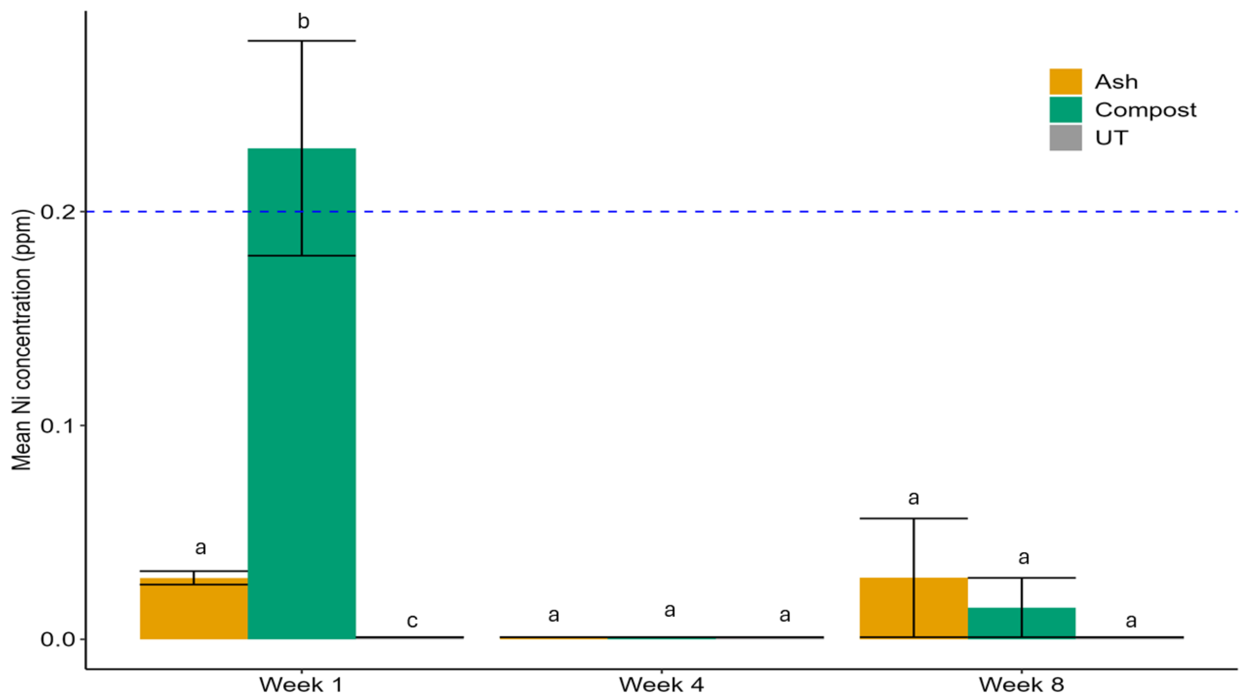

Figure 25. Ni fluctuations across amendment treatments over time. Error bars connote with ± SE, n = 36 for amendments, n = 6 for unamended controls. The CCME guidelines for Protection of Irrigation Water are denoted with the dotted blue line (200 µg/L).

Zinc concentrations fluctuated significantly through time ($X^2 = 133.7957$, df = 2, $p < 0.05$), but not between amendment technosols. Within the compost technosols, Zn concentrations significantly increased between week 1 and week 4, and then significantly declined between week 4 and week 8 ($X^2 = 97.9698$, df = 2, $p < 0.05$). At their height at week 4, Zn concentrations varied significantly along between proportions ($X^2 = 19.131$, df = 6, $p < 0.05$), with proportions receiving 50% or greater Zn concentrations than those below that threshold (*Fig. 26*). At week 8, proportion stops becoming significant and becomes replaced by plant treatment as a significant factor ($X^2 = 17.35$, df = 1, $p < 0.05$). Vetch treatments at week 8 demonstrated double the amount of Zn in their leachates compared to abiotic controls.

The CCME guidelines for irrigation water contain two benchmarks for zinc concentration as its availability is pH dependent, with pH of 6.5 being the demarcating value. Most compost technosols ranged between pH 7.5 and 6.5 and only dropped below that value at the higher end compost applications (*Fig. 26*). Thus, only the pure compost technosols exhibit values over the CCME guidelines for zinc concentrations, and the rest of the technosols fall below the alkaline threshold (*Fig. 26, Plot A*).

Ash technosols shared the same Zn dynamics as the compost technosols, with week 4 being showing significantly higher concentrations than weeks 1 and 8 ($X^2 = 60.84$, df = 2, $p < 0.05$). Week 1 was the only time when the proportion of ash to tailings showed a significant effect on variation ($X^2 = 33.31$, df = 2, $p < 0.05$). Thereafter, proportions did not factor into variation within Zinc concentrations. Since ash technosols at all times showed pH well above 6.5 (*Fig. 26, Plot B*), no technosols at any point were in exceedance of CCME values for Protection of Irrigation Water (5000 µg/L).

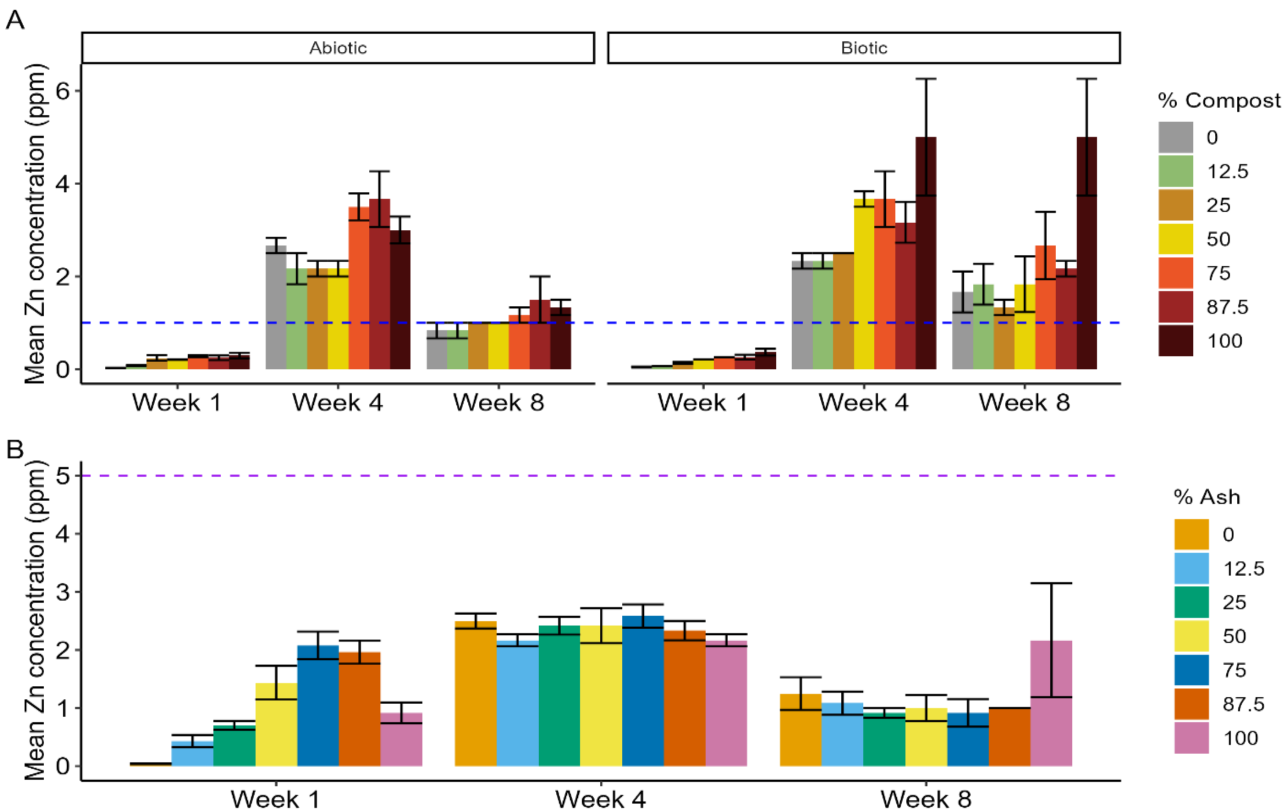

Figure 26. Zn fluctuations across amendment treatments over time. Plot A shows compost technosols, and Plot B contains the ash technosols. Error bars represent ± SE, n = 3. NC are represented by 0 % treatments. CCME guidelines for Protection of Irrigation Water are denoted with the dotted lines in the plots.

4. Discussion

4.1. Impact of amendments on plant growth

The disparity in observed germination success likely stems from the differences in alkalinity between the amendments. The decreasing germination success with increasing ash proportions (*Fig. 6*) reflects the increasing alkaline solution created by the high Na and K content of the ash (*Fig. 15, Fig. 16*). Similar trends have also been observed with ryegrass (*Lolium perenne*) and wheat (*Triticum aestivum)*, which showed depressed germination success when exposed to increasing concentrations of sodium carbonate salts (Bhatt and Santo 2016; Lin et al. 2018). Highly alkaline solutions lead to an intense concentration gradient within the medium, leading to extreme osmotic stress and possibly seed death (Guo et al. 2009). The unamended tailings also contained relatively alkaline solutions, but the pH of the leachates observed were three units of pH lower than the ash treatments (*Fig. 12, Plot A)*. Seeds from the aforementioned plant species have been noted to remain dormant under alkaline stress, and then germinate when solution conditions are not as alkaline (Guo et al. 2009; Bhatt and Santo 2016; Lin et al. 2018). Thus, the alkaline conditions present within the unamended tailings likely fluctuated enough around the alkaline tolerance of *V. vilosa* to provide a window for germination.

The extreme salinity of the soil solutions within the ash technosols also explains the depressed total biomass of the vetch that grew in the ash technosols. Osmotic stress within the substrate leads to increased desiccation of plant roots and prevents essential nutrient uptake (Guo et al. 2009). The decreased biomass observed along with the ash proportion gradient likely reflects the increased osmotic stress incurred through greater ash application. Even within the compost technosols, increased pH corresponded to lower vetch biomass, and increased Mg, Na and trace metals such as Cr and Ni (*Fig. 11*). A previous study noted a similar decrease in tailing pH with compost was applied; this corresponded to an increased in plant biomass (Párraga-Aguado et al. 2017). Soil pH has been dubbed the 'grand mediator' of all soil chemical activity, being the determinant of availability of plant nutrients and speciation of trace metals (Weil and Brady 2016). The slightly acidic pH of the compost mitigated the more alkaline tailings, which boosted plant growth success and germination.

However, osmotic stress is not the only factor explaining the variation between amendment technosols. The introduction of plant available nutrients from compost represents the crucial difference between the compost technosols and unamended tailings. PCA analysis revealed that technosols receiving higher compost by mass corresponded with increased plant nutrients, most notably phosphate, nitrate, B, Mo, and K (*Fig. 5*). The ultramafic tailings themselves showed a dearth of K across time (*Fig. 16*), so the incorporation of K from the compost made up for this shortfall. In their study, Párraga-Aguado et al. (2017) noted K and P as limiting nutrients in mine tailing media, suggesting that these nutrients are often lacking in many different tailings. The ultramafic minerals do not contain minerals that would supply these nutrients through natural weathering (e.g. K-feldspars and mineral apatite), so it is unsurprising that these nutrients would become limiting in the unamended tailings in absence of organic pools (Grandillo et al. 2020). Thus, plants grown in these media must rely on their concentrations found within the organic pools introduced by organic amendments.

While compost introduced important plant nutrients to the substrate, the proportion of compost did not yield significant differences between total biomass (*Fig. 7*). These finding mirror a similar studies observing the growth of ryegrass in ultramafic and Pb-Zn tailings (Cheng et al. 2022; Ball 2023). Based on the findings within this study, this consistent total biomass across compost proportions represents the change in carbon allocation between the roots and shoots of the vetch. Within the unamended tailings, RMF and SMF were evenly split at 50%, but then starkly declined with the introduction of compost at 12.5% (*Fig. 8*). The 12.5% technosols showed a greater RMF compared to other compost proportions, and RMF was shown to negatively correlate with plant nutrients such as phosphate and nitrate (*Fig. 7*). This trend reflects the 'functional equilibrium model', in which plants allocate biomass to tissue depending on abiotic limitations, and is consistent with other studies in technosols (Poorter et al. 2012; Watkinson et al. 2017). In the unamended tailings and 12.5% technosols, the dearth of nutrients likely requires more allocation to the roots for medium exploration. As compost application increases, the RMF decreases and the SMF increases, but the rebalancing of biomass allocation leads to a stagnation in total biomass.

The allocation to roots at lower compost proportions might also represent the response to increased trace metal content within the tailings. Fast-growing Fabaceae species (to which *V. villonsa* belongs) are noted for increased RMF when exposed to acute heavy metal stress (Audet and Charest 2008). This is corroborated within this study by the correlation of total trace metals such as Ni and Cr with increased RMF (*Fig. 7*). Thus, the combined effects of lower nutritional content and increased heavy metal stress of controls and 12.5% technosols lead to greater RMF compared to higher compost technosols. Thereafter, as compost application increases, plant nutrients become more available and heavy metal stress becomes lower, leading to less allocation to RMF and more to the SMF. Thus, the rebalancing of biomass allocation leads to a stagnating total biomass along the compost application gradient.

4.2. Mineral Weathering

4.2.1. Mineral Dissolution

Plants only affected the weathering of the tailings when no organic amendments were present. Mg (*Fig. 17*) and Si (*Fig. 21, Plot A*) concentrations are frequently used to reflect the rate of ultramafic dissolution, meaning their higher concentration within the biotic treatments reflects possible plant-driven bio-weathering (Chardot-Jacques et al. 2013; Thom et al. 2013; Power et al. 2020). Plant roots and their associated microbiomes have previously been shown to significantly release metals from primary minerals in common geologic and ultramafic settings (Chardot-Jacques et al. 2013; Burghelea et al. 2018). Dissolution of the ultramafic tailings likely occurs due to the low molecular weight organic acids (LMWOAs) and siderophilic root exudates associated with plants roots and rhizosphere microbes, with malic and citric acid being of particular note for the dissolution of Mg (Kanbar et al. 2020). However, Mg concentrations appear to spike at week 4, and then more or less stagnate between week 4 and week 8, while Si and Fe show more variability as it relates to plant treatment. Power et al. (2020) found that the most labile of minerals within the ultramafic tailings are dissolved within a similar timeframe as my study, suggesting that the plants within the unamended tailings preferentially dissolve these same labile Mg sources. These labile minerals are represented by brucite ($Mg(OH)_2$), which have been shown to readily dissolved in conditions like the ones in the greenhouse (Power et al. 2017). This would also explain why the Mg concentrations spikes at week 4 within the biotic treatments, while the differences in Si and Fe concentrations only appear within week 8.

The weathering conditions change when organic amendments are incorporated into the tailings. Ash technosols did not demonstrate any weathering related to plant growth. This is likely due to the low success of plant establishment within these media. Instead, ash treatment dissolution seems dictated by the extreme concentration of Na and K within the solutions. The extreme concentrations, along with their low valency, led to their preferential leaching compared to divalent base cations. The leaching of monovalent cations from soils and sediments has been well documented; K and Na are leached out due to their lower electro-positivity and large hydrated radius, both of which prevent sorption to soil colloids and organic ligands (Weil and Brady 2016). K in particular typically remains in its freely available K^+ form, making it highly mobile within ecosystems (Binkley and Fisher 2020). This is reflected well in the change in relative saturation in base cations with the ash technosols (*Fig. 19*), where the K saturation starts greater than the other three cations, but then dramatically declines over the sampling period. It is only after both K and Na have been leached in sufficient quantities that Mg and Ca begin to occupy more of the base cations within the soil solution. Considering the relatively low concentration of K in the tailings, it is probable that the K found within these media is introduced from the ash themselves. Thus, the introduction of K and Na leads to solution kinetics that arrest the release of Mg and Ca compared to compost technosols (*Fig. 19*), which is reflected by in-situ ultramafic soils (Hseu et al. 2018).

The Fe leached within the compost technosols, especially from the technosols receiving a higher compost application, likely originates from the organic pool. Compost has been noted to contain high concentrations of Fe depending on its source (Hargreaves et al. 2008). Regardless, the significantly greater Fe concentrations within the biotic treatments at week 8 suggest that plant growth is at least partially responsible for the release of Fe from the mineral and organic pools, either directly through root exudation or microbially mediated. The preferential leaching of Fe at week 8 within the plant treatments might reflect the initial stages of weathering of the iron oxides within the tailings. The tailings from the Baptiste Project contain an abundance of Fe-oxides, mainly in the form of antigorite, lizardite and magnetite (Grandillo et al. 2020). While plant root exudates have demonstrated the ability to weathering Fe-rich minerals in mine sites, it is equally plausible soil microbes are driving the leaching of Fe from these minerals (Rajkumar et al. 2009; Liu et al. 2020; Z. Li et al. 2021). Li et al. (2021) found that *Talaromyces* flavus, a serpentine adapted soil fungus, was able to excrete metabolites that were targeted at lizardite, antigorite and magnetite specifically. Future studies observing ultramafic tailings should endeavor to inoculate with serpentine-adapted microbes to observe if the leaching of Fe corresponds with these communities in ultramafic technosols. Given the lack of consistent Fe values above the detection limit in the compost technosols, I caution against putting too much weight on this correlation. Future studies should endeavor to employ methods that more selectively extract Fe, such as dithionite-citrate-bicarbonate (DCB), ammonium oxalate, and Napyrophosphate. Sequential extractions such as these will provide a clearer sense of where Fe is fractionated within the soil.

Unlike the unamended tailings, the concentrations of Mg and Si were not correlated with plant presence. Instead, the Mg seemed to be related to the proportion of compost incorporated, with the lower applications demonstrating greater Mg leaching compared to technosols receiving greater than 50% compost by mass (*Fig. 16*). The preferential leaching of Mg at 12.5% compost by mass likely represents the ratio of compost to tailings with the optimal exposure of minerals to the ligands and dissolved organic carbon (DOC) within the compost. It also could represent the dissolution of Mg-carbonates formed through the carbonation of labile brucite ($Mg(OH)_2$), which has been noted to occur under controlled conditions (Power et al. 2021). At higher compost applications, the medium is not as dense with minerals, which decreases the opportunity for Mg-silicate dissolution. It is difficult to determine the origin of this dissolved Mg without accompanying alkalinity data and DOC within the leachate. Future analysis should endeavor to record the concentration of carbonate and bicarbonate found within leachates, as well as the forms of DOC occurring in the leachate. Few studies have observed the kinetics of ultramafic minerals with organic ligands, so this effect highlights a future avenue of research.

However, within the context of this study, the lack of observed plant-driven bioweathering within the compost technosols likely stems from the increased abundance of plant nutrients within the organic pool. Ultramafic minerals have been noted for their dearth of plant nutrients, so plant roots and associated microbes are more likely to spend their energy decomposing nutrient-rich organic matter (Oze et al. 2008; Alexander 2010). Previous studies have noted that fertilizer greatly improves the growth of ryegrass grown in ultramafic tailings when concurrently amended with compost (Ball 2023). Fertilization represents a nutrient input that requires little energy from plants and microbes to exploit, with compost constituting a source that requires more energy inputs for the same effect. Thus, given that minerals represent a high energy, low reward source of nutrients for plants and microbes, it stands to reason that these organisms would not exploit minerals for nutrients in the presence of more labile organic matter. Assessing plant tissue elemental concentrations with trace isotope analysis will help to understand nutrient cycling within the technosols more effectively, as it will illustrate where the plants are acquiring their nutrients.

The change in CEC of the compost-tailings technosols reflects the pace of mineral weathering within these mediums. CEC values within these technosols are much lower than those of many more weathered soils, which suggests that the formation of secondary minerals and the decomposition of organic matter is relatively nascent (Weil and Brady 2016). The lack of significant differences between the proportions of compost is surprising given that organic matter is associated with higher CEC in field soils (Weil and Brady 2016). Considering that the compost used in these trials derives from yard and garden waste of residents, they likely are composed of more ligneous materials that are recalcitrant to decomposition in oligotrophic conditions (de Vries and Caruso 2016; B.C. Reg. 18/2002 2022). Thus, the compost used in the values of CECs constitutes organic matter composed of primary plant residues, which require priming with labile DOC to progress in decomposition. These low CEC values suggest that the tailing-compost technosols could become nutrient stressed if inputs from fertilization or compost become lacking. Consistent inputs of DOC and fertilizer might induce microbial priming, which has been shown to lead to elevated mineral dissolution (Fang et al. 2023). Thus, regular introduction of organic inputs could lead to more rapid pedogenesis of these ultramafic technosols and improve their capacity to retain nutrients. These efforts could help meet the capability requirements outlined in section 10.9.5 of the *Health, Safety and Reclamation Code,* where land capability reaches a point comparable to pre-excavation conditions (BCMEMPR 2010). It should be noted that methodology used to obtain CEC values in this study differs from the predominant methodology and may represent an underestimation of the true CEC value. Future studies should assess CEC values through the $BaCl_2$ method for more accurate measurements.

4.2.2. Trace Metals

The low CEC of the compost/tailing technosols might also explain the rapid leaching of the trace metal(loids) at the outset of the experiment. The process of repeated pulverization that creates the ultramafic tailings constitute an extreme and rapid form of physical weathering, which creates a medium containing a fairly uniform particle size (Grandillo et al. 2020). This process likely leaves many of the metal-bearing minerals more labile. It has been shown that soil medium containing an abundance of exchangeable trace metals will rapidly release these metals until the metal cations remaining are tightly sorbed to the remaining exchange sites (Diatta et al. 2004; Audet and Charest 2008; Sangiumsak and Punrattanasin 2014). In addition, many trace metals are likely still occluded in more the more recalcitrant fractions: carbonates, Fe-Al oxides and organic complexes (Massoura et al. 2006; Kumpiene et al. 2008). Organic matter from compost and ash has been noted to introduce trace metals depending on their manufacturing. Metal(loids) within ash originate from the paint used to mark trees and the various compounds used to treat the wood in the manufacturing process (Mollon et al. 2016; Mitchell et al. 2018). Compost can contain metals in abundance from the incorporation of materials receiving chemical treatments like that of ash materials, and from the natural concentrations of metals within plant residues (Hargreaves et al. 2008; Párraga-Aguado et al. 2017). Even though many metal(loids) of concern, such as As, Pb, and V, do not appear above their detection limit after week 1, this does not mean that they are not denuding from the media. While no CCME guidelines for these elements were noted in exceedance, their slow leaching from the media still constitutes an on-going risk as they could accumulate at depth or in biomass. Future studies should employ elemental scans that garner greater resolution, such as ICP-MS, and incorporate plant tissue concentrations. They should also seek to include sequential extractions to determine the pools of trace metals within the technosols.

Cr was the only trace metal that consistently leached out over time across all treatments (*Fig. 23)*. Cr denudation did not correspond to any plant driven weathering across any technosols and instead seemed tied to the amendment and its relevant proportion. Much of the risk associated with Cr contamination relates to its in-situ valency. Cr (III) is required by organisms for various metabolic functions and represents the most stable form, while Cr (IV) represents a noted carcinogen (Berkowitz 2014). While minerals from the Baptiste project are not known to contain Cr (VI), the transformation between Cr (III) and Cr (VI) is very dependent on oxidation conditions and pH (Grandillo et al. 2020; Dajčl et al. 2022). Within compost technosols, the organic fraction of Cr might contribute to some of the variation of the observed (*Fig. 23*). However, organically derived Cr (III) alone would not explain the concentration of Cr observed from the compost technosols. Cr in the tailings naturally occurs in the tailings in the form of chromite [$FeCr_2O_4$], which was the initial mineral of interest at the Baptiste project (Grandillo et al. 2020).

The results of this study differ from previous observations of Cr weathering in that the high pH ash technosols leached more Cr at week 1 compared to the lower pH compost technosols (*Fig. 23*). Lilli et al. (2019). observed that more acidic conditions lead to the leaching of Cr. The discrepancy in our study in week 1 likely stems from the abundance of exchangeable

Cr in the ash itself, as the positive controls were noted to have detectible Cr compared to the compost technosols (*Fig. 23*). At week 8, the more acidic or neutral pH of the compost demonstrates greater Cr compared to the ash technosols, which falls in accord with the aforementioned study. The spike noted at 12.5% compost by mass at week 8 likely reflects a similar effect as Mg, where the ratio of compost to tailings allows for maximized exposure of minerals to the organic ligands of the compost. It is beyond the scope of the information provided by our methodology to determine the species of Cr present within the leachate. Future studies should determine the species of Cr leaching from the tailings, as this would more accurately characterize the risk associated with such high Cr values in exceedance of the CCME guidelines for irrigation water (*Fig. 23*).

While Cu were not consistently detected above the detection limit of ICP-OES, within the samples analyzed, Cu leaching does not appear correlated with plant-driven bioweathering. Cu concentrations follow a similar pattern as the rest of the trace metals, where there are observable quantities at week 1, followed by a sharp decline thereafter (*Fig. 24*). This follows that all the abundance exchangeable Cu is removed from the system at the start and then whatever is left is locked within more recalcitrant organic and mineral forms (Kumpiene et al. 2008; Sangiumsak and Punrattanasin 2014). In fact, that observable spikes of Cu after week 1 of the trials occur in technosols receiving more compost by mass (*Fig. 24*), suggesting that the Cu leached is organic in origin. This is consistent with the observation that Cu tends to complex readily with organic matter (Kumpiene et al. 2008; Mitchell et al. 2018) . This is further enforced in week 1 where both amendments show greater Cu concentrations at greater application rate, albeit in very low quantities (*Fig. 24*). Cu leaching from organic amendments constitutes a source of potential secondary pollution and should be accounted for in long-term bioaccumulation monitoring.

However, the fact that vetch grown in compost technosols did not show any reduced growth related to total leached Cu suggests that Cu leaching in the observed quantities does not represent a pressing risk.

` Typically, Ni is one of the heavy metals of note that causes the adverse growth effects of plants in serpentine soils, and its hyperabundance leads to the natural selection of Ni hyperaccumulating species (Oze et al. 2008; Alexander 2010). Within this study, Ni toxicity does not seem to warrant an apparent risk to ecosystems within the time frame observed. This is evidenced by the low quantities of Ni leached from the technosols (*Fig. 25*), and by the fact that plants grown in mixed media did not produce adverse growth outcomes related to the amount of total leached Ni (*Fig. 25)*. Ni leaching was not related to any plant driven bioweathering and instead relates to treatment and proportion of organic amendment. The low Ni concentrations within the leachate might reflect its removal through the extraction process that precedes tailing generation. Whatever is left represents Ni bound in recalcitrant Fe-Al oxides, and thereby left to slowly leach in undetectable quantities with the current method (Massoura et al. 2006). Ni leaching follows a similar pattern to Cr within the compost-tailings technosols, albeit in not the same quantities (*Fig. 23 and 25*). Previous studies have noted that Ni and Cr covary in ultramafic soils across time, likely due to their residence in similar recalcitrant Fe-Al oxides (Hseu et al. 2018; Lilli et al. 2019). This is corroborated in our study by an observable correlation between Ni and Fe (*Fig. 7*). Future studies should look at the pools of Ni and Cr together and conduct mineralogical examples using XRD mineralogy and SEM microscopy.

Finally, Zn stands an outlier in the context of trace metal weathering. Zn release corresponds with plant driven bioweathering in the final week of the trial within the compost treatment (*Fig. 26, Plot A*). Technosols receiving more compost by mass at this time showed elevated Zn compared to those receiving lower compost by mass technosols. This suggests that plants and their associated microbes are likely decomposing the organic pool of Zn for uptake. This makes sense considering that Zn is a known micronutrient required by most organisms, so its release from the observed media reflects the residual Zn released through decomposition (Broadley et al. 2007; Hargreaves et al. 2008). Besides the disproportionate release of Zn at week 8 within the compost technosols, Zn concentration shows patterns over time that differ from most other trace metals. Instead of peaking at week 1 like the other observed trace metal(loids), Zn peaks at week 4 and then declines slightly at week 8 in both technosols (*Fig. 26*). Within compost technosols, Zn leaching is correlated with those of other nutrients, notably nitrate and phosphate (*Figs 5 and 6*). Previous work on zinc dynamics in organic amendments have noted that Zn covaries with phosphate, suggesting that organometallic complexes within the compost incorporate these two readily leached ions (Kumpiene et al. 2008). Zn also commonly occurs within both bottom and fly ash, with content varying with source material and temperature of combustion (Saqib and Bäckström 2014). The delayed release of Zn from these technosols contradicts previous findings about the adsorption capacity of Zn, which typically leaches preferentially compared to other metals such as Ni and Cu (Sangiumsak and Punrattanasin 2014). Zn concentration from the unamended tailings showed significant increase between week 1 and week 4, suggesting that the increased concentration of Zn in the technosols derived from the release from the mineral fraction (*Fig. 26*). However, prior mineralogical examinations of the ultramafic tailings did not show any notable abundance of Zn-oxides, and little literature exists on the concentration of Zn in ultramafic minerals (Grandillo et al. 2020; Power et al. 2020). The factors dictating Zn dynamics warrant further investigation.

Zn differs in its CCME guidelines due to its absorption being related to pH dependence. Within the compost technosols the pH only falls below the 6.5 threshold in the technosols receiving 87.5% compost by mass or greater (*Fig. 26*). Considering that this represents an unlikely fraction to be incorporated for in-situ remediation, the observed Zn concentrations do not present an apparent risk. However, if the compost was used as a cap for the tailings, as is done in some remediation contexts, then the concentration of Zn may pose a risk of secondary pollution. While the ash technosols present solutions pH values well in exceedance of 6.5, the concentration of Zn falls below the CCME guidelines, so there is not risk of Zn poisoning at these concentrations.

5. Conclusion

The two chosen amendments represent extreme contrasts whose qualities dictate the success of plant growth and mineral weathering outcomes. Compost technosols contained a suite of plant nutrients that corrected for the deficiencies of the tailings themselves and stimulated plant growth accordingly. It was demonstrated that the proportion of the compost is inconsequential to plant growth outcomes after their initial incorporation. This provides useful data for reclamation planning, as this reduces the cost of transporting and collecting enough compost required for reclamation. Ash technosols were harsh for plant growth even at small quantities, and increasing their proportion leads to more adverse growth outcomes for plants. The extreme alkalinity of the ash technosols makes them unideal for use in reclamation of the ultramafic tailings. Thus, the quantity of amendments incorporated appears to be related solely to the overall quality of the amendment itself. Amendments like composts, nutrient rich and circumneutral pH, do not require a high application rate.

Plant driven weathering was only observed in the unamended tailings, as evidenced by the greater concentrations of Mg, Si and CEC within the biotic treatments. Thus, revegetation of ultramafic minerals does not pose a risk of releasing trace metals through bioweathering of the minerals, provided that the remediation strategy properly addresses the needs of the vegetation.

Chapter 3

How do reclamation practices impact carbon sequestration on reclaimed ultramafic mine tailings?

1. *Introduction*

In the context of climate change and ecological degradation, mining operations face a paradox: extracting materials essential for a decarbonized future generates significant carbon emissions and habitat loss. The Baptiste project exemplifies this challenge. Its primary product, nickel, in the form of awaruite, is a key material for electric vehicle batteries (Wong and Coates 2010). However, the Baptiste project is estimated to emit 70,000 t CO_2e per year, with its tailings management facility covering 5 km^2 adjacent to a provincial park and treaty lands (FPX Nickel 2024). Mine tailings are often reclaimed through the application of organic amendments and revegetation (Larney and Angers 2012; Jayapal et al. 2022). The mixture of tailings and organic amendments constitutes a novel parent material, meaning that carbon sequestration in these settings can be observed through monitoring soil formation processes (Huot et al. 2015). Soils contain both organic and inorganic carbon, and the formation of both pools offers potential for carbon sequestration. Thus, the objective of my study was to observe the effects reclamation treatments have on both pools of carbon, and the impact their formation has on the potential for carbon mitigation for the Baptiste Project.

Incorporating organic amendments and vegetation introduces organic carbon into mine tailings, creating the potential for organic carbon sequestration. Soils sequester organic carbon through forming mineral-associated organic carbon (MOAC) and aggregates, which prevents their decomposition and subsequent respiration (Lehmann and Kleber 2015b; Keiluweit et al. 2017; Cotrufo et al. 2019). Formation of MAOC is controlled by parent material mineralogy, soil pH and organic ligand character (Kleber et al. 2015; Lehmann et al. 2020; Kögel-Knabner and Amelung 2021). Aggregates form through the action of clay particles, plant and microbial exudates and physical processes such as shrink-swell and cryoturbation (Weil and Brady 2016).

Little attention has been given to the carbon occlusion on reclaimed mine tailings, which makes it difficult to assess how reclamation treatments might improve carbon emission mitigation in mine tailing settings. For projects that reclaim tailings through amendment application, the amendment application rate offers an important control on the formation of MAOC and aggregates. Thus, using the Baptiste tailings as a case study, my study asks: *How does organic matter application rate affect carbon occlusion on ultramafic mine tailings?* I hypothesize that a change in organic matter application rate will lead to a change in carbon occlusion, as determined by aggregate stability and MAOC formation.

In addition to organic carbon sequestration, the mineralogy of the Baptiste tailings offers the opportunity for inorganic carbon sequestration. Mineral carbonation, a natural process in which silicate minerals convert to carbonate minerals, has been a large contributor to the natural sequestration of carbon from the atmosphere (Sanna et al. 2014). Recently, the possibility of carbonating minerals through industrial means, particularly in ultramafic mineral deposits, has garnered attention (McCutcheon et al. 2016; Gras et al. 2020; Power et al. 2021). Through the serpentinization process, labile Mg-silicates and hydroxides are formed, which then readily transform into carbonates when exposed to the atmosphere (Power et al. 2020). While this approach could potentially offset the Baptiste project emissions, its application is still in the preliminary stage, and its long-term effects remain uncertain. Power et al. (2021) observed the formation of stable carbonates in ultramafic tailings mixed with compost, suggesting that organic amendments have the potential to promote natural mineral carbonation. This phenomenon has yet to be observed in conditions closer to those of the Baptiste site. Thus, in addition to organic carbon sequestration, my project asks: *How does organic amendment application rate affect the* *rate of mineral carbonation?* I hypothesize that a change in organic matter application will be followed by a change in mineral carbonation.

To answer these two questions, a mesocosm trial was conducted using ultramafic tailings treated with organic amendments and revegetation. Municipal compost and pulp ash were selected for the organic amendments; both amendments are produced in large quantities within the region, offering the potential for mitigating two waste streams at once. In addition to organic amendment application rate, two conifer species (*Pinus contorta* and *Picea glauca*) were selected based on their prevalence in the region of the Baptiste project (Bergh 2012). Their inclusion offers insight into any species-specific effect that may occur as it relates to carbon occlusion. This study is the first to simultaneously examine carbon occlusion and mineral carbonation in ultramafic technosols.

2. *Methods*

2.1. *Mesocosm design*

Mesocosms containing ultramafic technosols were set up using a randomized-complete block design (RCBD) outside of the Irving K. Barber Enhanced Forestry Lab (EFL) at the University of Northern British Columbia. Ultramafic tailings were sourced from the Baptiste Project and mixed with fly/boiler ash (Canfor Pulp and Paper Mill, Prince George, BC) and Grade A compost (Municipality of Prince George) in the same mass to mass proportions described in Section 2.1 of Chapter 2. These mixtures were placed in 3-L fabric containers (Viagrow, Athens, Georgia) and then planted with 2-year-old tree saplings (Figure 1). Half of the saplings were two-year old *Pinus contorta* saplings and the other half *Picea glauca*. Each treatment was replicated five times. With two amendments, two plant species, seven amendment ratios, and five replicates, the experiment utilized a total of 140 experimental units. However, since both amendment treatments share the same unamended control, the total experiment size includes 135 units. The experiment ran from May 2023 to October 2023 to simulate a growing season. Trees were watered three times weekly with reverse osmosis water sourced from the EFL. Hereafter, treatments are referred to by their organic amendment and technosol type (e.g. compost technosols), with the tailings control, referred to as the “unamended tailings”.

Figure 27. An image of the RCBD container experiment.

2.2. Data Collection

At the end of the trial, trees were removed, and the remaining substrate was sieved to < 2mm. Subsamples were collected from the sieved materials and sent to the Northen Analytical Laboratory at UNBC services for acid fumigation to determine TIC, TOC, and TN. Briefly, samples were combusted using a Costech 4010 elemental analyzer, with standard sequential combustion/reduction setup recommended by the manufacturer with a flowrate of 100 mL/min of helium 5.0 as the carrier gas. Total carbon was assessed in 4 mm x 6 mm tin capsules. The combustion oven was set to 1000°C, the reduction oven to 650°C, and the GC oven to 75°C for C and N. Gases were then separated on a standard 3 m column and quantified with the built-in thermal conductivity detector (TCD).

For TIC determination, the acidified samples, which had already been weighed, were placed in 5 mm X 8 mm silver capsules, and pre-wetted with 50 µl of deionized water. The samples were then exposed to 60 mL of concentrated HCl in 100 mL beakers for 6 hours. The capsules were refreshed with another 60 ml of HCl and were left for 24 hours. Once the HCL gas was cleared, the samples were oven dried at 55 Ċ prior to analysis.

The C:N ratios were then calculated using the derived TC and TN values in the following equation:

$$R = \frac{M_w(TC * (100 - \%W))}{M_w(TN * (100 - \%W))}$$

Where *R* represents the C:N ratio, M_w is the wet mass of the soil in the container, *TC* and *TN* are total carbon (%) and total nitrogen (%), respectively, and %W refers to the moisture content of the soil as a percentage. The moisture content was found to be approximately 55.75% for the compost and ~ 16% for the ultramafic tailings.

MAOC was assessed using the Par1-Den method described by Poeplau et al. (2018). All compost technosol treatments were homogenized using a Riffle-type soil splitter (Hoskin Scientific), combining five replicates into one. 10 g of soil sample was then placed in a 50 ml centrifuge tube (Oakridge, Thermo-Fisher Scientific, Waltham, MA), and hydrated with 35 ml of deionized water. These tubes were placed on a rotational shaker for 15 min at 60-rpm and then centrifuged at 1069 gravitation for 30 min. The supernatant was removed from the tube using a vacuum filtration system fitted with 0.22 µm polycarbonate filter. The filtered supernatant constitutes the water-extractable organic matter fraction (WEOM) and was frozen for further analysis. The remaining solids were further treated with 0.5% hexametaphosphate solution and 12 glass beads shaken at 60 rpm for 18hr. This procedure was designed to disperse both macro and microaggregates, enabling the fractionation of particulate organic matter (POM, > 53 µm) and the mineral-associated organic matter (MAOM, < 53 µm). After 18-hours, solutions were passed through a fine sieve (< 53 µm), and rinsed with DI water until the water ran clear. Both POM and MAOM fractions were then oven-dried at 60°C for 24 hours and then weighed for their respective masses. These samples were sent for TC analysis following previously described method. Mass recovery during the fractionation process was typically ~80%. The observed mass loss is likely due to the disruption of coarse woody debris in the compost treatments by the vacuum filtration system.

Wet aggregate stability indices (WASI) were measured using the SLAKES app (University of Sydney – Soil Health Institute). The app tracks aggregate disintegration over a 10 minute period by comparing the difference in the amount of pixels occupied by the aggregates from the start to the end of the test (Fajardo et al. 2016). Briefly, for the test,10-mm sized aggregates were collected from each treatment, and placed in a petri-dish on a gridded background. A phone camera was mounted above the petri dish, and the petri dish was then filled with water. The SLAKES app was set to record the disintegration of aggregates over 10 minutes after submersion. After the recording period, the app calculates the WASI. WASI is delivered on a continuum of values ranging from 0 to 1, with higher values indicating greater aggregate stability.

Carbon mineralization is increasingly recognized as an important soil health indicator, and was included in this study to observe microbial activity of each treatment (Laffely et al. 2020). This, in turn, provides important insight into the turnover of POM in the soil and how treatments control this factor. Carbon mineralization was measured following the method of Joshi Gyawali et al. (2019). Briefly, 40 g of air-dried soil was hydrated with 20 ml of DI water in a 250 ml flask, then immediately sealed with a rubber stopper. A syringe was inserted into the stopper and pumped three times to ensure adequate mixing of air prior to gas analysis. 1 ml of extracted gas was injected into a Qubit S150 infrared gas analyzer with a N_2 gas carrier at 150 ml per min (Qubit Systems, Kingston, ON, Canada). CO_2 concentrations (ppm) were measured by taking the integral of the curve observed by the gas analyzer as recorded into the Logger Pro software (Vernier Science Education, Beaverton, OR). Gas samples were collected at 0, 8, 24, and 32 hours to measure the changes in CO_2 flush following sample rehydration. The rate of CO_2 flush over time was calculated by subtracting the 0-hr ppm values from the 32-hr ppm values and then converting to µg of CO_2 by applying the ideal gas law. This value was divided by the number of hours and the size of the sample in kg to obtain the µg CO_2 per kilogram per hour.

Finally, to determine the texture of the ultramafic tailings, a subsample was collected, and the particle size was analyzed using a Malvern Mastersizer 3000 at NALS. The sample was treated with a dispersant at 2300 rpm at 50% sonication for 10 min prior to running. Samples were run in five replicates to obtain averages. Texture was classified based on particle size distributions corresponding to the typical sand/silt/clay particle size, following CSSC guidelines. Particle size analysis aids in understanding aggregation and organic carbon occlusion in the tailings.

Tree stem height was measured at the base at both the start and end of the trial to assess growth via changes in height. The absolute growth rate of the trees was calculated by dividing the change in stem height by the number of days the experiment was conducted, and then multiplied by 96, the average number of frost-free days in Fort St. James (Environment Canada) to approximate the annual growth rate observed in-situ. Given the extreme alkalinity observed (see section 3.2.1 in Chapter 2), no further analysis was conducted on the ash treatments.

2.3. Statistical Analysis

All data soil carbon data and plant biomass was subjected to Shapiro-Wilks tests for normality and Levene's test for homoscedasticity in the 'rstatix' package (Kassambara 2019). Factorial ANOVA followed by Tukey's post-hoc analysis was used to compare treatment means for stem height, TC, TN, C:N ratios, C mineralization and WASI. Tree stem height ANOVAs included species, proportion, and amendment type as the predictor variables. After it was found that ash would not be an optimal amendment, TC, TN, C:N ratios, C mineralization and WASI, were assessed using organic matter proportion and tree species as the predictor variables. Linear regressions examined stem change in relation to TN, TC, respiration, and C:N ratios using Pearson's correlation coefficient with a 95% confidence interval. Statistical analyses were conducted in R 4.3.1 (R Core Team, 2023).

Due to the lack of replication, POM and MAOM data were not analyzed statistically. Carbon content for each fraction was calculated by multiplying the TC values by the corresponding fraction mass recorded after fractionation. The proportions of POM and MAOM were determined by dividing each fraction's mass by the total accounted mass at the end of the trial and multiplying by 100.

3. *Results*

3.1. *Soil Carbon dynamics*

The acid fumigation technique used to measure total inorganic carbon in the technosols produced small and highly variable percentages (ca. 0.01%), meaning no statistical analysis could be conducted. Thus, the rate of mineral carbonation could not be directly assessed during this trial period.

TOC varied significantly with proportion of compost incorporated (*Fig 28)*, with the 12.5 – 50% compost treatments showing significantly lower in TOC levels compared to the 75-100% compost treatments ($F_{6,63}$ = 641.1, $p < 0.001$). A similar trend was observed for TN ($F_{6,63}$ = 795.1, $p < 0.001$) where the 12.5-50% compost treatments had significantly lower TN compared to those receiving 75 – 100% compost (*Fig. 28a)*. Tree species did not have any significant effects on TC, TN, or C:N ratio. C:N ratios ranged from 12.35 to 16 (mean of 13.0), with the highest values observed in negative controls (mean 15). Again, no notable variation in C:N ratios was attributed to tree species.

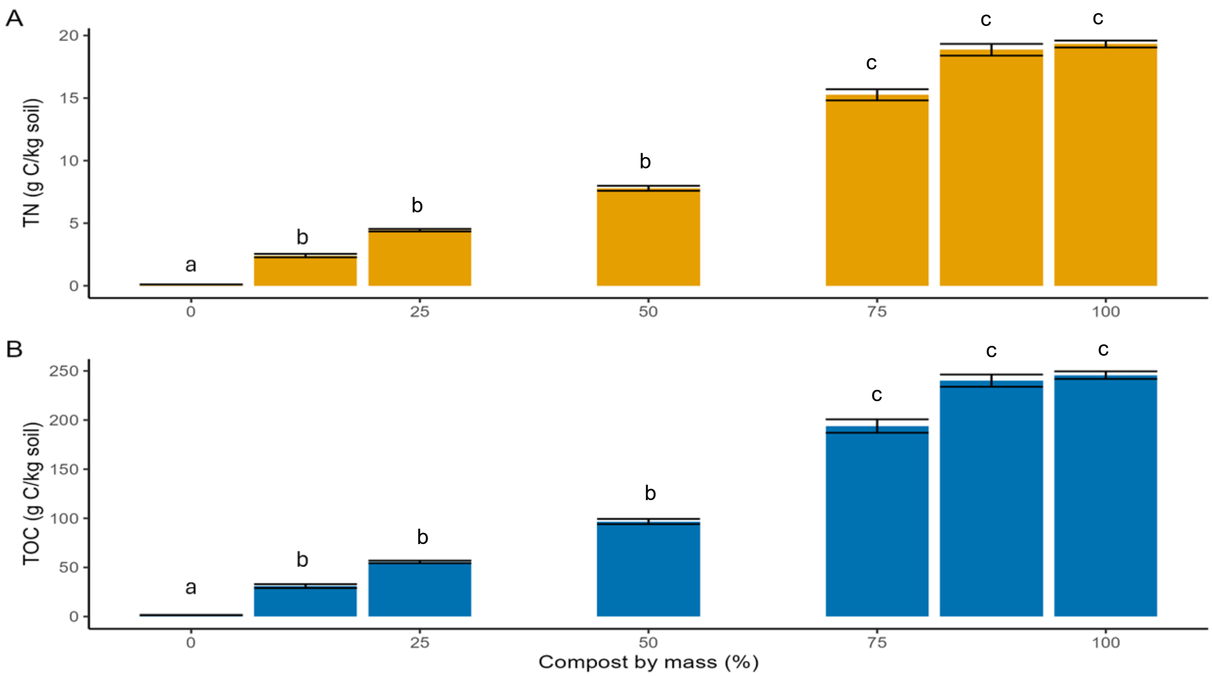

Figure 28. Relationship between proportion of compost and TN (Plot A) and TOC (Plot B). Errors bars indicate ± SE (n = 10).

Carbon mineralization rates increased significantly with a higher proportion of compost ($F_{6,56}$ = 4.9, $p < 0.001$). Tree species only impacted carbon mineralization at the 75 and 100% compost treatments ($F_{1,56}$ = 7.9, $p < 0.05$), with spruce at the 100% compost treatments showing the highest respiration, exceeding the negative controls by up to 40 times (*Fig. 29*). Compost proportions formed distinct groups: 0-25% compost treatments had the lowest mineralization, the 50% treatments were intermediate, and the 75-87.5% treatments showed the highest rates. Linear regression analysis showed that carbon mineralization was positively correlated with TC and TN (R^2 = 0.897, and R^2 = 0.896 respectively, F_{68} = 278, $p < 0.05$, *Fig. 30*).

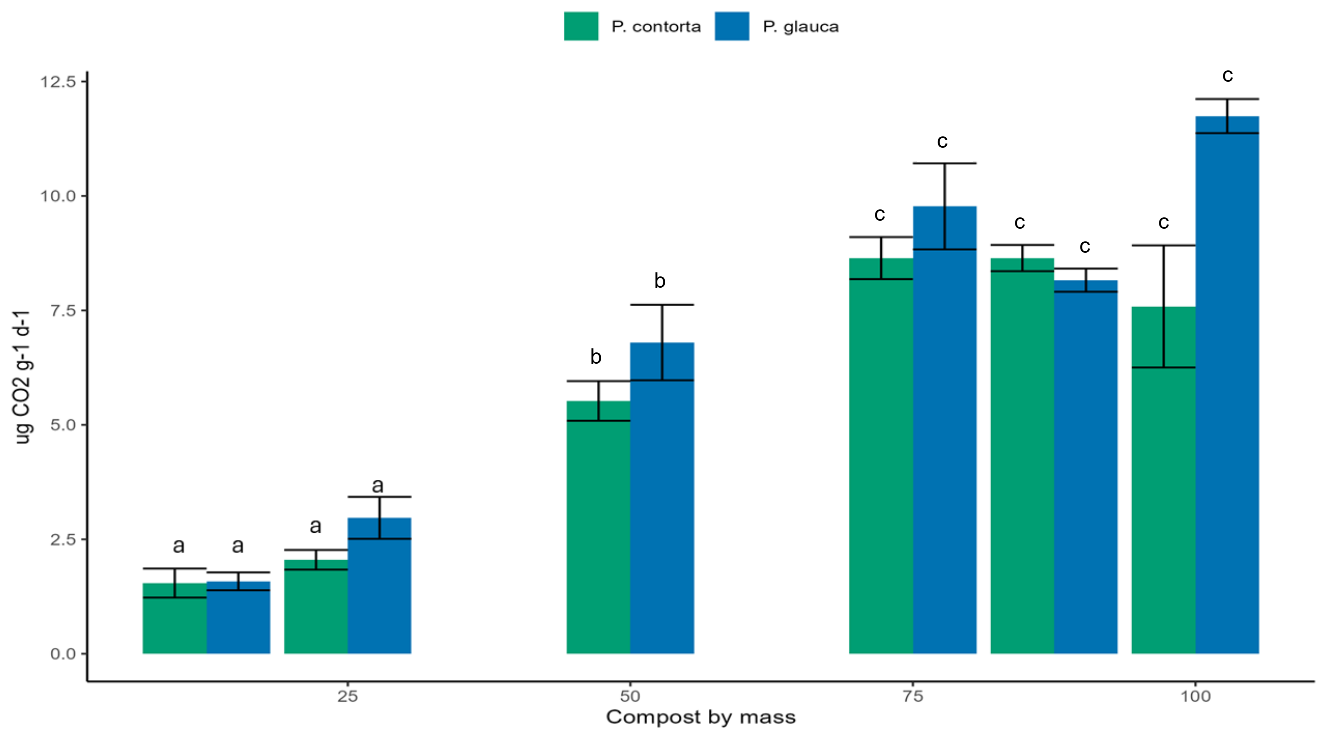

Figure 29. Mean C-mineralization from tailings as it relates to tree species and compost proportion. Error bars indicate ± SE (n = 5).

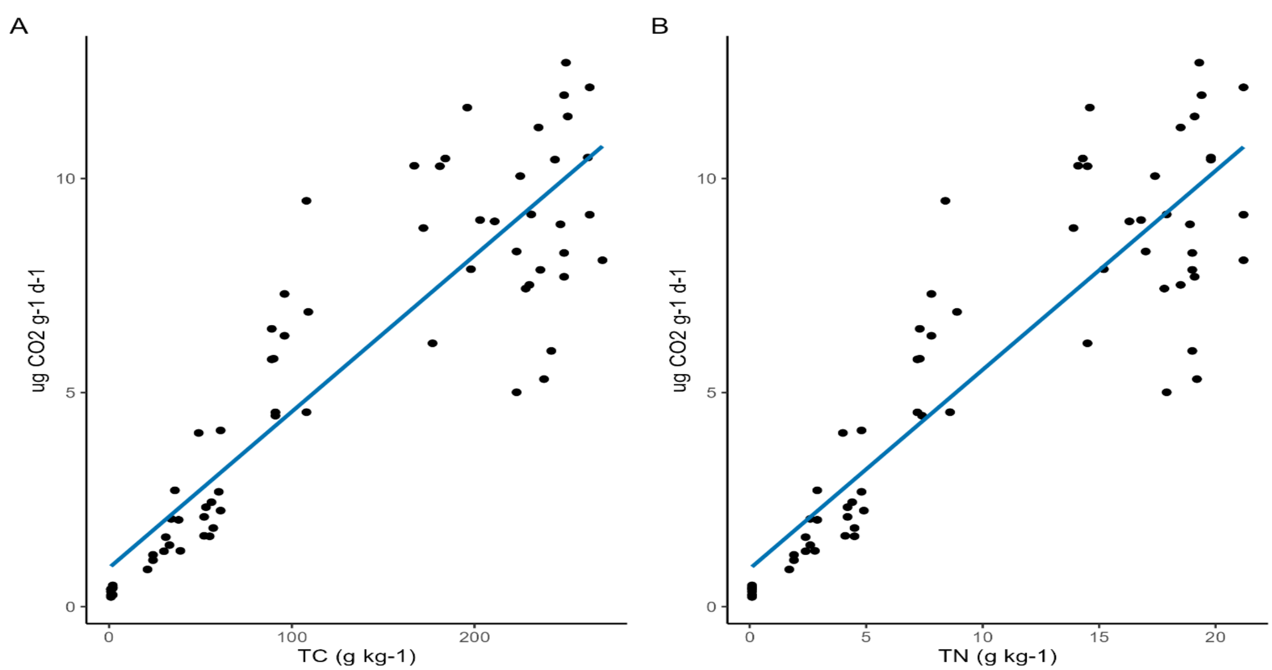

Figure 30. Relationship between C-mineralization and total carbon (Plot A) and total nitrogen (Plot B).

The Par1-Den method was challenging to apply to technosols receiving 75% or greater compost by mass. The lack of minerals and lower bulk density of the compost made it difficult to adequately fractionate the samples, especially at the WEOM step. The filter in the vacuum pump would rapidly become clogged with DOC, making it difficult to get a fully representative sample. Since anything greater than 75% compost by mass does not represent a logistically realistic application rate, these samples were not observed through the Par1-Den method. In addition, the unamended tailings were not initially included in the analysis of the fractionation of organic matter as there was no organic matter incorporated. Thus, the focus of the results on SOM fractionation will be on technosols receiving 12.5% - 50% compost by mass.

Spruce treatments with 50% compost by mass demonstrated the highest MAOC value (249.6 mg C), nearly double that of the next highest treatment (pine with 25% compost by mass, 136.5 mg C). POC content was higher than MAOC content across all treatments (*Fig. 31*), with pine at the 50% compost displaying the highest POC value (471.7 mg-C). Spruce with 12.5% compost by mass demonstrated the highest proportion of MAOM (32.91%), followed by the pine and spruce at 50% compost treatment (24.28% and 20.25% respectively). However, the 12.5% spruce treatment appears to be an outlier (*Fig. 32)*.

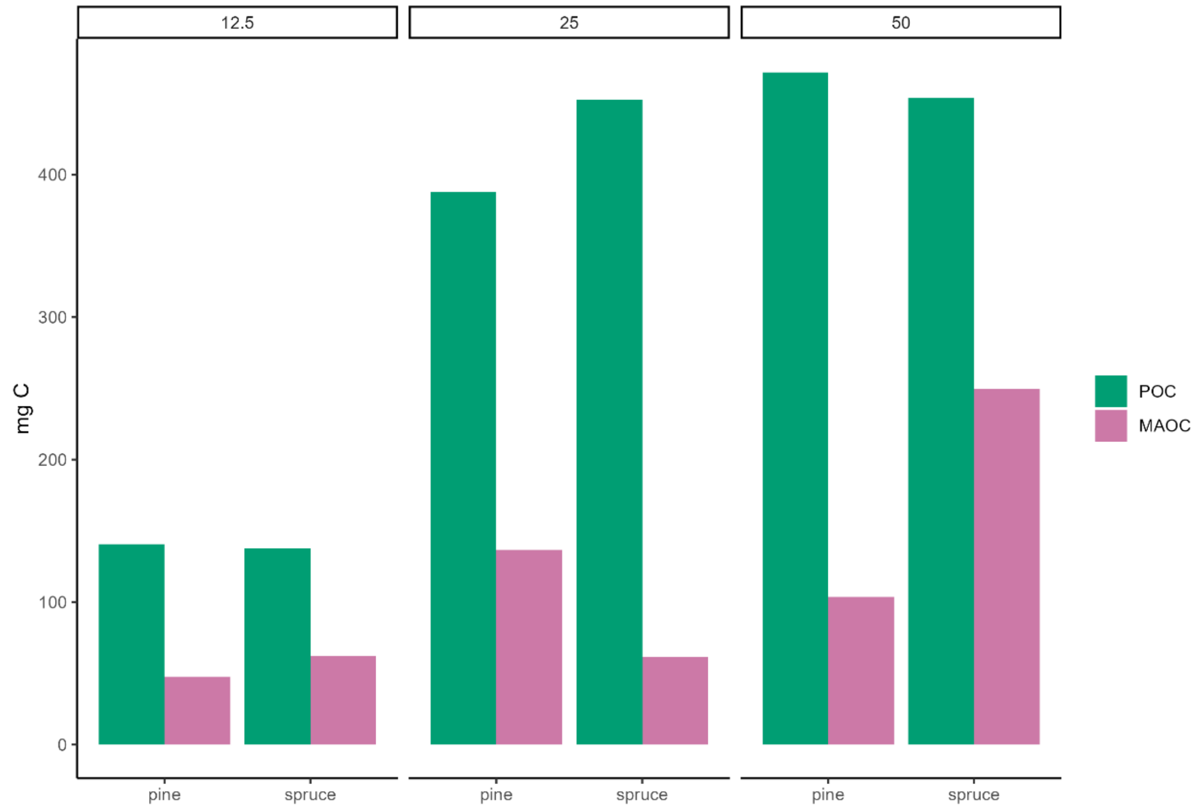

Figure 31. Carbon concentrations in the POM and MAOM fractions of compost-amended technosols, determined using Par-Den method with a 10 g subsample.

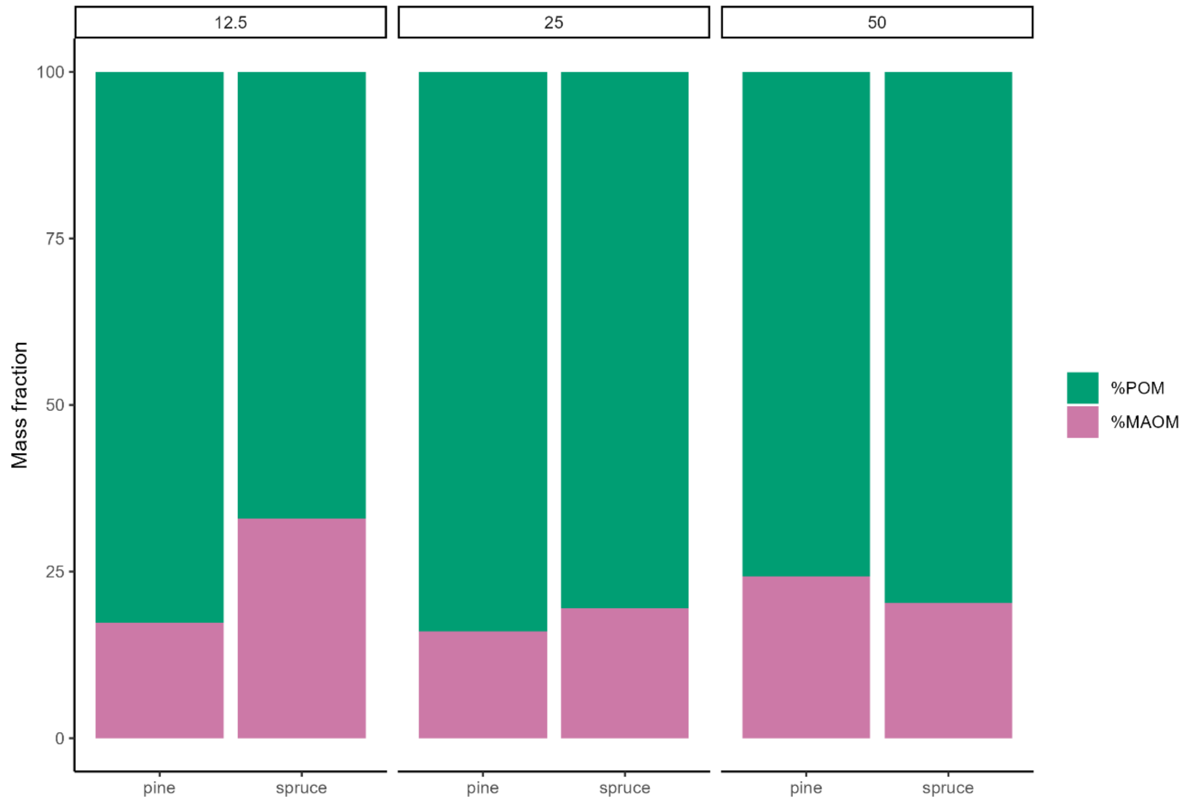

Figure 32. Proportion of organic matter attributed to POM and MAOM based on a 10 g subsample analyzed using the Par-Den Method.

Based on the average total carbon content and the corresponding mass fractions, the proportion of total technosol mass occupied by each organic matter fraction was calculated. This provides a basis for estimating potential organic carbon sequestration, discussed further in Chapter 4, Section 2. No obvious differences between tree species were observed, so data were averaged across species (*Fig* 33). The technosols receiving 50% compost by mass demonstrated the highest POC (75.3 ± 6.1 g/kg) and MAOC (21.4 ± 0.8 g/kg) values, follow by the 25% technosols (POC = 45.7 ± 1.3, MAOC = 9.9 ± .88), and the 12.5% treatments (POC = 23.01 ± 0.5, MAOC = 7.9 ± 3.1).

3.2. Texture and aggregate stability

The mine tailings exhibited a texture primarily composed of silt (48.8 ± 0.6%), followed by sand (39.3 ± 0.8), and clay (11.9 ± 0.1%). This results in a texture that is at the boundary between a 'loam' and a 'silt loam' on the soil texture triangle.

Aggregates primarily appeared as subangular blocky clods, occasionally reaching up to 22 cm in diameter, albeit this size occurred rarely (*Fig. 33*). However, the treatments receiving 75–100% compost did not regularly form aggregates and were challenging to handle when collecting POM and MAOM fractions. Consequently, the analysis of aggregate stability focused on the unamended tailings and the treatments receiving 12.5–50% compost. Aggregate stability indices did not significantly change among treatments and had an average value of 0.3, except for one outlier in the spruce treatment at 25% compost (*Table* 1).

Table 2. Mean (± S.E.) wet aggregate stability indices (WASI) of the four proportions (0%-50% compost) and the two conifer species (P. glauca and P. contorta) (n = 5). Parentheses indicated standard error (n=5).

	Mean WASI	
Proportion	*P. glauca*	*P. contorta*
Unamended Tailings	0.272 (0.01)	0.306(0.01)
12.5%	0.294 (0.01)	0.278 (0.01)
25%	0.414 (0.06)	0.288 (0.01)
50%	0.296 (0.02)	0.324 (0.02)

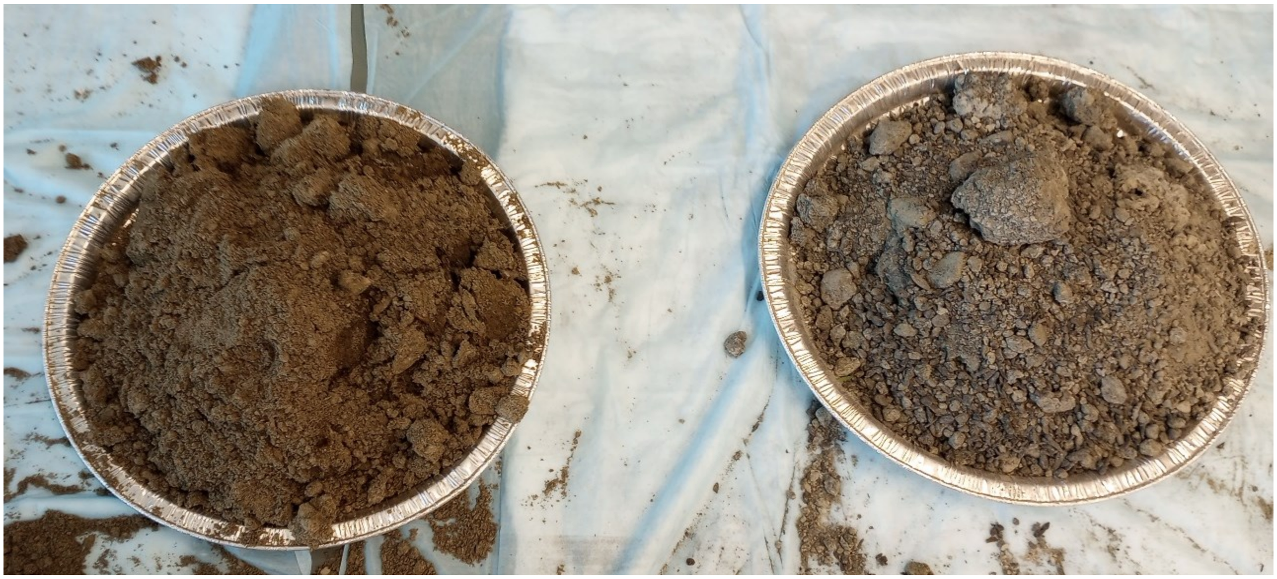

Figure 33. Examples of the aggregates observed within the compost technosols. The pan on the left contains aggregates from the 12.5% compost treatment and the pan on the right shows a sample from the negative controls.

3.3. Tree Growth

Tree growth was most influenced by amendment type and species, with proportion showing no significant effect (*Fig 34*). *P. contorta* showed significantly greater stem heigth increase than *P. glauca* ($F_{1,120} = 23.8$ $p < 0.05$), growing 1.5 times larger regardless of amendment type. *P. contorta* grown in municipal compost showed the highest mean growth, growing 1.34 times greater than the unamended tailings, and the ash treatments over the course of the experiment. *P. glauca* grown in the ash treatments showed the lowest growth, while it grew 1.8 times more in the compost compared to the ash, and unamended treatments. Due to the negative impacts of ash, discussed in section 3.2.1, further analysis will focus on compost treatments.

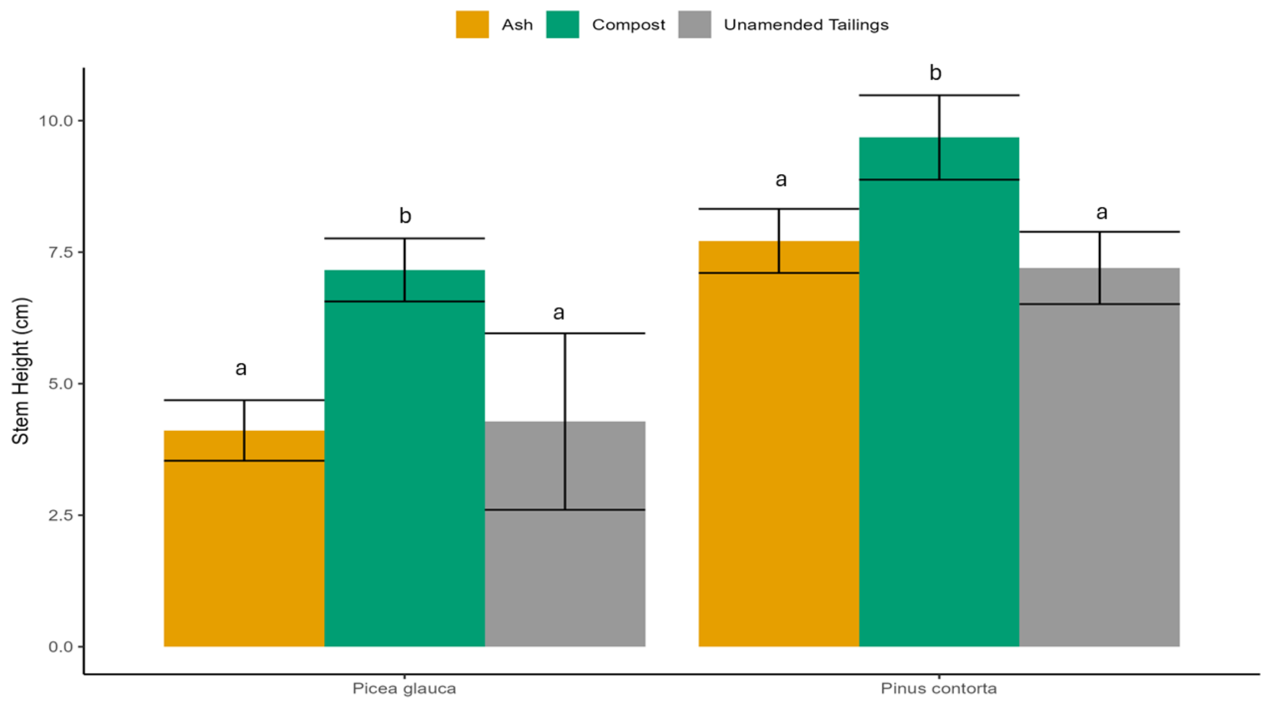

Figure 34. The mean change in stem height by conifer species and amendment treatment. Errors bars indicate ± SE (n = 65 for the amended treatments, n = 10 for the unamended tailings).

Annual growth rate estimates aligned with stem height change results, where amendment and tree species were found to be the most significant determinants of tree growth rate ($F_{122} = 24.1$, $p < 0.001$). *P. contorta* growth rates were 1.5 times greater than those of *P. glauca* regardless of amendment (*Fig. 4*). Compost-amended technosols showed 1.5 times greater growth rates compared to the controls and ash treatments, regardless of tree species (*Figure 35*). No differences were observed between ash and control treatments. Proportion of organic amendment did not significantly affect the growth within the trial.

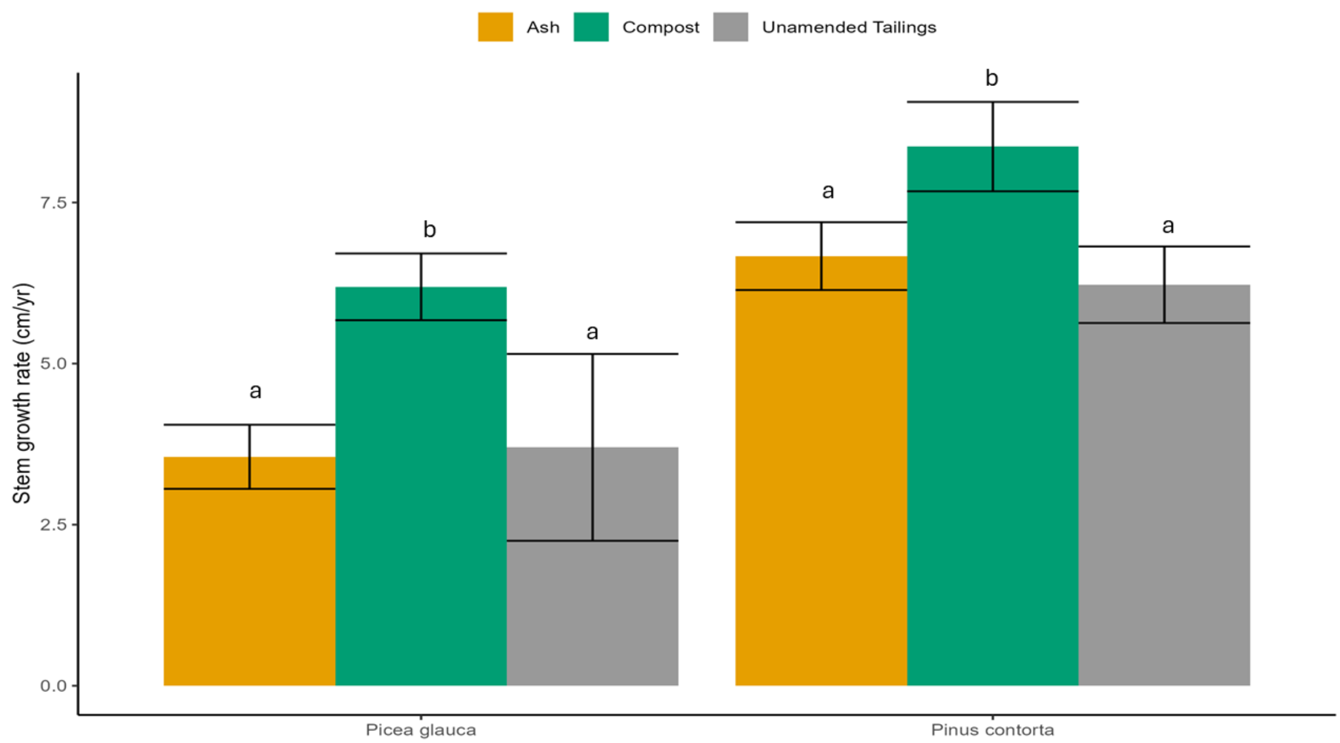

Figure 35. Estimated annual growth rates for conifer species based on amendment treatment. Errors bars indicate ± SE (n = 65 for the amendments, n = 10 for the unamended tailings).

4. *Discussion*

4.1. Carbon Occlusion

The results from the aggregate stability, organic matter fractions, and carbon mineralization suggest that organic matter proportion does not significantly impact carbon occlusion below 50% compost by mass.

All the treatments between 0 – 50% compost by mass did not display any variability between tree species and proportion, suggesting that the mere presence of the ultramafic minerals themselves drives the formation of aggregates. While this consistency is remarkable, the relatively low clay content of the ultramafic technosols leads to lower stabilities compared to more weathered soils (Six et al. 1998; Flynn et al. 2020). Clay minerals are crucial to the formation of aggregates, as their high specific surface area and net negative charge act as a binding agent for organic ligands (Vogel et al. 2014). With a lower clay content, aggregates are only weakly adhered to the coarser particles within the soil, a phenomenon observed in another mine tailing study (Saiyed and Goh (2009). In addition, the short time span of the experiment limits tailings exposure to the binding action of plant roots. The correlation between aggregate stability and plant cover is well-documented; the roots and microbes within the rhizosphere excrete substances that bind soil particles together, thereby enhancing aggregation (Poirier et al. 2018). The macroaggregate stability observed within the ultramafic technosols was similar to those observed in other short-term alkaline technol studies (Li et al. 2022). This suggests that, much like naturally occurring soils, the aggregate stability of the ultramafic technosols can increase through the action of biota and the formation of secondary minerals.

Despite the lower clay content of the ultramafic technosols, MAOC managed to form in observable quantities within the timeframe of this study (*Fig.* 9). While the proportion of POC increased consistently with the compost proportions, the proportion of MAOC remained relatively stable. This suggests that the minerals within the tailings are reactive enough to adsorb organic ligands, and that the proportion of incorporation does not affect its short-term formation. This partially confirms my initial hypothesis; the proportion of organic matter corresponds to a stable MAOC formation before rapidly declining between 50% to 75% compost. Treatments receiving greater than 50% compost were not able to be fractionated due to their disperse nature, likely due to the overwhelming amount of organic matter compared to mineral surfaces. This would also explain the lack of water stable aggregates formed at this proportion. Given that anything above 50% compost by mass is unfeasible for in-situ application, this decline does not pose any major constraints to organic carbon sequestration potential on the reclaimed tailings.

While MAOC is observable within this study, the methods employed in this experiment could not provide insight into the mechanism. There are two possibilities. The first is ‘cation bridging', which has been observed to be the main driver of MAOC formation within the ultramafic technosols. 'Cation-bridging' usually occurs when a polyvalent cation acts as a bridge between two negatively charged particles, usually clay particles or organic ligands (Bronick and Lal 2005; Rowley et al. 2018b). The cation in question is highly pH-dependent; alkaline soils tend to showcase cation bridges mediated by Ca^{2+} or Mg^{2+}, which form inner-sphere and outer-sphere complexes with the target particles (Falsone et al. 2016; Rowley et al. 2018b). Mg^{2+} forms weaker outer-sphere complexes than Ca^{2+} in serpentine soils, which may explain the lower aggregate stability observed in the ultramafic technosols (Falsone et al. 2016). It should be noted that cation-bridging results in weaker mineral-organic associations compared to those of Al-phyllosilicates and Fe-oxides, found in more acidic soils (Kleber et al. 2015; Falsone et al. 2016).

However, ultramafic soils in-situ have had a much longer amount of time to form these cation-bridges, which might not be possible to replicate within the length of time this trial. Thus, its possible that the MAOC fraction observed within these technosols constitutes the carbon fraction contained within microaggregates, rather than truly chemically complexed. MAOC often requires the presence of reactive secondary minerals, such as Fe-Al oxides and clay minerals (Kleber et al. 2015). Considering that the tailings constitute the primary minerals of the ultramafic parent material, it is unlikely that the length of time of the trial was adequate for these for reactive species to form. Thus, the observed MAOC within these technosols likely corresponds to the carbon *physically* occluded within microaggregates, rather than *chemically* bound to mineral surfaces. Future studies should employ XRD mineralogy to observe the mineral phases of the tailings, as this would provide valuable information about the organic sequestration potential of the minerals themselves.

4.2. Carbon Mineralization

Carbon mineralization rates were strongly correlated with the proportion of organic matter (*Fig. 29*). This trend reflects an increase in nitrogen and labile carbon (*Fig. 30*), consistent with findings from Franzluebbers et al. 2000. In addition, conifer species influenced mineralization rates, but only at higher organic matter proportions (*Fig. 29*). This aligns with other studies observing tree species variations in mine soils (Mukhopadhyay et al. 2013; Chodak et al. 2019). Despite ultramafic technosols' higher alkalinity, C-mineralization rates at 50% compost were comparable to those in lignite and tantalite deposits experiencing afforestation, and even some endemic forests (Józefowska et al. 2017; Neina et al. 2017; Chodak et al. 2019). However, the unamended tailings and technosols with 12.5% - 25% compost showed lower C-mineralization rates than the aforementioned studies. It should be noted that these values were 24-32 hours after rewetting, meaning in-situ respiration rates are likely much lower (Józefowska et al. 2017). This fits well with the findings on in-situ serpentine soils, which typically exhibit reduced C-mineralization compared to non-serpentine substrates (Aka Sagliker et al. 2018). Previous research on serpentine soils and polluted soils highlight that even small quantities of Ni and Cr can significantly affect microbial community structures, enzymatic activities and rates of mineralization (Aka Sagliker et al. 2018; Ma et al. 2022). While Ni leachates were minimal, Cr levels were inconsistently elevated (see *Section 2.3.)*. It is likely that microbes in the ultramafic technosols are adsorbing or accumulating heavy metals, which could potentially disrupt metabolic activity in sufficient quantities (Fashola et al. 2016). However, microbes endemic to serpentine soils demonstrate adaptations to high metal stress (Power et al. 2009; Rajkumar et al. 2009). Considering that amendments introduced to the Baptiste tailings displayed distinct structures of microbial communities, these communities may not yet be adapted to the heavy metal stress of ultramafic tailings (Ball 2023). As discussed in *Section 3.4.1.,* incorporating compost derived from organic matter native to the Baptiste site into ultramafic technosols could introduce microbial communities better adapted to the heavy metal stress of the tailings. To optimize amendment strategies for re-storing ecosystem productivity, a comparative analysis of C-mineralization rates between municipal compost and site-specific organic matter, alongside assessment of genetic markers for heavy metal uptake and stress, would provide valuable insights.

4.3. Mineral carbonation

Due to the low and inconsistent levels of inorganic carbon in the tailings, this study could not conclusively address whether organic amendment application affected mineral carbonation rates. This could potentially be a limitation of the acid fumigation methodology employed. Power et al. (2020) observed that inorganic carbon levels increased as brucite converted to dypingite, with the maximum percent of inorganic carbon reaching about 0.2% around the 150 days — a timeline similar to the tree mesocosm experiment in my study. In the mesocosm study, TC in the unamended tailings ranged from 0.1% to 0.2%, aligning with the values reported by Power et al.'s study. Similar findings from other in-situ mineral carbonation studies (Gras et al. 2020) support the likelihood that carbonation is occurring here as well. Given that the carbonation of brucite acts as a cementing agent, this process could also account for the formation of the macroaggregates described in *Section 4.2.1* (Bronick and Lal 2005).

It is, however, equally likely that another stochastic factor influenced the small quantities of TIC. The tree root balls may have introduced residues that appeared in trace amounts within the medium. Power et al. (2020)'s study also noted that the distribution of brucite was uneven within the tailings, meaning that the tailings received for this study might have been especially brucite-poor. While other serpentine minerals have the potential to be carbonated, they are much more recalcitrant to this transformation compared to brucite (Lacinska et al. 2016; Power et al. 2020). Natural mineral carbonation occurs over decades to centuries timescales, meaning that the timeline of this study was not adequate to observe unabetted carbonation (Monger et al. 2015). Carbonates may precipitate but then rapidly dissolve in the compost treatments due to the presence of organic acids exuded by plants and microbes, meaning that there is a potential for reprecipitation at depth. However, without corroborating methodology, it is difficult to discern the cause of carbon variability in the unamended tailings. The variability within the mesocosm study appears to have hindered the accurate measurement of total inorganic carbon content.

To prevent future inconsistencies in carbonation results, I recommend using more robust methodologies for assessing the inorganic fraction than acid fumigation. Power et al. (2020) employed coulometry, which showed more consistent results. It should be noted that their study introduced CO_2 through direct injection in very controlled conditions, which may have contributed to this consistency. Techniques like scanning electron microscopy and quantitative XRD mineralogy would better characterize the mineralogical composition of the tailings and provide further evidence of mineral transformation within the ultramafic technosols. Quantifying DOC and carbonate ion content in the leachate or pore water would be more direct evidence of carbonate dissolution and precipitation. Currently, the link between organic matter amendment and mineral carbonation remains inconclusive.

4.4. Species response

The proportion of organic matter did not significantly affect the growth of trees across amendment treatments. This corroborates the findings in Section 2.3 and aligns with other previous studies on in-situ mine spoils. Jack pine (*Pinus banksiana*) growth has been shown to be tied closely to organic matter content on coal deposits, but the increase in biomass diminishes after 5-10% organic matter (Farnden et al. 2013). Similarly, studies with native plants and grasses showed diminishing returns in canopy cover with higher organic carbon inputs, further confirming the results observed in both the leaching column and the conifer mesocosm studies (Young et al. 2015; Gil-Loaiza et al. 2016). As discussed in Section 2.4, the diminishing returns on plant growth with increasing organic matter can be explained by the 'functional equilibrium' hypothesis, which suggests that plants allocate more biomass to shoots when nutrients are abundant (Poorter et al. 2012). In all the aforementioned studies, increasing organic matter content corresponded with elevated plant nutrient concentrations, suggesting that organic matter influences plant growth through enriching the nutrient pool available to plants (Farnden et al. 2013; Young et al. 2015; Gil-Loaiza et al. 2016).

The link between organic matter and nutritional content also explains the reduced growth observed in ash-treated technosols (*Figure 2*). As described in Section 3.4, the ash treatments contained far less plant nutrients and created alkaline conditions that were prohibitive to plant growth. Thus, the benefits of organic matter incorporation depend on the amendment's nutrient content. However, this does not rule out the potential of ash as an amendment, as previous studies have shown its effectiveness in moderate applications as a liming agent (Park et al. 2012). Given that the ultramafic technosols already display circumneutral to alkaline conditions, ash application regardless of proportion, does not appear to be a suitable amendment (see Section 3.3 for ash pH details). While compost demonstrated the best growth response in this study, studies in similar tailing environments suggest that combining compost with native mineral soils provides greater benefit to trees than using compost alone (Guittonny-Larchevêque and Pednault 2016). Integrating the native topsoil and its organic matter harvested on-site has the potential to further improve reclamation logistics by eliminating the need to transport compost from nearby municipalities. Additionally, using topsoil and endemic composted materials would conserve the indigenous microbial community at the site, which would help with recruitment of native plant species (Rajkumar et al. 2009; McMahen et al. 2022). Future studies should explore ultramafic technosols constructed with organic matter from the Baptiste project site and assess the growth responses compared to municipal compost. These studies should also compare microbial diversity in the endemic organic matter to that of municipal compost to discover if indigenous microbes affect the growth of vegetation in these substrates. Previous studies with the Baptiste tailings showed mixed plant growth responses when using inoculations, but these studies did not use inoculants derived from the site's endemic organic matter (Ball 2023).

Across all amendment treatments, *P. contorta* exhibited a significantly faster growth rate than *P. glauca* (*Fig. 3*). *P. contorta's* resiliency has been well documented; it consistently out performs *P. glauca* in disturbed soils where the forest floor is removed (Kranabetter et al. 2017). *P. contorta* behaves as a pioneering, stress-tolerant species, while *P. glauca* is typically associated with late-stage ecosystems (Kranabetter et al. 2017). The success of *P. contorta* also stems from its mycorrhizal associations, most notably *Suillus spp.,* which enhances N acquisition in nutrient-limited environments (Kranabetter et al. 2006). This would also explain why *P. contorta* is more prevalent on in-situ ultramafic soils, as it can thrive in low-nutrient conditions (Alexander 2020). While *P. contorta* appears to be the best adapted tree species within the context of this study, similar research on mine tailings has shown higher mortality rates for conifers compared to deciduous species such as *Salix spp.* and *Populous* spp. (Larchevêque et al. 2013; Guittonny-Larchevêque and Pednault 2016). Given the relative success of the nitrogen-fixing *Vicio vilonsa* in leaching column experiments, plantings nitrogen-fixing *Alnus spp*. may be effective for reforestation efforts at the Baptiste site. These tree species, along with *Salix spp.*

and *Populous spp.*, are endemic to the area, and their revegetation would spur the formation of an organic layer in the Baptiste technosols (Bergh 2012a). However, considerations of elevation and relief should be taken into consideration for plant selection.

Revegetation with native plants provides an opportunity to create a source of forage for herbivores, supporting habitat creation and aiding in site restoration. The circumneutral pH and minimal heavy metal contaminants observed in the leaching column study suggest minimal risks of bioaccumulation. Plant tissues from the Baptiste project showed little elevated concentrations of toxic heavy metals (with the exception of Cr, was discussed in Section 2.4.), further suggesting that the minimal risk of bioaccumulation (Bergh 2012a). However, future studies should investigate metal concentrations in the tissue of plant grown in constructed technosols to ensure that plant uptake is not altered by the creation of the tailings. These studies should also evaluate the effects of ultramafic technosol construction on a wider range of tree species to help restoration strategists identify the most suitable species to initiate revegetation.

5. *Conclusions*

Ultramafic technosols displayed substantial carbon occlusion within five months of receiving compost application. Proportion did not introduce any significant changes to MAOC and aggregation, except for the treatments receiving greater 50% compost by mass. In this ratios, the overly abundant compost did not have enough reactive polyvalent cations and mineral surfaces to occlude carbon. Tree species did not introduce any effects on carbon occlusion. While proportion did not affect physical carbon occlusion, carbon mineralization was greatly linked to organic matter proportion, likely driven by introduction of labile N and the reduced heavy metal stress. The connection between reclamation treatments and mineral carbonation remains unclear. The lack of evidence for mineral carbonation in these settings might be related to the timespan of the study, or other stochastic variables such as lack of reactive minerals or their rapid dissolution.

The findings of this study align with those from the leaching columns (see *Section 2.3.*), indicating that lower compost application rates yield similar growth responses to higher rates. Ash was not found to be an effective amendment for tree growth. *P.* contorta demonstrated significantly greater growth rates than *P.* glauca. This study further highlights that using lower amendment quantities can improve ecosystem productivity while simultaneously alleviating logistic challenges. Future studies should observe different plant species response to ultramafic technosol construction, which would help to inform effective remediation efforts. Thus, through revegetation and organic amendment application, the Baptiste technosols have high potential to sequester organic carbon through its occlusion within the substrate, and through its immobilization in plant and microbial biomass.

Chapter 4

General Conclusion

4.1. Review

The objective of this study was to assess the risks and benefits of technosol construction at the Baptiste Project. More specifically, I set out to observe how each component of the technosol system impact metal mobility, biomass generation, and carbon sequestration. Observing these key components helps to inform future reclamation efforts and identify risks and benefits that can be addressed in future studies.

Identifying the optimal application rate for organic amendments is crucial to developing cost-effective and impactful reclamation protocols for tailings. It was found that the quantity of organic amendments incorporated was not nearly as consequential as the quality of amendment. Ash technosols did not foster plant growth and proved far too alkaline to warrant further investigation. By contrast, the nutrient-rich compost yielded significantly improved plant growth even at the lowest level of application compared to the unamended tailings. From an implementation perspective, reclamation practitioners can afford to apply compost at a minimal rate and still observe a return to the prior capability.

In addition, the leaching of toxic metals from the technosols appeared minimal, suggesting that their entry into the soil solution is slow and not affected by bioweathering. The one exception to this pattern is Cr, which exhibited consistent leaching in quantities exceeding those acceptable for irrigation water. Future studies should attempt to elucidate the valency of the Cr within the soil solution, as this will better inform reclamation practitioners about the risk associated with its leaching. The limited metal leaching from the ultramafic technosols demonstrates that the process leading to the ultramafic tailings does not introduce risks that are beyond those already present at the site. In fact, the evidence from this study suggests though the removing Ni through the extraction process reduces the risk of heavy metal contamination compared to the existing soils at the site. However, long-term bioaccumulation studies should be conducted to ensure that any heavy metals do not run the risk of moving up the trophic chain at the Baptiste site.

In the context of carbon sequestration, the ultramafic technosols amended with compost displayed impressive carbon occlusion within less than a year after construction. Macroaggregate stability and MAOC formation were not significantly affected by proportion of compost application. This highlights that the lowest compost application rate can bolster plant growth while simultaneously mitigating the lifetime emissions from the mine. However, inorganic carbon sequestration proved to be elusive, likely due to several stochastic and methodological factors. Mineral carbonation still holds potential at the Baptiste site, but this study demonstrates the challenges of inducing this type of mineral transformation in-situ.

The findings from this study spearheads the increasingly global externality posed by ultramafic tailings. As these mineral substrates encounter increased utilization, the findings from this study will provide an important starting point for any team interested in reclaiming these unique substrates.

4.2. Implications

While the short-term nature of this study makes discerning long-term trends difficult, extrapolating long-term trends in ecosystem development and pedogenesis will help clarify important details for implementation of ultramafic tailing reclamation.

When attempting to reclaim the tailings with compost (or similarly composted organic material), the depth of incorporation will determine the quantity of carbon occluded within the technosols. The deeper the compost is tilled, the less exposure to aerobic microbial decomposition and greater potential for macroaggregate and microaggregate formation.

Assuming 12.5% compost by mass, and an area of 5 km^2 (the area the tailings are projected to occupy at the Baptiste project), incorporating compost at a depth of 1 m has the potential to occlude tens of thousands of tonnes of carbon within the first year. The presents the possibility of carbon neutrality for the Baptiste project. The sheer mass of compost required to amend to this scale would pose a logistical challenge, but composted overburden and soil salvage can alleviate such constraints.

In addition to mitigating carbon emissions for the Baptiste Project, incorporating organic material at the surface of the tailing area provides the additional benefit of restoring ecosystem productivity, and kickstarting natural pedogenic processes at the Baptiste Project. Thus, constructing ultramafic technosols has the potential to serve as a multifunctional solution to addressing regulatory constraints for "prior capability" and mitigating the carbon emissions of the Baptiste project, and ultramafic tailings at large.

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