Technosol evaluation for mine site reclamation in the Boreal Shield

by

Renate Vanderhorst

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> Faculty of Graduate Studies Laurentian University Sudbury, Ontario, Canada

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Abstract

Technosols, soils substantially influenced by human activity, are often found on former mine sites. In some cases they develop as a result of natural processes on abandoned sites; in others, they are intentionally manufactured as part of the mine's reclamation program. In this study Technosols with either 40% woody residuals and 60% mine rock or 80% woody residuals and 20% mine rock were constructed and placed over mine rock lysimeters in either 30 cm or 60 cm layers to evaluate their potential for use in the reclamation of a gold mine in the Canadian boreal forest. The Technosol plots were constructed in summer 2012 and have been continuously monitored for water chemistry, soil microclimate, and vegetation health. In 2016 soil pits were excavated to examine the physical, chemical, and microbial development of Technosol profiles.

The high organic Technosols had higher concentrations of bioavailable nutrients, reduced availability of Mo and Cd, less extreme soil temperatures, increased soil moisture, and reduced soil pH compared to the low organic. In all Technosol plot water samples pH was between $7 - 8$, high levels of DOC were measured, and no elements exceeded site compliance limits specified by the Ontario government. Little profile development was observed in the Technosols, but there were differences between comparison field and forest soils in terms of chemistry and microbial functional diversity. Green alder (*Alnus viridis* subsp. *crispa*) grew well on the Technosol plots, but bearberry (*Arctostaphylos uva-ursi*) struggled and after two years almost complete mortality was seen. This is likely due to a combination of factors including high soil pH, low moisture, and low nutrient availability in the Technosols. Overall the high organic Technosol appears more suitable for reclamation, and green alders are a good choice for initial planting.

Keywords: Technosol; mine reclamation; pedogenesis; revegetation; water chemistry

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Table of contents

List of tables

List of figures

[Figure C67. Annual and perennial ryegrass blades upon Technosol plots in \(top\) June 2016 and](#page-189-2) [\(bottom\) July 2016..](#page-189-2) 173

[Figure C68. Concentrations of B \(mg/kg\) in vegetation samples. Source sites are Technosol plots](#page-193-1) [\(1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic,](#page-193-1) 60 cm; 4: 80% organic, 60 cm) [and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-193-1) comparison bearberry). [...](#page-193-1) 177 [Figure C69. Concentrations of Cu in vegetation samples. Source sites are Technosol plots \(1:](#page-193-2) [40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm\) and](#page-193-2) [comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC: comparison](#page-193-2) [bearberry\)..](#page-193-2) 177 [Figure C70. Concentrations of Fe \(mg/kg\) in vegetation samples with alder root sample excluded](#page-194-0) [\(7540 mg/kg Fe\). Source sites are Technosol plots \(1: 40% organic, 30 cm; 2: 80% organic, 30](#page-194-0) [cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm\) and comparison vegetation \(AN: nursery](#page-194-0) [alder, AR: alder root, AS: source alder; BC: comparison bearberry\)...](#page-194-0) 178 [Figure C71. Concentrations of Mn \(mg/kg\) in vegetation samples. Source sites are Technosol](#page-194-1) plots (1: 40% organic, 30 [cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60](#page-194-1) [cm\) and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-194-1) comparison bearberry). [...](#page-194-1) 178

[Figure C72. Concentrations of Ni \(mg/kg\) in vegetation samples. Source sites are Technosol](#page-195-0) [plots \(1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60](#page-195-0) [cm\) and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-195-0) [comparison bearberry\).1 alder Technosol sample < DL \(0.2 mg/kg\)...](#page-195-0) 179

[Figure C73. Concentrations of Zn \(mg/kg\) in vegetation samples. Source sites are Technosol](#page-195-1) [plots \(1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60](#page-195-1) [cm\) and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-195-1) comparison bearberry). [...](#page-195-1) 179

[Figure C74. Concentrations of As \(mg/kg\) in vegetation samples. Source](#page-196-1) sites are Technosol [plots \(1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60](#page-196-1) [cm\) and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-196-1) comparison bearberry). [...](#page-196-1) 180

[Figure C75. Concentrations of Sb \(mg/kg\) in vegetation samples. Source sites are Technosol](#page-196-2) [plots \(1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60](#page-196-2) [cm\) and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-196-2) comparison bearberry). [...](#page-196-2) 180

[Figure C76. Concentrations of Cd \(mg/kg\) in vegetation samples. Source sites are Technosol](#page-197-0) [plots \(1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60](#page-197-0) [cm\) and comparison vegetation \(AN: nursery alder, AR: alder root, AS: source alder; BC:](#page-197-0) comparison bearberry). [...](#page-197-0) 181

List of abbreviations

ANOVA: analysis of variance AWCD: average well colour development DL: detection limit DOC: dissolved organic carbon *E:* species evenness EC: electrical conductivity Gravity through-flow: water flowing through Technosol plot collected from barrel at base *H:* Shannon Diversity Index MISA: Municipal Industrial Strategy for Abatement MMER: Metal Mining Effluent Regulations OD590: optical density at 590 nm pH_w : soil pH measured in 18 M Ω deionized water $pH_{Ca}:$ soil pH measured in 0.01 M CaCl₂ PWQO: Provincial Water Quality Objectives *R:* species richness Tukey HSD: Tukey Honest Significant Differences Technosol treatments 1: 40% organic woody residuals & 60% mine rock in 30 cm Technosol layer 2: 80% organic woody residuals & 20% mine rock in 30 cm Technosol layer 3: 40% organic woody residuals & 60% mine rock in 60 cm Technosol layer 4: 80% organic woody residuals & 20% mine rock in 60 cm Technosol layer

Tension water: pore water; water collected from leachate sampling plate within Technosol plot

Chapter 1. Technosols and reclamation

Introduction

Mining and land reclamation

The expanding range and variety of human-impacted land and the increasing global awareness over past decades of the value of natural ecosystems (Brandt, Flannigan, Maynard, Thompson, & Volney, 2013) has lead to an increase in research and regulation aiming to mitigate damage and return land to a more natural state. Mining companies typically chose to reach this more natural state through reclamation, which establishes a self-sustaining ecosystem on the land. This is in contrast to restoration, which returns land to an ecosystem resembling the one which was originally present (Bradshaw, 1997; SER, 2004). Reclaimed ecosystems are often designed with specific goals in terms of ecosystem function, vegetation assemblage, and future land use (SER, 2004).

Mining, particularly surface mining, can severely disturb large areas through the deposition of waste materials such as tailings and mine rock, and by the associated removal of vegetation, topsoil, and geological materials (Turcotte, Quideau, & Oh, 2009). This impact effectively returns the land to a state where primary succession may occur (MacKenzie $\&$ Quideau, 2010). In the past mine sites in Canada could simply be abandoned once mining had finished (Bradshaw, 1997) but today there are specific legislative requirements mandating their reclamation. In Ontario, mine sites are not considered closed out until a self-sustaining vegetation cover is established, and monitoring of sites must continue until this point (Mine Rehabilitation Code of Ontario, 2012).

In order for most sites to be successfully revegetated, a soil cover must first be added to the site (Macdonald et al., 2015). Soils are a crucial component of ecosystems; not only do they

support vegetation, they are highly complex systems which regulate energy and water storage and mediate the activity of organisms responsible for ecosystem functions such as nutrient cycling and productivity (Voroney & Heck, 2015). Mining companies often stockpile the topsoil they remove before starting operations to use as their soil cover (Gaster, Karst, & Landhäusser, 2015; Sorenson, Quideau, MacKenzie, Landhäusser, & Oh, 2011) or they may construct a soil from a combination of stockpiled overburden materials and organic materials such as peat (MacKenzie & Quideau, 2010). Soils can also be constructed from tailings material with additions of organic material, often in combination with inorganic fertilizers (Young, Renault, & Markham, 2015).

Unlike natural soils, these manufactured soils are likely to have few active soil organisms and unusual physical and chemical characteristics, along with potential toxicity problems (Leguédois et al., 2016). Because manufactured soils are heavily human-influenced, they are classified separately from natural soils in the World Reference Base for Soil Resources (WRBSR)vas Technosols (IUSS Working Group WRB, 2014). Technosols can be found in all types of human-altered environments, not simply mines (Huot, Simonnot, & Morel, 2015); however, their presence on mine sites means that Technosols formed from mining activities have been the focus of a large portion of research, and this will likely continue (Capra, Ganga, Grilli, Vacca, & Buondonno, 2015). As their properties can be very different depending on their parent materials (Huot, Simonnot, et al., 2015), an examination of the literature provides a preliminary understanding of common themes in their development and characteristics.

Technosols

A Technosol is a soil which 1) contains more than 20% by volume of artefacts, which are substances created, modified, or moved by human activity, in either the upper 100 cm of soil or

to a continuous rock layer or technic hard material, and without a layer which qualifies as another soil or a continuous rock or cemented layer within 10 cm of the surface; or 2) has a continuous impervious or semi-impervious geomembrane within 100 cm of the soil surface; or 3) has a technic hard material such as asphalt or concrete within 5 cm of the surface (IUSS Working Group WRB, 2014). The Technosol Reference Soil Group was originally added to the WRBSR in 2006 as a result of the expanding global range of human-altered soils; they can be found in urban areas, roads, landfills, mine sites, and military sites, among others (IUSS Working Group WRB, 2006). Technosol research has increased in the past few decades, with many studies looking at their physical-chemical properties and evolution (Capra et al., 2015). Most Technosols can be considered young soils, geologically speaking, but in many cases they appear to develop rapidly (Leguédois et al., 2016), meaning early pedogenic trends are observable and some projections can be made about future evolution.

Pedogenesis of natural soils is controlled by the five soil-forming factors of parent materials, climate, organisms, relief/topography, and time (Jenny, 1941). The interaction of the first four factors through time and over varying spatial scales determines the nature of the soil that is present in a given area (Bockheim, Gennadiyev, Hammer, & Tandarich, 2005). Human activity, including the anthropogenic origin of the parent materials, the placement of the materials, the disturbances they experience, and management activities, is considered a sixth soil forming factor in the pedogenesis of Technosols (Leguédois et al., 2016).

The accumulation of parent materials is the first step in soil genesis (Simonson, 1959). The soil parent materials are very important in determining the characteristics a soil can have and are particularly important at the regional scale (Bockheim et al., 2005). A soil can be expected to inherit basic physical and chemical properties from parent materials, influencing properties such

as soil texture and pH (Brady & Weil, 2010). As Technosol parent materials consist of artefacts, substances either strongly altered by humans or taken from one environment to another where they would not naturally occur, they may have unusual characteristics such as very high or low pH, high levels of contamination with metals or organic compounds (Leguédois et al., 2016), or minerals with different compositions than is usual for soils under the same conditions (Huot, Simonnot, et al., 2015).

Climate controls the weathering of the parent materials; high temperatures and levels of precipitation accelerate pedochemical weathering by increasing leaching and runoff, and promoting higher levels of microbial activity and plant growth (Brady & Weil, 2010). Technosols are associated with populated areas by their very nature, which means they are often in climates which are relatively warm and moist, subjecting them to higher levels of weathering (Leguédois et al., 2016).

The presence of organisms, both vegetation and microorganisms, also influences the weathering of soils, as well as controlling the input and cycling of nutrients and organic matter in the soil (Brady & Weil, 2010). Initially Technosols have very little biological activity, however once there is activity accumulation and humification of organic matter begins (Huot, Simonnot, et al., 2015) and changes in soil dynamics and structure can be seen (Leguédois et al., 2016). Climate and vegetation effects operate on large, continental scales, but the effects of organisms on soil formation are also seen on the microscopic level (Bockheim et al., 2005).

The topography of a site influences soil formation at a landscape level (Bockheim et al., 2005) and can change the magnitude of the effects of climate and vegetation. For example, soils at the base of a slope receive increased water inputs while soils on slopes erode more easily (Brady & Weil, 2010). The influence of topography on Technosols is typically weak, however,

due to the leveling and filling which often occurs when the initial materials are placed on a site (Leguédois et al., 2016).

In terms of time, as has been stated above, Technosols are recent soils. This, along with their presence in generally favourable climates and the fact that their parent materials are not yet in equilibrium with their environment, suggests they could have rapid initial pedogenic development (Leguédois et al., 2016).

In all soils, the five soil-forming factors govern processes within the soils which control horizon differentiation, the second step in soil genesis (Simonson, 1959). Horizon differentiation happens through the addition, removal, transfer, and transformation of materials, such as organic matter, clay minerals, carbonates, sesquioxides, and soluble salts, within the soil system (Simonson, 1959). These four major processes operate through a variety of simpler processes, including hydration, leaching, precipitation, and dissolution (Simonson, 1959). The relative importance and balance of processes determines the nature of the soil (Simonson, 1959).

Typically the first processes which occur in Technosols are leaching of soluble compounds, mineral transformations, and organic matter accumulation (Huot, Simonnot, et al., 2015), though the specifics vary with the Technosol conditions. For example, in a ten-year old Technosol formed from excavated Callovo-Oxfordian claystone Na^+ and K^+ were already leaching out of the upper 80 cm, while gypsum formation was detected in the lower layers of the Technosol (Scholtus et al., 2015). These and other processes observed were evidence of rapid weathering of the parent material, bringing the Technosol profile closer to natural profiles in the region formed from similar material (Scholtus et al., 2015). Scalenghe and Ferraris (2009) found increased biological activity, organic carbon, aggregation, and structure, as well as lower pH, in the upper layers of earthy material deposited behind crib walls four years after installation. A

forty-year old Technosol designed in the same manner had developed further, with six observable horizons (O/A/AB/Bw/BC/C) (Scalenghe & Ferraris, 2009). Séré et al. (2010) observed the rapid development of additional horizons in their constructed soils, as well as dramatic changes in compaction and weathering of soluble minerals, over the course of their three-year study. On the other hand, Huot et al. (2013) found slower evolution in Technosols developing on blast furnace sludge, attributed in part to the vertical heterogeneity of the soil resulting from multiple dumping events, with high water retention preventing transfers and interactions between the layers. High levels of metal contamination and toxicity can slow the development of Technosols, possibly by reducing rates of colonization by vegetation and soil fauna (Ciarkowska, Gargiulo, & Mele, 2016).

Deliberate amendment additions can help improve the physical and chemical properties of the Technosol and add nutrients commonly not present in the mineral components (Rokia et al., 2014). Using amendments, Technosols can be designed for specific purposes and areas, such as urban landscaping (Koolen & Rossignol, 1998) and reclaiming mine- and industry-impacted areas (e.g. Sanborn, Bulmer, & Coopersmith, 2004; Séré et al., 2010).

Technosol use in mine reclamation

Although Technosols formed from mine waste material without the addition of organic material have been seen to support vegetation (Ciarkowska et al., 2016) and to perform ecosystem services such as water regulation (Huot, Séré, Charbonnier, Simonnot, & Morel, 2015), the sites examined in these studies have been abandoned for $70 - 400$ years, too great a period for modern mining companies. The addition of organic material can speed the process considerably and thus make it a viable option for reclamation; in fact, this method is being used in the Alberta oil sands (e.g. Sloan, Uscola, & Jacobs, 2016; Sorenson et al., 2011; Turcotte et

al., 2009). Chosen amendments typically have local origins to keep costs low, and include vegetation and peat removed during site opening (MacKenzie & Quideau, 2010). When these are unavailable or not present in large enough quantities for the entire site, industry waste products such as paper mill sludge and wood chips (Young et al., 2015) or sewage sludge (Bradshaw, 1997) are also used.

Organic amendments not only add nutrients and organic material, they can improve bulk density (Sanborn et al., 2004), aeration and water retention/infiltration (Young et al., 2015), aggregation and structure (Larney & Angers, 2012), and reduce the mobility of toxic compounds (Hattab, Motelica-Heino, Faure, & Bouchardon, 2015). However, amendments must be carefully selected if the objective is to reduce contamination issues, as they have also been shown to increase leaching of dissolved or colloidal materials in some cases (Hattab et al., 2015; Yao et al., 2009) and sometimes can contain contaminants (Elkhatib & Moharem, 2015). Even without toxicity problems it can take many years for soil microbial communities in Technosols to resemble those of natural soils, and the amendments used to create the soil have an important influence on their development (Dimitriu, Prescott, Quideau, & Grayston, 2010; Larney & Angers, 2012). The speed at which nutrients are available for plant and microbial use varies and depends on the quality and origins of the organic amendment used. Treatments received by amendments before being incorporated into the Technosol also have an effect on nutrient availability (Larney & Angers, 2012). Commercial fertilizers are often added alongside amendments for several years to meet initial nutrient requirements to boost vegetation establishment and growth (Sloan et al., 2016) and combinations of amendments are frequently required (Larney & Angers, 2012). In addition to consideration of amendment physical and

chemical properties, transport costs and availability often dictate what can be used, particularly in remote mines sites.

Consideration must also go into the vegetation species selected for establishment on the Technosol. Often a fast-growing cover of annual grasses and forbs is quickly established on reclamation sites to stabilize soils, retain nutrients, and provide organic matter input (Macdonald et al., 2015). However, if non-native plants are used for the cover they may be very difficult to remove and replace with native vegetation later in the reclamation process (Pinno & Hawkes, 2015; Young et al., 2015). Legumes and other nitrogen-fixing species such as alder are often planted to add nitrogen to the soils (Bradshaw, 1997; Macdonald et al., 2015), and droughtsusceptible Technosols may require species which can tolerate dry periods (Bradshaw, 1997). Other physical barriers may also be present, such as high bulk density and low porosity due to compaction, which can prevent root growth (Stumpf, Pauletto, & Pinto, 2016). Chemical restrictions such as pH extremes may mean species should be chosen based on tolerance limits, such as acid-tolerant species for sulphidic mine wastes (Yang, Liao, Yang, Chai, & Li, 2016).

If contaminants are present in the Technosol, their effects on plant growth must be considered. Plants are continually exposed to contaminants in soil through their root systems, which is where the highest concentrations will be found (Wanat, Joussein, Soubrand, & Lenain, 2014). Although some common metal contaminants are essential micronutrients, such as copper, in high concentrations symptoms of toxicity are seen (Nagajyoti, Lee, & Sreekanth, 2010). Some soluble metal and metalloid contaminants are toxic to most plants at low levels, like chromium. For others such as arsenic, some plants have evolved systems to suppress uptake and transform it into less toxic forms (Nagajyoti et al., 2010).

Apart from direct problems arising from toxicity, the effects of vegetation on contaminant concentrations can vary. Some studies have found that grass covers reduce the leaching of contaminants, while others have seen no effect and still others have found increases in the leaching of organic contaminants such as β-endosulfan (Dousset, Ondo Zue Abaga, & Billet, 2016). Different plants uptake contaminants at different concentrations. In one study on contaminated gold tailings, the uptake of arsenic, antimony, and lead by *Graminea* spp. was higher than found in the birch, horsetail, or fern species tested (Wanat et al., 2014). The location of the contaminant within the plant tissue can also be a concern; for example, though *Festuca pratensis* successfully produced a dense cover on a Technosol developed from steel mill wastes, the concentration of molybdenum in its shoots was higher than permitted maximum levels for forage, making exposure through herbivory possible (Oustriere et al., 2016). Sometimes plants can be harvested to prevent the return of contaminants to the soil after senescence. This approach has been done with wetland plants such as *Typha latifolia* to completely remove contaminants from the site after they have been sequestered by the plants (Jeke, Zvomuya, Cicek, Ross, & Badiou, 2015). However, this may require a degree of management that is difficult to maintain in remote mine sites after closure.

All these considerations aside, vegetation can survive and ecosystem development can occur on Technosols associated with mining activities. With careful planning of the materials used in construction and the vegetation chosen for planting, the use of Technosols remains a good option for mine reclamation.

Using Technosols to reclaim a gold mine

Rationale

Despite the increasing use of Technosols in mine reclamation there are still many questions that are yet to be answered. Studies examining Technosols which have developed on mine wastes without intervention (e.g. Ciarkowska et al., 2016; Huot, Séré, et al., 2015) often miss their early development. Many studies focus on the fate of contaminants in the system, whether it be by looking at stabilization (e.g. Fang, Tsang, Zhou, Zhang, & Qiu, 2016), leaching and through-flow (e.g. Jordán et al., 2017), or vegetation uptake (e.g. Oustriere et al., 2016). Studies in the Alberta Oil Sands have less of a focus on contamination and look more at microbial and vegetation community development (MacKenzie & Quideau, 2010; Pinno & Hawkes, 2015), but as they are all examining Technosols with the same parent materials in the same region results may not be applicable to all Technosols. Therefore, long-term studies with continuous monitoring must be conducted on Technosols of various types to provide a more complete understanding of their behaviour and effectiveness.

The current study was designed to provide the opportunity to monitor the development of Technosols designed for mine reclamation from construction onwards. Using local mine and forest industrial waste products, the goal was to observe conditions important for reclamation, such as trace metal concentrations in through-flow and vegetation growth, and Technosol profile development through time.

Study site

The study was conducted on a gold mine in northern Ontario, specifically Barrick Gold's Williams Mine in Hemlo, Ontario. This mine consists of both an open pit and underground mine, which has resulted in the generation of a large pile of mine rock; the need to revegetate this pile was the original driver of the study.

The mine is located in the Canadian Shield, in the eastern part of the Schreiber-Hemlo greenstone belt of the Wawa subprovince, part of Archean Superior province (Muir, 2002). The deposit has experienced large amounts of deformation and metamorphism and is unusual for an Archean deposit in that it has no mafic volcanic rocks and lacks major quartz and carbonate veins in the mine sequence (Muir, 2002), although there are locally present carbonate-rich ores in the main orebody and western extension (Pan & Fleet, 1995). There is a significant amount of molybdenite throughout the deposit and pyrite is also common (Pan & Fleet, 1995). The metasedimentary and intermediate volcanic rocks which form a large part of the rock pile are considered non-acid generating; they have a large neutralization potential due to the presence of carbonate minerals.

As part of the boreal forest ecosystem, the forest is dominated by a mixture of coniferous species including black spruce (*Picea mariana* (Mill.) B.S.P.), jack pine (*Pinus banksiana* Lamb.), tamarack (*Larix laricina* (Du Roi) Koch), and balsam fir (*Abies balsamea* (L.) Mill.), with deciduous hardwoods such as trembling aspen (*Populus tremuloides* Michx.), white birch (*Betula papyrifera* Marsh.), willow (*Salix* sp.), and alder (*Alnus* sp.) (Sims, Baldwin, Kershaw, & Wang, 1996; Zoladeski & Maycock, 1990). Podzols are the dominant soil type in the region, characterized by a reddish illuvial spodic (Bhf/Bf) horizon rich in Fe/Al overlain by a bleached eluvial (Ae) horizon from which these elements were leached (IUSS Working Group WRB, 2014). Mean monthly temperatures range from 15°C in July and August to -14°C in January; precipitation varies from 122 mm in September to 47 mm in February (Environment Canada, 2017).

Study design

Initial research determining which combinations of mine rock and local organic amendments were most able to support plant growth was done by Watkinson, Lock, Beckett, & Spiers (2016). Using crushed non-acid generating intermediate volcanic or metasedimentary rocks from the Williams mine rock pile in combination with woody residuals from the former Domtar White River Sawmill (White River, ON) or primary paper sludge from Tembec Pulp Inc. (Marathon, ON), a series of Technosols with varying organic contents were created. A 10-week growth chamber study with annual ryegrass was conducted on the various Technosols, with the results suggesting Technosols made from the mine rock combined with the woody residuals were the best option for use at the mine scale studies (Watkinson et al., 2016).

Based off these results, two Technosols were constructed in summer 2012 using woody residuals combined with the crushed, non-acid generating intermediate volcanic and metasedimentary rocks in either an 80:20 or 40:60 ratio by volume, giving high organic and low organic Technosols. These Technosols were then placed on test lysimeters constructed in the following manner: an impermeable geomembrane-lined berm was covered with a layer of coarse mine rock followed by a layer of crushed mine rock, then capped with either 30 cm or 60 cm of one of the Technosols. The surface area of the plots once the Technosol was placed was approximately 4 m x 4 m, with each Technosol-depth combination replicated three times for a total of twelve plots. At the base of each lysimeter a drainage tube was attached to the geomembrane and connected to a large holding vessel designed to collect water samples representative of the through-flow from the plot (Watkinson, 2014).

Within the Technosol layer of each system a series of sensors was installed three weeks after plot construction at multiple depths to monitor soil microclimate conditions. 5TM soil temperature/moisture sensors (Decagon Devices) were initially installed at 10 cm, 30 cm, and, in

the thick treatments, 60 cm depths. In July 2014 5TM sensors were added at 5 cm depths in nine of the plots; the remaining three had 5 cm sensors added in July 2015. One MPS-2 water dielectric potential/temperature sensor (Decagon Devices) was installed in each plot at either 30 cm or 60 cm, depending on the treatment thickness. There was also a soil leachate sampling plate made of porous borosilicate glass (UMS, SPG120 Leachate Sampling Plate) installed in each plot approximately 5 cm above the Technosol/mine rock interface to sample plant root-available water held in the Technosol (Watkinson, 2014). The leachate sampling plates were connected via tubes to 1 L bottles stored in boxes below ground-level. There were three sample bottles plus one empty bottle per box, and they were connected to a single system which allows the bottles to be placed under a vacuum to draw water out of the plots into the bottles. Three rainwater collectors were also present on site.

In summer 2014 two comparison soil sites were selected for sensor installation to provide a comparison to the plot microclimate measurements. The first of these was the field immediately behind the plots; this field was once covered with infrastructure including buildings and parking as part of the Newmont Golden Giant Mine, but has since been reclaimed using a combination of stockpiled soil, fertilizer, and MTO grass and legume seed mix. This site is referred to as the successional field, and has 5TM sensors installed at 5 cm, 10 cm, 15 cm, 30 cm, and 60 cm depths. The second site is a little over 1 km from the site, in an upland secondary forest stand. 5TM sensors were installed here at 5 cm, 10 cm, 15 cm, 20 cm, and 25 cm, just above where the soil meets bedrock. A climate station was also installed in summer 2014, located between the centre two plots, to record air temperature and relative humidity at 2 m (VP-3 Humidity/Temperature sensor, Decagon Devices) and precipitation (ECRN-100 Precipitation sensor, Decagon Devices).

Sixteen green alders (*Alnus viridis* subsp. *crispa* (Aiton) Turrill) were planted on two of the three replicates of each treatment in 2013, and more were planted in 2015 to bring the total number of surviving alders on each vegetated plot to nine. Green alders were selected because they are native nitrogen fixers capable of growing on disturbed sites (Lefrançois et al., 2010). They were obtained from roadsides in the area. Also in 2015, twelve bearberry plants (*Arctostaphylos uva-ursi* (L.) Spreng.) were planted on each of the vegetated plots. Bearberries are a low-growing shrub which forms a ground cover on dry soils low in nutrients (Krpata et al., 2007); they are also native to the area. Bearberry plants were purchased from Connon Nurseries, Waterdown, ON.

Previous findings

Work on the Technosol plots was conducted in 2013 and 2014, focusing on the soil moisture and temperature regimes of the four Technosol plots types, as well as initial vegetation survival rates. The results indicated that though none of the plot moisture contents dropped below the permanent wilting point, there may not be sufficient moisture for long-term successional growth, particularly on in the low organic Technosol plots (Watkinson, 2014). The high organic content and 60 cm Technosols covers also showed more of an insulating effect against temperature extremes, though all Technosols still dropped below 0°C in winter (Watkinson, 2014).

Alder survival rates were low after the 2013 planting; by the end of that summer the highest mean survival rate in a Technosol treatment was 43% in the 80% organic, 30 cm plots. The 40% organic, 60 cm plots had the lowest mean survival rate (13%). The differences in the plots appeared to have been due to the differences in the soil moisture content; plots with higher moisture particularly at 10 cm depth had higher survival rates. The timing of the planting

(August) and the method of transplant (bare root) were also though to have contributed to the low alder survival rates (Watkinson, 2014). Only 18 of these original alders survived to 2015, a survival rate of 14%.

Objectives of this study

The objectives of this study were to continue the evaluation of the ability of a Technosol constructed from local mine rock and woody residuals to be used in the revegetation of that mine, and to monitor biopedochemical development through time. Chapter 2 of this thesis focuses on the dynamics of chemistry of the Technosol plots, monitoring the changes in chemical parameters such as pH and concentrations of trace elements in soil porewater and gravity through-flow over four years. Results are contrasted to the initial soil chemistry of the Technosols. In Chapter 3, changes in the Technosol profiles with depth are examined. Observations of the profile development, the soil temperature and moisture through time, and the microbial functional diversity within the profiles are documented. Chapter 4 describes the health and survival of the green alders and bearberries planted on the Technosol plots, and includes an analysis of the nutrient concentrations present in the leaves of the two species.

Chapter 2. Chemistry of a mine rock Technosol in the Boreal Shield

Introduction

Technosols are defined as soils which are heavily influenced by human activity, typically having parent materials which have been created, modified, or brought to the surface by industrial activities such as mineral extraction (IUSS Working Group WRB, 2014). Technosols can be formed from mine waste materials such as tailings (Santini $\&$ Fey, 2016) and waste rock (Ciarkowska et al., 2016), or stockpiled soil, vegetation, and geologic materials removed during site establishment. The transformation of these materials can occur slowly through natural pedogenic processes (Ciarkowska et al., 2016; Huot et al., 2013) or be facilitated through human intervention. Intentionally constructed Technosols frequently contain organic amendments such as peat (Audet, Pinno, & Thiffault, 2015), wood chips or paper mill sludge (Young et al., 2015), and sewage sludge (Asensio, Covelo, & Kandeler, 2013) to improve soil conditions for vegetation growth. They often play a large role in mine reclamation, as part of the reclamation process involves returning soil material to sites which have been stripped of overlaying vegetation, soil, and geological materials prior to site revegetation (MacKenzie & Quideau, 2010).

Due to their industrial origins, Technosols can contain metals and other contaminants in concentrations which are harmful to both human health and the environment (Huot, Séré, et al., 2015; Yao et al., 2009). In addition to the elements within the mine waste itself, the effect of the organic amendments on the mobility and release of toxic trace elements is not straightforward and depends on the amendment used. Additions of chipped wood have been seen to decrease trace element mobility while composted sewage sludge decreased the mobility of some elements and increased others (Hattab et al., 2015). Sewage sludge has been found to increase the leaching of Cu and Zn, as well as other metals depending on chemical characteristics such as pH,

electrical conductivity, and redox conditions; the sludge treatment process has also been seen to have an impact (Yao et al., 2009). Biochar can act as a metal immobilizer (Puga, Melo, de Abreu, Coscione, & Paz-Ferreiro, 2016) but again the source of the organic material used to create the biochar has an impact on its chemical and physical properties (Fang et al., 2016).

With these considerations in mind, site-specific studies must be conducted on Technosols constructed from mine rock with organic amendments intended for use in mine reclamation. The current study was conducted on Technosols consisting of mine rock from a gold mine in northern Ontario, combined with wood waste from a local lumber mill. Assessments of the total and bioavailable content of elements within the Technosol were done not only provide information about the potential environmental risks, but give information on the nutrient status of the Technosol, a potentially limiting factor to plant growth on these soils (Young et al., 2015). Water through-flow samples were examined to document element leaching profiles from the Technosol into surrounding surface or groundwater. Soil pore water samples were taken to determine the chemical composition of water in direct contact with plant roots (Concas, Ardau, Di Bonito, Lattanzi, & Vacca, 2015).

Methods

Study area

The Williams Mine is a gold mine owned by the Barrick Gold Corporation and located in Hemlo, ON, north of Lake Superior and approximately 350 km east of Thunder Bay. It consists of both an open pit and underground mine. The mine is situated in the Canadian Shield, in the eastern part of the Schreiber-Hemlo greenstone belt of the Wawa subprovince, part of Archean Superior province (Muir, 2002). There is a diverse set of metals including antimony, arsenic,

barium, mercury, molybdenum, and vanadium present in the Hemlo deposit, with no mafic volcanic rocks and a lack of major quartz and carbonate veins (Muir, 2002).

The soils of the area are typically Podzols, having a reddish illuvial (Bhf/Bf) horizon rich in iron and aluminum that has been leached from the overlying grey eluvial (Ae) horizon (IUSS Working Group WRB, 2014). As part of the boreal forest ecosystem, the dominant trees of the region include black spruce (*Picea mariana* (Mill.) B.S.P), jack pine (*Pinus banksia* Lamb.), balsam fir (*Abies balsamea* (L.) Mill.), trembling aspen (*Populus tremuloides* Michx.), and white birch (*Betula papyrifera* March.) (Sims et al., 1996; Zoladeski & Maycock, 1990).

Site design

Two Technosols were manufactured in summer 2012 by combining woody residuals and crushed non-acid generating mine rock in fixed ratios (40:60 and 80:20 by volume). The residuals included sawdust, bark, and off-cuttings of boreal coniferous trees, and were produced by the White River Forest Products sawmill which is approximately 60 km east of the mine site (Watkinson, 2014). These Technosols were then placed on test lysimeters in the following manner: an impermeable geomembrane-lined berm was covered with a layer of coarse mine rock followed by a layer of crushed mine rock, then capped with either 30 cm or 60 cm of one of the Technosols. Each Technosol-depth combination was replicated three times for a total of twelve plots (Watkinson, 2014); the area of the plots was approximately 4 m x 4 m.

Attached to the geomembrane at the base of the lysimeter was a drainage tube connected to a large holding vessel designed to collect water samples representative of the through-flow from the plot. Water samples taken from these barrels are referred to as gravity through-flow. There was also a soil leachate sampling plate made of porous borosilicate glass (UMS, SPG120 Leachate Sampling Plate) installed in each plot approximately 5 cm above the Technosol/mine

rock interface to sample plant root-available water (soil pore water) held in the Technosol. These samples are referred to as tension water. The leachate sampling plates were connected via tubes to 1 L bottles stored in boxes below ground-level. There were three sample bottles plus one vacuum bottle per box, and they were connected to a single vacuum system for sample collection. Three rainwater collectors were also present on site.

Green alders (*Alnus viridis* subsp. *crispa*) were planted on eight of the twelve plots in 2013 and again in 2015 to bring the number on each up to at least nine. At this time twelve bearberries (*Arctostaphylos uva-ursi*) were also planted on each of the vegetated plots.

Near the research plots were mine rock weathering test cells operated by the Williams Mine. These cells were set up in a similar manner to the Laurentian ones, but lack soil cover. They were designed to monitor the release of elements from the uncovered mine rock piles through time.

Soil sampling and analysis

Samples were collected from all plots following construction in July 2012 and analysed for moisture, CNS, and total and bioavailable ions. Samples were sent to the Forest Resources & Soils Testing Laboratory (Lakehead University) for CNS analysis (Elementar Vario EL Cube).

Samples measured for total ions were sieved and the 2 mm portion ground to ensure homogenization. 0.5 g of sample was weighed into 50 mL Teflon™ tubes and run through a 4step digestion process:

- 1) 10 mL of 9:1 concentrated HF: concentrated HCl added; samples cooked for 210 min at 112°C until dry
- 2) Step 1 repeated
- 3) 7.5 mL concentrated trace HCl and 7.5 mL concentrated trace HNO³ added; samples cooked for 250 min at 112°C until dry
- 4) 0.5 mL concentrated HF, 2 mL concentrated trace HCl, and 10 mL concentrated HNO₃ added; samples cooked for 60 min at 112°C

Samples were allowed to cool before being diluted to 50 mL with 18 $\text{M}\Omega$ deionized water.

To measure bioavailable ions, 3 g of the 2 mm portion of sieved soil was weighed into Falcon[™] tubes and 30 mL of 0.01 M LiNO₃ was added. Samples were placed on the shaker overnight at 200 rpm and centrifuged the next day at 2000 rpm until most particulates were removed from the supernatant. Samples were then gravity-filtered through Whatman® 42 filter paper and the supernatant collected and stored at 4°C until analysis.

Both total and bioavailable samples were quantified by quadrupole ICP-MS (Varian 810) using normal sensitivity mode optimized to maximize the signal-to-noise ratio. To correct for mass bias and calibration drift an internal standard solution containing 10 μg/L of Be, Re, Ru, was bled into the sample uptake line using a glass T-shaped mixing chamber (Glass ExpansionTM). Spikes, duplicates, and certified reference materials (TILL-1, LKSD-2 (NRCan); Montana II Soil NIST® SRM® 2711a) were included for quality control.

Elements monitored throughout the study include the macronutrients calcium, magnesium, potassium, and phosphorus; the micronutrients boron, copper, iron, manganese, molybdenum, nickel, and zinc; and the trace elements arsenic, antimony, and cadmium. These last three are elements of interest to the mine (along with molybdenum) due to the presence of molybdenite, arsenopyrite, realgar, and stibnite in the deposit (Muir, 2002).

Statistical analysis was performed using R 3.3.2 (R Core Team, 2016). Moisture, CNS, and bioavailable nutrient results were compared between treatments and organic content levels using analysis of variance and independent t-tests. When calculating mean bioavailable values, samples below detection limit were excluded from the calculations; elements with means where this occurred are noted in the text.

Water sampling and analysis

Water samples were collected once in October 2012 (where available) after the initial site set-up was completed. Water sampling trips were then made three times between the months of May and October for 2013, 2014, and 2015. In 2016 sampling trips were made once monthly from May to November.

Two gravity through-flow samples were collected in 500 mL jars from each barrel during sampling trips. Water samples were also collected when possible from the Williams mine rock cells in the same manner as for the gravity through-flow samples. Tension water samples were obtained by pressurizing the bottles with a hand pump to -0.5 PSI and leaving the bottles to collect water overnight. In 2016, bottles were pressurized to -0.75 PSI with a vacuum pump (UMS, VacuPorter) before being left overnight. Rainwater samples were collected each trip and the collectors emptied. In 2013 natural water samples were taken from the White River, a rain gauge in White River, and the Aubrey Falls 'public spout' on Highway 129. Samples were refrigerated and transported to Laurentian University for filtering and subsequent analysis.

The pH and electrical conductivity of the unfiltered water samples were analyzed with a pH combination electrode (Accumet) and a conductivity probe (Accumet) attached to an Accumet (AB15) meter and Accumet (AB30) meter, respectively. Quality control included a duplicate analysis and recalibration of pH, EC, and Eh instruments approximately every 20 samples.

Water samples were analyzed for the anions Cl^- , NO_3^- , and SO_4^2 after being filtered through 0.45 µm filters by ion chromatography ($DX-120$, 0.5 M/0.5 M Na₂CO₃/NaHCO₃ eluent).

Quality control samples consisted of a blank, duplicate, spike, and certified reference material (QCSPEX-AI, Fisher Scientific) approximately every 20 samples.

Dissolved organic carbon water samples were filtered and acidified to 2% with trace metal grade HCl. DOC was then quantified by the non-purgeable organic carbon (NPOC) method on a Shimadzu TOC-5000A/5050A TOC-analyser. Quality control included method blanks, duplicates, and internal standards.

The concentration of dissolved ions was analysed by ICP-MS, as described above. Water samples were filtered through 0.45 µm filters and acidified with trace metal grade HNO₃ to 2% acid prior to analysis. Quality control samples consisted of a blank, duplicates, two spiked samples, and certified reference water material (TMDA 51.3). The same elements examined in the soil samples were chosen for the water chemistry (Ca, K, Mg, P; B, Cu, Fe, Mn, Mo, Ni, Zn; As, Cd, Sb).

Results

Soil chemistry

Soil organics

The moisture content of the Technosol treatments reflected the organic content, and to a lesser extent the depth, of the treatments ([Figure 1](#page-39-0)). Though there were large variations in moisture present in the replicates for each treatment, particularly in the 80% organic, 60 cm treatment, overall the trend was increasing moisture with increasing organics and depth. A oneway ANOVA did not find the differences in treatment significant ($F(3, 8) = 3.09$, $p = 0.090$); however, when the treatments were grouped by organic content the difference in moisture between the 80% organic Technosol and the 40% organic Technosol was significant $(t(10) =$ 3.05, $p = 0.012$).

Figure 1. Percent moisture content of Technosol treatments.

Because depth has no influence upon the CNS content of the Technosols, results are reported for the two levels of organics only [\(Table 1\)](#page-39-1). These results were consistent with expectations: the 80% organic Technosol had significantly higher %C and %N content than the 40% organic Technosol (t(10) = 12.96, p < 0.0001; t(10) = 8.10, p < 0.0001). However, the C/N ratio between the two Technosols was not significantly different $(t(10) = 0.74, p = 0.476)$. Though the 80% organic Technosol appeared to have slightly higher %S, this difference also was not significant (t(10) = 1.23, p = 0.247).

Table 1. Mean $(\pm SD)$ percentages of C, N, and S with C/N ratios in low organic and high organic Technosols. Letters indicate significant differences within columns.

Technosol	C%	$N\%$	$S\%$	C/N
Low organic	4.37 ^a	0.05 ^a	0.38 ^a	81.7 ^a
(40%)	± 0.56	± 0.01	± 0.13	± 24.0
High organic	15.48 ^b	0.17 ^b	0.47 ^a	92.4 ^a
(80%)	± 2.01	± 0.03	\pm 0.12	± 10.3

Total and bioavailable ions

Differences in total and bioavailable ions were examined between the high and low organic Technosols, but not between treatments with different depths as that should have no impact upon the concentrations within samples, but simply increases the overall quantity present in the plots. Mean concentrations $(\pm SD)$ of total ions including macronutrients (Ca, K, Mg, P), micronutrients (B, Cu, Fe, Mn, Mo, Ni, Zn), and elements of interest (As, Cd, Sb) are presented in [Table 2](#page-41-0) and 3. The mean bioavailable concentrations of the same elements (when above detection limits) are shown in [Table 4.](#page-41-1)

Where possible, t-tests were performed to determine whether there were significant differences in bioavailable concentrations with changing organic content [\(Table 4\)](#page-41-1). The 80% organic Technosol contained significantly higher concentrations of bioavailable macronutrients (Ca, K, Mg, and P) as well as Mn. The high organic Technosol also contained significantly lower concentrations of bioavailable Mo. There was no significant difference in concentrations of As, B, Cu, or Fe. Differences in Cd could not be tested as all 80% organic samples were below detection limits. All bioavailable concentrations of Ni, Sb, and Zn were below detection limits.

The percentage of the total concentration which was bioavailable was below 1% for all elements where comparison was possible apart from arsenic. In the 40% organic Technosol, 2.79% of the total As was potentially bioavailable, while 1.55% was bioavailable in the 80% Technosol.

Technosol	Β mg/kg	Ca mg/kg	Cu mg/kg	Fe mg/kg	V mg/kg	Mg mg/kg	Mn mg/kg	Mo mg/kg	Ni mg/kg	mg/kg	Zn mg/kg
Low org	25	33800	26	21400	16650	12567	408	56	23	487	105
(40%)	± 4	± 2503	± 4	± 2626	± 596	± 1097	± 38	± 14	$±$ 3	± 46	± 5
High org	27	28916.7	25	18233	15667	11500	392	47	18	452	115
(80%)	± 9	± 1797	± 11	± 1751	± 771	± 1022	± 31	± 10	± 2	± 30	± 25

Table 2. Total concentrations of nutrients in low and high organic Technosols (mean \pm SD, n = 6)

Table 3. Total concentrations of elements of environmental interest in low and high organic Technosols (mean \pm SD, n = 6)

Technosol	As	Cd	Sb
	mg/kg	mg/kg	mg/kg
Low org	6.06	0.29	0.42
(40%)	± 0.40	± 0.01	± 0.07
High org	5.37	0.36	0.33
(80%)	± 1.00	± 0.10	± 0.03

Table 4. Bioavailable concentrations of nutrients and elements of interest in low and high organic Technosols (mean \pm SD, n = 6^{*})

* Means ± SD in italics represent mean concentrations of samples above the detection limit (at least one sample < DL excluded from calculations) All Ni, Sb, and Zn concentrations were below detection limits (0.0003 mg/kg, 0.018 mg/kg, and 0.001 mg/kg respectively) Letters indicate significant differences between means with a column ($p < 0.05$)

Other elements where a decreasing proportion was bioavailable with higher organic content were B, Cu, Fe, and Mo. The change in Fe was small (0.00018% to 0.00014%), while Cu availability decreased by half (0.08% from 0.16% bioavailable). Boron availability went from 0.76% to 0.32%. Molybdenum, which is an element of environmental interest as well as a micronutrient, decreased 0.19% from 0.35% availability in the low organic Technosol to 0.16% in the high organic.

Higher percentages of macronutrients (Ca, K, Mg, P) and Mn were bioavailable in the 80% Technosol, with increases ranging from 0.08% in K to 0.27% in Ca and Mn. Phosphorus availability increased 0.25%, from 0.04% in the low organic to 0.29%. Magnesium availability went from 0.14% to 0.24%, an increase of 0.1%.

In the low organic Technosol 0.94% of Cd was bioavailable; as the high organic samples were all below the detection limit no further comparison was possible. Again, Ni, Sb, and Zn could not be analysed due to all bioavailable concentrations being below detection limits.

Water chemistry

Basic chemistry

The pH of both the gravity through-flow and the tension water samples was neutral to alkaline in all years, typically between 7 and 8.5, and no different from the pH of the uncovered Williams mine rock water samples ([Figure 2](#page-43-0)). There was no difference observed between the high and low organic Technosols, or between the 30 cm and 60 cm thick covers.

Figure 2. pH of Technosol gravity through-flow (GT), Technosol tension water (TW), and Williams mine rock test cell (MR) water samples from Oct 2012 to Nov 2016 with loess curve and 95% CI.

There were clear differences in electrical conductivity $(\mu S/cm)$ between gravity throughflow, tension water, Williams mine rock, and rainwater samples ([Figure 3](#page-44-0)). However, there were no treatment differences observed between low and high organic soils, or thick and thin covers. The conductivity of the gravity through-flow samples averaged 700 to 1000 μS/cm, with a high value of 2090 μS/cm from the 60 cm high organic treatment in November 2016. This was noticeably higher than the tension water samples which are typically between $400 - 500 \mu S/cm$, though values as high as 1000 μS/cm were seen in 2012 and 2013. Below the Technosol plot samples are the Williams mine rock water samples, which are around $100 - 300 \mu S/cm$. The rainwater samples have the lowest conductivity, at less than 100 μS/cm. Natural water DOC concentrations measured in 2013 were similar to rainwater concentrations.

Figure 3. Electrical conductivity (μS/cm) of Technosol gravity through-flow (GT), Technosol tension water (TW), and Williams mine rock test cell (MR) water samples from Oct 2012 to Nov 2016 with loess curve and 95% CI.

Concentrations of sulphate showed a similar pattern to electrical conductivity: though there were no differences observed between Technosol treatments, the concentration based on sample type was highest in gravity through-flow, then very similar between tension water and Williams mine rock water ([Figure 4](#page-45-0)). Gravity through-flow varied between $100 - 1400$ mg/L, while the average fluctuated from 400 to 600 mg/L between years. Tension water and Williams mine rock water samples both had an average concentration of approximately 100 mg/L, though tension water samples had a larger range up to around 300 mg/L, occasionally being over 600 mg/L. The rainwater sulphate concentrations were low, under 10 mg/L for all.

Figure 4. Concentrations of SO_4^2 (mg/L) from Technosol gravity through-flow (GT), Technosol tension water (TW), and Williams mine rock test cell (MR) water samples from July 2013 to Nov 2016 with loess curve and 95% CI. 1 sample \langle DL (0.1 mg/L).

The chloride concentrations within the Technosol plots showed no difference between high and low organic thick and thin cover treatments. There was also no difference visible between the Technosol chloride concentrations and the Williams mine rock or rainwater samples from 2014 to 2016: all chloride concentrations were below 10 mg/L (excepting a single sample at 23 mg/L). However, in the summer of 2013, one year after the plots were established, larger fluxes of chloride were seen in several of the low organic Technosol plots (from $30 - 55$ mg/L).

Nitrate concentrations were below 10 mg/L in all samples, and apart from the May 2014 sampling period remained below 5 mg/L. Gravity through-flow samples had higher concentrations than tension water samples, about 70% of which were below the detection limit of 0.01 mg/L (126 of 189 TW samples). Williams mine rock water samples had similar nitrate levels to the gravity through-flow samples, while rainwater samples were typically below the detection limit or very low. There was no difference in nitrate concentrations in water samples taken from vegetated and unvegetated Technosol plots.

All Technosol plots had higher concentrations of DOC than the Williams mine rock water samples, which were extremely low, and the rainwater samples, which were slightly higher. There was no difference between gravity through-flow and tension water samples, but there was a clear difference between high and low organic Technosols ([Figure 5](#page-47-0)). The high organic 60 cm cover had the highest concentrations of DOC (average 40 – 70 mg/L), followed by the high organic 30 cm, the low organic cover 60 cm, and the low organic 30 cm covers (average $20 - 30$ mg/L). Concentrations were highest in 2013, where there was a difference visible in the low organic covers, but decreased and stabilized with time and by 2016 no difference between the low organic Technosol covers was visible.

Figure 5. DOC (mg/L) of Technosol treatment water samples and rainwater samples from July 2013 to Nov 2016 with loess curve and 95% CI.

Macronutrients

Concentrations of calcium and magnesium followed the same pattern, though Ca concentrations were approximately an order of magnitude higher (up to 500 mg/L compared to up to 40 mg/L; [Figure 6](#page-49-0)). Gravity through-flow samples had higher concentrations than tension water samples, particularly in terms of Ca, with an approximate range of $100 - 400$ mg/L compared to a range of 30 – 100 mg/L. Williams mine rock water had concentrations equivalent to the lower half of the tension water range, while natural water and rainwater samples were below 20 mg Ca/L and 2 mg Mg/L. Though there was no clear treatment effect, the low organic Technosol samples tended give the highest concentrations in both gravity through-flow and tension water samples.

Potassium levels were comparable to magnesium levels, but unlike calcium and magnesium there was no clear differences in gravity through-flow and tension water samples. Tension water samples in 2013 were typically higher than gravity through-flow $(30 - 70 \text{ mg/L})$ vs. $10 - 40$ mg/L) but the difference lessened with time and by 2016 concentration ranges were identical (approximately 10 – 50 mg/L). Williams mine rock water samples were below 20 mg/L in all cases, and were higher than the natural water and rainwater samples, which were under 5 mg/L apart from two samples (12 and 7 mg/L).

Phosphorus showed a decreasing trend with time; initially gravity through-flow concentrations were between $50 - 100 \mu g/L$, but samples in 2015 were below 30 $\mu g/L$ and in 2016 below 10 µg/L. Tension water samples followed the same trend, though concentrations in 2013 started below 50 µg/L in general. No difference in treatments was observed. However, approximately half of all samples were below the detection limit of 0.45 µg/L (334 out of 579); these low phosphorus samples were found in all sample types across all years. Concentrations in Williams mine rock water was typically very low, while rainwater samples were equal to or substantially higher than Technosol waters. Only one of the natural water samples had detectable levels of phosphorus $(61.4 \mu g/L)$.

Figure 6. Concentrations (mg/kg) of a) Ca and b) Mg in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 1 sample Ca < DL (20 μ g/L); 1 sample Mg < DL (1 μ g/L).

Micronutrients

Boron concentrations in tension water were highly variable, ranging from $300 - 21700$ μ g/L ([Figure 7](#page-51-0)a). They also increased with time, from approximately 600 – 5000 μ g/L in 2013 to the full range in 2016. There was no difference between treatment for tension water samples; however, in gravity through-flow samples it appeared that the low organic Technosols had higher B concentrations than the high organic Technosols ([Figure 7](#page-51-0)b). These samples ranged in concentration from around $20 - 200 \mu g/L$ and did not exhibit the same trend in increase as the tension water samples; instead levels appeared consistent. Williams mine rock and rainwater samples had low levels of B, and natural waters were below the detection limit of $0.06 \mu g/L$.

Copper was fairly consistent across all sampling periods. Most samples were under 30 µg/L and gravity through-flow samples were generally higher than tension water, while Williams mine rock, rainwater, and natural water were similarly low. The high organic Technosol treatments appeared to have slightly higher gravity through-flow values than the low organic Technosol, but this trend was not always visible.

Iron levels were high in gravity through-flow samples throughout 2013, with the highest values being found in the 60 cm low organic treatment (up to 21900 µg/L; [Figure 8](#page-52-0)a). Concentrations were dramatically in lower 2014, with only four tension water samples higher than 150 μ g/L (2000 – 3000 μ g/L) and 60 samples below the 1 μ g/L detection limit. This trend continued into 2015 and 2016, with concentrations in 95 samples below the detection limit and all others below 125 µg/L ([Figure 8](#page-52-0)b). There was no trend with treatment seen after 2013. In 2013 and 2014 seven Williams mine rock samples had concentrations between 1000 – 3000 μ g/L; in 2015 and 2016 all were below 125 μ g/L. Rainwater samples were consistently low, generally under 30 μ g/L, and natural water samples ranged from 62.7 – 672 μ g/L.

Figure 7. Boron concentrations of a) Technosol gravity through-flow (GT), Technosol tension water (TW), and Williams mine rock test cell (MR) water samples and b) without TW from July 2013 to Nov 2016. Treatments are 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm. 23 samples <DL (0.06 µg/L).

Figure 8. Iron concentrations of a) all samples and b) samples below 125 µg/L; samples are Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) from July 2013 to Nov 2016. 155 samples < DL (1 µg/L).

Manganese followed the same trend of decrease as iron, but the initial concentrations were below 40 μ g/L and a larger portion were below the 0.1 μ g/L detection limit. Gravity through-flow concentrations were typically higher than tension water samples, which were higher than Williams mine rock and natural water samples. Though there were no obvious treatment differences, the low organic 30 cm treatment appeared to have lower concentrations in general than the high organic treatments and the thick cover low organic.

Nickel concentrations showed an increase from 2013 to 2015, peaking in October 2015 with values from $75 - 100 \mu g/L$, mostly in gravity through-flow samples. However, in 2016 all samples had concentrations below 10 μ g/L and there was no difference between tension water and gravity through-flow samples, though rainwater and Williams mine rock water samples were still most often the lowest concentrations.

Zinc levels were typically 50 μ g/L or less in tension water samples, but were as high as 400 µg/L in several gravity through-flow samples. There appears to be a trend of slight decrease from 2013 to 2016. The 60 cm low organic Technosol treatment generally had the highest values, though the 30 cm high organic treatment was occasionally equal or higher. Williams mine rock samples had low values, with the exception of two in May 2014 which were approximately 125 µg/L.

Molybdenum

Molybdenum concentrations in Technosol samples were lower than in Williams mine rock test cells of intermediate volcanic and quartz eye muscovite schist rock ([Figure 9](#page-55-0)a). Gravity through-flow samples had slightly higher concentrations than tension water on average, but the difference was small ([Figure 9](#page-55-0)b). The low organic Technosol treatments also tended to have

slightly higher concentrations, particularly in the tension water, where the high organic Technosol samples were under 40 μ g/L. Low organic Technosol tension water samples ranged from approximately $40 - 600 \mu g/L$, the same range as gravity through-flow samples.

Elements of environmental interest

Cadmium had a similar trend to Mo with regards to the Williams mine rock samples; the concentrations of Cd in the intermediate volcanics and quartz eye muscovite schist test cells was noticeably higher than the Technosol gravity through-flow and tension water (up to around 20 µg/L). There were no differences in tension water and gravity through-flow concentrations, which ranged from below the detection limit of 0.03 μ g/L to 4.8 μ g/L. Concentrations increased from 2014 to 2015 but decreased in 2016 to 2014 levels, with most samples under $2 \mu g/L$.

Arsenic was present in higher concentrations in tension water than gravity through-flow, from approximately $5 - 15 \mu g/L$ ([Figure 10](#page-56-0)a). With the exception of the October 2013 sampling period, all gravity through-flow samples were below 5 µg/L. This was equal to or lower than the Williams mine rock water levels but higher than the natural waters.

Antimony levels from Technosol gravity through-flow and tension water appeared consistent with Williams mine rock test cell water concentrations ([Figure 10](#page-56-0)b). There was no difference between treatments or sample types, and most were below 10 μ g/L. There was also no trend of increase or decrease visible.

Figure 9. Mo concentrations (µg/L) of a) all samples including Williams mine rock test cell water (MR) and b) Technosol gravity through-flow (GT), Technosol tension water (TW), natural water (NW), and rainwater (RW) from July 2013 to Nov 2016. 8 samples \langle DL (0.13 µg/L).

Figure 10. Concentrations of a) As and b) Sb in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 64 samples As < DL (0.35 µg/L); 1 sample Sb < DL (0.01 µg/L).

Discussion

Effects of increasing soil organics

The soil moisture content was significantly higher in the 80% organic Technosols than the 40% organic Technosols, which was expected as organic matter often is capable of holding more water than the mineral components of a soil (Larney & Angers, 2012). However, it should be noted that this is a single measurement providing a snapshot of soil conditions shortly after plot construction in 2012, prior to the establishment of vegetation on the plots. Although increasing organic content has been seen increasing water retention, particularly in coarse textured soils such as these (Rawls, Pachepsky, Ritchie, Sobecki, & Bloodworth, 2003), it would be difficult to make such an inference in this situation without the long-term moisture monitoring data in Chapter 3, which indicates that the trend of the high organic Technosol containing more moisture generally continues in the following years.

Differences in organic content also significantly influenced the total C and N contents, both of which tripled from the 40% to 80% organic Technosols. Again, this is to be expected as higher levels of organic matter necessarily mean higher levels of carbon and nitrogen. On the other hand, sulphur was not significantly different between the two Technosols. The S content of the metasedimentary rocks and woody residuals used to construct the Technosols was measured as 0.003 and 0.03%, respectively (Watkinson et al., 2016). This means the majority of the sulphur in the Technosol came from the intermediate volcanics, which had a S content of 0.41. The lack of difference between the two Technosols despite the large difference in mine rock content implies that the increasing contribution of sulphur from the increasing organic content was enough when combined with the presence of intermediate volcanics in the rock to maintain the sulphur content around 0.4%.

The C/N ratios of the Technosols were very high, above 80, and did not significantly change between the two organic levels. A high C/N ratio is often thought to limit decomposition rates by limiting bacterial growth (Enríquez, Duarte, & Sand-Jensen, 1993), though there is also evidence which suggests that it is respiration rates and ATP content which control differences in rates of nitrogen immobilization and mineralization (Bengtsson, Bengtson, & Månsson, 2003). Regardless, the quantity of nitrogen in both Technosols was low, particularly in the 40% organic, which at 0.017% had levels comparable to those of subsoils (Barker & Pilbeam, 2007). These levels could limit the growth of plants upon the Technosols.

There was also a clear link between the amount of woody residuals on a plot and the amount of DOC in the water samples from that plot. Again, this was likely due to the increasing amount of carbon available; the thick, high organic plots simply had more overall carbon than the thin high organic plots or either of the low organic plots. The initial flush of DOC in 2013 was likely due to the system equilibrating, as the following three years showed consistent levels of DOC leaving the Technosols. DOC in the high organic Technosol waters was high compared to levels in natural lakes; in a review of global DOC content, 0.4% of lakes had concentrations above 40 mg/L, and 4.2% had $20 - 40$ mg/L, which is what was found in the low organic Technosol waters (Sobek, Tranvik, Prairie, Kortelainen, & Cole, 2007). DOC can have a large effect on the bioavailability and toxicity of metal compounds; higher concentrations of DOC are linked to lower bioavailability (Merrington, Peters, & Schlekat, 2016). The high levels we saw coming out of the Technosol plots may have limited the bioavailability of metals for plant and microbial uptake. However, as these were measurements taken from through-flow samples, it can also be assumed that large areas covered by the Technosols would result in large amounts of DOC entering surface waters. Many boreal lakes have much lower concentrations of DOC, under

10 mg/L, so large increases could affect not only metals and nutrients, but also transparency, stratification, pH and alkalinity, microbial production, and UV penetration (Hudson, Dillon, & Somers, 2003).

Chemical parameters of water samples

The pH of the water samples was surprisingly alkaline; most samples were above pH 7. This was not expected because, although the pH of both mine rock types was around 8.8, the pH of the woody residuals was 6.5, consistent with them being mostly composed of boreal coniferous trees (Watkinson et al., 2016). Particularly in the high organic Technosol, which only contained 20% by volume of mine rock, it was thought a larger influence on the pH by the organics would be seen. It is possible that the lysimeter design and sampling routine contributed to the high pH values seen; because sampling occurred a maximum of once a month, water often sat for weeks prior to removal. In the case of gravity through-flow samples, this means the water either remained in the barrel or at the base of the geomembrane, among the large mine rocks used for construction. Tension water would have been held within the Technosol, where it may have continued interacting with the mine rock in the Technosol matrix.

Both the intermediate volcanic and metasedimentary rocks from the mine are known to have high neutralization potential due to the presence of carbonate minerals; it is possible their buffering capacity was high enough to neutralize any acids from the organics and still raise the pH above 7, even with low volumes of rock within the soil. For example, in the intermediate volcanics the sulphur content was 0.41% while the carbon content was 1.16% (Watkinson et al., 2016). This means the carbonate content in these rocks was likely high enough to compensate for any acidity due to sulphur compounds.

The high pH of water samples, while potentially problematic for boreal vegetation, is good in the sense that it means acid generation in the mine rock did not increase when the Technosol cover was used. Increased acid generation would have raised concerns about the consequences of covering a large area of the mine rock pile with the Technosols, as large amounts of acidic runoff would be damaging to the surface and groundwater in the area.

The electrical conductivity (EC), a proxy for the number of dissolved ions in solution, was higher in the Technosol water samples than the Williams mine rock water samples. Substances released from the organic material may have contributed to greater weathering in the Technosol than the mine rock plots; increases in EC have been seen in other cases after organics have been added, including chipped wood (Hattab et al., 2015). Some of the difference could have been due to the fact the mine rock in the Technosol was crushed and therefore much finer than the mine rock used in the Williams mine rock test cells, making it more reactive. The higher EC in the gravity through-flow samples compared to the tension water samples indicated that a larger amount of these ions were washed out, rather than remaining in the soil pore water. Again, this could be partially due to lysimeter design; the tension water samples were taken directly from the Technosol but the gravity through-flow samples travelled through an additional layer of crushed and coarse mine rock before meeting the geomembrane and going into the collection barrels. This may have given the water more time to react with the mine rock and pick up additional ions on its way.

As for the identity of these ions, a look at the anions present in the samples revealed sulphate had a similar pattern to EC and was present in high amounts, meaning it was likely to be a large contributor. Nitrate and chloride, the other anions measured, had much lower concentrations than sulphate and therefore contribute less. In terms of cations, calcium and

magnesium had the same pattern, and along with potassium had high concentrations, particularly calcium. This means they are probably also important in determining EC.

Nutrients and trace elements

Macronutrients

The total macronutrient content was higher in the low organic Technosol, but the bioavailable macronutrient content was significantly higher in the high organic Technosol. We know more organic compounds are released by the high organic Technosol, as the DOC concentrations are higher. This could mean that there was increased weathering and freeing of nutrients in the 80% organic Technosol due to the increased amount of organic compounds released. It could also have been because of the increased proportion of macronutrients held in the woody residuals. Though total Ca and P content was similar between the parent materials and total Mg and K were lower in the woody residuals than in the mine rocks (Watkinson et al., 2016), these elements may have been more easily available through decomposition from the wood than through rock weathering.

Calcium, Mg, and K were all present in the water samples at high levels, particularly Ca which was present in higher concentrations in the Technosols. Phosphorus levels were much lower, probably due to lower concentrations within the soil itself. The large number of samples below detection limit and the apparent decrease in P in water over time means there would be few eutrophication concerns associated with the runoff of P into surface water from these Technosols if they were used as an extensive soil cover; however, the low concentrations especially in tension water may make it difficult for plants to obtain the P they require. From these results it seems that of the measured macronutrients, only P may have acted as a limiting nutrient to plant growth.

Micronutrients

Mn in the Technosols showed the same pattern as macronutrients: a significantly higher amount was bioavailable in the 80% organic Technosol despite it containing slightly less Mn in total. The increased availability could be due to the higher level of Mn in the woody residuals than the mine rock; like with the macronutrients, more Mn from the organics may be in a bioavailable form than the Mn released from the rock.

Of the other micronutrients, Ni and Zn bioavailable levels were below detection limits and so no inferences can be made other than the fact that their bioavailable concentrations are quite low (below 0.0003 and 0.001 mg/L). For B and Cu no significant difference was observed in total or bioavailable concentrations. Bioavailable Fe was lower in the high organic Technosol but the difference was not significant. The bioavailability of these elements is controlled more by their content in the soil than by their interactions with organic material, as least at this stage in the Technosol development. Therefore, the lower total amounts of B, Cu, and Fe in the high organic Technosol meant lower bioavailable amounts as well.

In water, Fe and Mn concentrations were highest in the first year after plot installation and decreased to summer 2015, where they appear to have levelled off in the past two years. The initial high levels were mostly found in the gravity through-flow samples; although tension water Fe concentrations decreased with time, the Mn concentrations remained fairly constant. This initial flush was probably due to the Technosols equilibrating, and the less tightly-bound compounds being released. The Fe concentrations were so elevated during the first year, much higher than the Provincial Water Quality Objectives (PWQO) of 300 μ g/L and exceeding the monthly average concentration limit of 1.0 mg/L under the Metal Mining Effluent Regulations

(MMER) and Municipal Industrial Strategy for Abatement (MISA), that large quantities could be initially released if the Technosol was used for a cover. However, it appears that after the first year the amount would no longer be a concern, at least in terms of regulatory requirements, as all through-flow samples were below 120 μ g/L.

Boron concentrations in tension water samples were very high, much higher than the gravity through-flow samples. Boron in the Technosol is present in fairly low amounts; what is there is likely from the presence of tourmaline, a boron-rich mineral which has a wide distribution over the area (Muir, 2002). Though there is no difference in the tension water samples by treatment, in the gravity through-flow it appears the low organics contain more, possibly due to a longer interaction with the Technosol. Organics may slowly react with boron to form water-soluble compounds, but that these compounds are then held in the soil pore water rather than leaching out. The increase in B in tension water through time adds support to the idea of slow weathering and water-soluble compounds trapped in by soil particles in the pore water.

Cu and Zn in water were similar; both were generally higher in gravity through-flow with the exception of some samples in summer 2014, and both appeared to be stable through time. Cu concentrations were generally above the PWQO of 5 μ g/L and Zn was above the 20 μ g/L PWQO, so it is possible the amount of these elements in runoff could be a concern in time. However, both were below the required compliance levels of MMER and MISA.

The dramatic increase in Ni in gravity through-flow in 2015, followed by the dramatic decrease in 2016, was unexpected. It could be there was a large amount of soluble Ni that took several years to flush through the Technosols; it could be that all easily weathered Ni in the Technosol was removed in 2015. 2015 was not a wet year; it experienced periods of dryness particularly around mid-July. It is possible that prolonged dryness followed by heavy rain

resulted in large system flushes, removing all the less tightly held Ni from the system. Regardless, it would be interesting to see if Ni remained low in following years, or if increases were again seen.

Molybdenum

Molybdenum is one of the main elements of concern on the Hemlo site because there are significant amounts of molybdenite present throughout the deposit (Pan & Fleet, 1995), therefore, knowledge about its behaviour in the Technosols is important. There was a significant decrease in the bioavailable Mo from the low to high organic Technosol. While there was also less total Mo in the high organic Technosol, a look at the percent of the total which is bioavailable showed that there was 0.35% bioavailability in the 40% organic, twice as much as in the 80% organic which was 0.16% bioavailable. Interactions between Mo and organic compounds within the soil could have limited its bioavailability, and these interactions would have increased as the organic content increased.

Molybdenum concentrations in Technosol gravity through-flow and tension water samples were lower than in the intermediate volcanic and quartz eye muscovite schist mine rock weathering test cell plots. Intermediate volcanics were one of the parent materials of the Technosols, so the difference between the two implies something occurred within the Technosol plots to lower Mo release. Fewer intermediate volcanic rocks may have been present, simply because the Technosol plots also contain metasedimentary rocks and woody residuals, unlike the mine rock test cells. Interactions between organic compounds and Mo in the Technosols could also have reduced the mobility of Mo. Generally the gravity through-flow samples had higher concentrations than tension water samples, likely because they were exposed to more

intermediate volcanic rocks as they travelled through the mine rock layers of the lysimeter. The fact they still had much lower levels of Mo than samples from the pure test cells supports the idea the organics in the Technosols reduced the mobility of Mo. The increase in Mo in tension water in 2016 could mean that as time passes more Mo was being held in the pore water.

Samples exceeded the PWQO of $40 \mu g/L$, some by quite a lot. However, they were below the monthly average of 1.0 mg/L required by MMER/MISA. It is possible that the Mo in the water was not bioavailable. This is supported by the fact that wood chips have been seen to decrease the mobility of Mo in other studies (Hattab et al., 2015), and by the Mo content in the plant leaves grown on the Technosol (Chapter 4). Mo is also a micronutrient, particularly important for nitrogen-fixing plants (Barker & Pilbeam, 2007), and therefore the presence of some available Mo is beneficial.

Elements of environmental interest

As, Cd, and Sb are elements of environmental interest on the mine site, along with Mo, because they are present in the deposit in minerals such as arsenopyrite, realgar, and stibnite (Muir, 2002). Total concentrations of Cd and Sb in both Technosols were under 0.5 mg/L; there was no bioavailable Sb detected in either, and bioavailable Cd was found in only two of the six 40% organic Technosol plots. Cadmium in Technosol water samples was noticeably lower than from the intermediate volcanic Williams mine test cells. All but two Technosol water samples had less than 3 µg/L Cd, above the Ontario PWQO of 0.2 µg/L. Most Sb concentrations were under 10 μ g/L, below the 20 μ g/L PWQO. Both elements had concentrations below the maximum monthly average of 1.0 mg/L specified by MMER and MISA.

Arsenic totals in the Technosols were also relatively low. There was no significant difference in the bioavailable concentrations between the two Technosols, but the percentage which was bioavailable was almost twice as high in the 40% organic Technosol. This could mean that the higher organics were binding a larger percentage of the As into forms available for plant uptake. Though no significant difference was seen here, if large amounts of the Technosol were used the 1% decrease in bioavailability could become important, particularly as As concentrations were higher in the tension water samples than the gravity through-flow. All As concentrations were well below the MMER/MISA requirement of 1.0 mg/L per day and the current PWQO of 100 μ g/L, but most of the tension water samples were above the interim PWQO of 5 µg/L. This means that there may fewer risks associated with As leaching from the Technosols into the environment, but more opportunity for plants to take it up. One form of As, arsenate, is an analog of phosphate and can compete for the same uptake mechanisms (Nagajyoti et al., 2010). If the arsenate ion was the dominant form in the tension water, it could have negative impacts on plant growth. However, though we did not examine the speciation of elements in this study, in Chapter 4 of this thesis the concentration of elements in plant leaves grown on the Technosol was measured. Arsenic in leaves was below 1 mg/kg in all detectable leaf samples, and comparable to samples taken from reference plants. This suggests that the As in the water samples was not in a highly bioavailable form.

Conclusion

The 80% organic Technosol had significantly higher total C and N, and bioavailable Ca, Mg, K, P, and Mn concentrations than the 40% organic Technosol. Higher organic content also significantly decreased the amount of bioavailable Mo and resulted in undetectable levels of Cd. High DOC levels were measured in Technosol water samples, particularly those from 80%

organic plots. Water samples from both Technosols had high pH $(7 – 8)$, probably due to the high pH of the mine rock used in Technosol construction. Concentrations of Mo and Cd in gravity through-flow samples exceeded PQWO limits, though they were lower than the plots constructed purely of mine rock. Arsenic in tension water samples exceeded interim PWQO limits, but As in through-flow samples rarely did. This means monitoring of the through-flow from the Technosols, particularly for Mo and Cd, would be required.

Chapter 3. Early profile development of a four-year-old mine rock Technosol Introduction

Humans have become the major driving force of environmental change in the world, affecting all aspects of the biosphere including soils (Leguédois et al., 2016). Activities like mineral and energy extraction, urbanization, and agriculture have resulted in heavily altered soils (Leguédois et al., 2016). In response to the growing distribution of human-altered soils, the 2006 World Reference Base for Soil Resources added Technosols as a Reference Soil Group (IUSS Working Group WRB, 2006). In essence, they are soils which contain more than 20% by volume of artefacts (substances created, modified, or moved by human activity), a constructed geomembrane, or technic hard material such as asphalt (IUSS Working Group WRB, 2014).

There has been a noticeable increase in the number of studies on Technosols in the last few decades, with particular emphasis on their physical-chemical properties and evolution (Capra et al., 2015). In natural soils, the soil-forming factors climate, parent materials, organisms, relief, and time are accepted as controlling evolution (Bockheim et al., 2005), and these factors control the development of Technosols as well, with the addition of whatever human activity resulted in their original formation (Leguédois et al., 2016). However, due to the often unusual physical and chemical properties of Technosols, a consequence of their human origins, their development can exhibit large differences from those of natural soils as well as occur on much shorter timescales (Huot, Simonnot, et al., 2015). For this reason monitoring the early profile development of Technosols can provide valuable information, particularly when they are constructed for a specific purpose.

Development can be seen in the physical, chemical, and biological properties of soil, and all aspects should be studied for a more complete understanding of Technosol pedogenesis (Leguédois et al., 2016). Soil pH and electrical conductivity are two important variables which

have a large impact on many physical, chemical, and biological processes, such as metal speciation, vegetation growth and survival, and microbial community structure (Brady & Weil, 2010). They are also important in classification of natural soils (IUSS Working Group WRB, 2014). The leaching of soluble compounds and oxidation of minerals such as sulphides from the upper layers of the soil are commonly among the first changes to occur in the soil (Huot, Simonnot, et al., 2015), so the measurement of pH and conductivity can also provide some indication of weathering within the profile.

Soil moisture and temperature are also important in many soil processes. They influence decomposition rates, nutrient availability, plant growth and distribution, erosion, and runoff generation (Özkan & Gökbulak, 2017). The capacity of a soil to hold moisture will change with its aggregation and porosity, both physical properties of soil that change with time (Ciarkowska et al., 2016). Though the structure of Technosols is typically weak, increased aggregation has been seen in Technosols when vegetation is present (Huot, Simonnot, et al., 2015). Thus, changes in the Technosol temperature and moisture regimes might reasonably be expected as time passes, particularly under vegetation coverage.

The soil microbial community has increasingly been recognized as a critical part of biogeochemical cycling within the soil, responsible for the decomposition of complex organic molecules and therefore important in nutrient cycling (Stefanowicz, 2006). Microbial communities of Technosols can be quite different from those in natural soils and they are often left out of studies on Technosol properties (Dimitriu et al., 2010). However, even initially sterile parent materials can be quickly colonized and become biologically active soils (Leguédois et al., 2016), so the inclusion of some sort of measure of microbial diversity is a good policy. Microbial communities vary between seasons and years (MacKenzie & Quideau, 2010) and with

temperature, humidity, composition of organic matter, vegetation type, and soil properties (Stefanowicz, 2006), meaning that a single measurement is likely not representative of the entire soil profile.

Using these parameters, a description of four Technosol profiles was undertaken, examining changes within the profiles four years after they were manufactured from mine rock and woody residuals and placed on site in Canada's boreal shield.

Methods

Study site

The Williams Mine is in Hemlo, ON and operated by the Barrick Gold Corporation. This area is within the Canadian Shield, specifically in the eastern part of the Schreiber-Hemlo greenstone belt of the Wawa subprovince, part of Archean Superior province (Muir, 2002). The deposit is characterized by a lack of major quartz and carbonate veins, no mafic volcanic rocks in the mine sequence, and a diverse set of metals (Muir, 2002). The most common soils are Podzols, which have a reddish illuvial spodic (Bhf/Bf) horizon rich in Fe/Al overlain by a bleached eluvial (Ae) horizon from which these elements were leached (IUSS Working Group WRB, 2014). Mean monthly temperatures of the region range from 15°C in July and August to -14°C in January; precipitation varies from 122 mm in September to 47 mm in February (Environment Canada, 2017). The area is part of the boreal forest ecosystem and as such is dominated by black spruce (*Picea mariana* (Mill.) B.S.P), jack pine (*Pinus banksia* Lamb.), balsam fir (*Abies balsamea* (L.) Mill.), trembling aspen (*Populus tremuloides* Michx.), and white birch (*Betula papyrifera* March.) (Sims et al., 1996; Zoladeski & Maycock, 1990).

Plot set-up

In summer 2012 crushed, non-acid generating metasedimentary and intermediate volcanic rocks removed during mining operations were combined with boreal coniferous woody residuals from the White River Forest Products Sawmill, located 60 km east of the mine, to create two Technosols. The first of these consists of 40% woody residuals and 60% mine rock by volume; this is referred to as the low organic Technosol. The second, high organic Technosol was formed with a ratio of 80% woody residuals to 20% mine rock. The Technosols were placed in 30 cm or 60 cm layers upon a lysimeter constructed with a layer of crushed mine rock over a base of coarse mine rock on an impermeable geomembrane. Each treatment was then replicated three times to give a total of twelve plots (Watkinson, 2014). Green alders (*Alnus viridis* subsp. *crispa*) were planted on eight of the twelve plots in 2013 and again in 2015 to bring the number on each up to at least nine. At this time twelve bearberries (*Arctostaphylos uva-ursi*) were also planted on each of the vegetated plots. In April 2016 a mixture of annual and perennial ryegrass seed (Home Gardener Overseed Premium Grass Seed Mix with Kickstart™ Technology) was spread over the vegetated plots in an attempt to establish a nurse crop to promote and protect the bearberries.

Within each plot sensors were installed in 2012 three weeks after plot construction at multiple depths to monitor soil microclimate conditions. 5TM soil temperature/moisture sensors (Decagon Devices) were initially installed at 10 cm, 30 cm, and, in the thick treatments, 60 cm depths. In July 2014 5TM sensors were added at 5 cm depths in nine of the plots; the remaining three had 5 cm sensors added in July 2015. One MPS-2 water dielectric potential/temperature sensor (Decagon Devices) is also present in each plot at either 30 cm or 60 cm, depending on the treatment thickness. Sensors were connected to EM50 series data loggers (Decagon Devices) and measurements were taken at varying intervals, ranging from every 5 minutes for portions of the
summer to every 60 minutes throughout the winter. Data was retrieved from the loggers using the ECH2O software from Decagon Devices.

In summer 2014 two comparison soil sites were selected for sensor installation to provide a comparison to the plot microclimate measurements. The first of these was in the field immediately behind the plots; this field was once covered with infrastructure including buildings and parking as part of the Newmont Golden Giant Mine, but has since been reclaimed using a combination of stockpiled soil, fertilizer, and MTO grass and legume seed mix. This site is referred to as the successional field, and has 5TM sensors installed at 5 cm, 10 cm, 15 cm, 30 cm, and 60 cm depths. The second site was just over 1 km from the plots, in an upland secondary forest stand. 5TM sensors were installed here at 5 cm, 10 cm, 15 cm, 20 cm, and 25 cm, just above where the soil meets bedrock. A climate station was also installed in summer 2014, located between the centre two plots to measure air temperature and relative humidity at 2 m (VP-3 Humidity/Temperature sensor, Decagon Devices) and precipitation (ECRN-100 Precipitation sensor, Decagon Devices).

Data from all loggers was collected multiple times per summer and compiled using R 3.3.2 (R Core Team, 2016). Daily averages were calculated for the temperature and moisture measurements in all plots and comparison locations; for precipitation the daily sum was calculated. Despite batteries dying and several sensors and one logger failing on separate occasions and requiring replacement, the plot replication ensured that a continuous soil temperature series was obtained for each treatment. The soil moisture series were also continuous, with the caveat that any data obtained when the temperature was below 0°C could not be used, as the sensors cannot read soil moisture when water is in a solid state. Therefore, all data from December 1 to May 1 each year was removed, as well as any moisture readings

obtained when the soil temperature at the same depth was below 1°C to ensure that all measurements could be trusted. Water potential measurements were handled in the same way. For comparison purposes, the forest soil temperature and moisture at 25 cm was used as a proxy for 30 cm depth measurements.

Soil profile sampling

On September 27 and 28, 2016, profile sampling was done on four of the treatment plots as well as in the successional field and forest to examine profile development and collect samples for analysis. The treatments selected were the 40% organic, 60 cm thick and 80% organic, 60 cm thick plots; the thick plots were chosen over the thin as the development in the upper layers was expected to be similar to the thin plots, while the development at depth could be more fully examined in the thick plots. Two of each were chosen, one with vegetation and one without. Pits were dug on the back right corner of plots to avoid interfering with sensors, which are located near the plot centre. On vegetated plots, pit locations were governed by vegetation: while we wanted to be near enough to roots, particularly alder roots, to be able to determine what influence the vegetation was having on soil development, we wanted to avoid extensive damage to root systems. In keeping with the wish to minimize disturbance to plots, soil pits were kept as small as possible (approximately 45 cm wide; [Figure 11](#page-74-0)). Profiles were dug to the base of the Technosol layer, where the Technosol meets crushed mine rock. Once digging was complete, a brief description of the profile was done prior to sample collection.

Figure 11. Soil pit on vegetated, 40% organic plot.

Samples were taken continuously from the surface down in the following increments: 2 cm from $0 - 20$, 5 cm from $20 - 30$, and 10 cm from $30 - 60$ or the base of the profile. These were chosen because more rapid changes were expected in the top 20 cm than at the base of the profile. This resulted in 15 samples per profile. Samples were removed from top down using a tile setter trowel and a finishing trowel. Because part of the intended analysis was an assessment of microbial functional diversity, gloves were worn, tools were wiped down with 90% ethanol between each sample, and samples were immediately placed in a cooler after removal.

Samples were taken in the field and forest in the same manner but the profiles were shorter; we were only able to reach 42 cm in the field, and hit bedrock after 30 cm in the forest.

For an estimate of microbial community, 1 g of soil was removed from each sample prior to any other analysis. Of the remaining portion, 5 g of soil in 20 mL of deionized water was used to determine the $pH (pH_w)$ and electrical conductivity of the sample; the sample equilibrated for

at least 30 min before being measured by a pH combination electrode and a conductivity probe (Accumet) attached to an Accumet (AB15) meter and Accumet (AB30) meter, respectively. The pH of the soil was also determined using a solution of 0.01 M CaCl₂ in the same manner (pH_{Ca}). Additional chemical analyses can be found in Appendix D.

Measuring microbial functional diversity

Changes in the microbial functional diversity with depth and location were examined with the Biolog EcoPlate™, which is a plate of 96 microwells containing three each of 31 different sole-carbon sources selected for environmental analysis, plus three control water wells (Biolog Inc.). Sources fall into one of the following groups of chemical compounds: amines, amino acids, carbohydrates, carboxylic acids, polymers, or phenolic compounds.

The EcoPlate[™] is a redox system which contains tetrazolium dye along with the solecarbon source substrate (Biolog Inc.). The colourless tetrazolium dye is reduced to violet formazan by microorganisms as they oxidize the substrate (Stefanowicz, 2006). Specifically, electrons from the NADPH which is formed as the cells metabolize the substrate are passed to the tetrazolium dye, reducing it to formazan (Mauchline & Keevil, 1991). Optical density at 590 nm is then measured to determine substrate usage. However, it should be noted that fungi do not metabolize the tetrazolium dye used in the EcoPlate™ well, and therefore fungal activity is not measured by this technique (Preston-Mafham, Boddy, & Randerson, 2002).

As mentioned previously, 1 g of soil was removed from each of the soil profile samples (40% organic, vegetated and not; 80% organic, vegetated and not; successional field; forest) for this analysis. The soil was suspended in 99 mL of sterile deionized water and placed on a Benchmark Incu-Shaker Mini at 100 rpm for 20 min. The samples were then placed in a refrigerator for 30 min to allow soil particles to settle. EcoPlates[™] were inoculated with 150 µL

of the soil solution and the optical density at 590 nm was immediately read using a Synergy H1 Microplate Reader (BioTek). Plates were wrapped in aluminum foil and incubated at room temperature (approx. 25°C). Readings were done 0, 3, 6, 12, 24, 48, 72, and 96 hours after inoculation. The multiple readings ensured that colour development could be examined in all wells, as lag times can vary (Preston-Mafham et al., 2002).

Once all readings were completed, the average well colour development (AWCD) for each sample was calculated for each time point, with the control water well values first subtracted from the substrate well values. The time point with the largest mean AWCD (0.1 or greater) and the smallest range between samples was selected at the comparison time, to ensure that colour development was occurring in most or all substrate wells and AWCD was approximately the same for each sample. Accordingly, the point 48h was selected, with an AWCD of 0.29 and a range of 0.7. This large range is due to the large differences between the four Technosol sites, which ranged from around 0.15 to 0.35, and the forest site, which had very low AWCD in most samples taken from below 10 cm in depth. AWCD has been found to be correlated with inoculum density, suggesting colour production is linked to the growth of bacteria within the wells rather than simply the respiration of the existing community (Garland $\&$ Mills, 1991). Although in some cases it has been suggested that readings with similar AWCD be chosen for all samples regardless of the actual incubation time it takes to reach this development in order to limit the effect of inoculum density (Stefanowicz, 2006), in this case our aim was to compare the actual microbial functional differences between sites and depths. As a result we did not wish to perform any sort of manipulation to reduce the effect of inoculum density, as that in fact could be the main difference between sites (Preston-Mafham et al., 2002).

The same time point, 48h, was chosen for a comparison of the richness and evenness of the response of each sample (Frąc, Oszust, & Lipiec, 2012), with an OD of 0.1 selected as the threshold for positive response to remove weak false positives (Garland, 1997). Species richness (*R*), is the number of oxidized substrates, while the Shannon-Weaver index (*H*), is calculated as:

$$
H = -\sum_{i=1}^{N} p_i \ln p_i
$$

Where p_i is the proportion of microbial activity on substrate *i* and *N* is the number of substrates on the plate (Stefanowicz, 2006). Evenness (*E*) could then be calculated as:

$$
E = \frac{H}{\log S}
$$

Where *S* is the number of species. Two-way ANOVAs with Tukey HSD were used to determine whether significant differences exist between sites and depths in terms of AWCD, *R*, *H*, and *E*.

Principal component analyses (PCAs) were performed to further evaluate the relative similarity of the responses at 48 h among the samples (Garland, 1997). PCAs were done on all samples, on Technosol and field samples, and on only Technosol samples in order to try and improve the separation of samples. The same three PCAs were then performed again using only samples which had an $AWCD > 0.1$. As no differences were observed between the six PCAs, the original including all samples was selected for further analysis.

From the PCA including all samples, six substrates were selected based on their correlation with multiple other substrates and with PC1 or PC2 for graphical representation of the colour development through time. The substrates chosen were the amine putrescine, the carbohydrates D-mannitol and β-methyl-D-glucoside, the carboxylic acids α-ketobutyric acid and D-glucosaminic acid, and the phenolic compound 4-hydroxybenzoic acid. All statistical analyses were performed using R 3.3.2 (R Core Team, 2016).

Results

Profile descriptions

Profiles were initially a homogenous mixture of $2 \text{ mm} - 1 \text{ cm}$ diameter mine rock with woody residuals which are on average $0.2 \text{ mm} - 3 \text{ cm}$ in length. However, though an attempt was made to remove the largest pieces of wood (i.e. firewood sized logs), some large pieces still made it into the Technosols and are particularly likely to be found in the 80% organic.

After one year, a pavement-like crust was observed on the surface of the Technosols. The crust was thin, 0.2 – 0.3 cm in thickness, and hard enough to prevent footprints from being left on the plot surface. There was a thin lag deposit on the surface of the crust, mainly composed of woody residuals with the fines removed. The crust disappeared by July 2015.

The 40% organic plot with no vegetation had a 59 cm thick profile and several large wood pieces were removed during excavation. The surface layer was slightly lighter in colour and coarser in texture than the lower layers, but overall there was little variation with depth observed. The soil structure was massive and fine granular. The soil was quite wet due to rain starting during the sampling, which also prevented us obtaining an image.

The 40% organic vegetated plot was similar to the unvegetated plot in that there was little variation observed with depth in terms of structure and colour ([Figure 12](#page-79-0)). However, overall the soil had more structure than the unvegetated plot and could be classified as granular. There were numerous fine ryegrass roots present at the surface of the pit which held the top layers of soil together much more than was observed in the unvegetated profile, despite the grass being by this time almost completely dead. Alder roots were also visible throughout the profile; most were fine, but medium roots were also visible. The roots in the top 20 cm were primarily vertical, while lower down some horizontal roots were seen. There were few roots present under 50 cm depth.

Figure 12. 40% organic, 60 cm deep, vegetated Technosol profile. Fine roots visible in upper layers of profile; little colour or structure variation seen with depth.

The 80% organic unvegetated plot had a 58 cm profile and little variation with depth, though the lower layers were slightly darker than the top ([Figure 13](#page-80-0)). The top $0 - 2$ cm contained the least fines and was mainly composed of larger pieces of rock and wood. The structure was similar to the 40% organic unvegetated plot, being massive and fine granular. There were many larger pieces of wood removed during pit excavation.

Figure 13. 80% organic, 60 cm deep, non-vegetated Technosol profile. Little variation in colour or structure seen with depth.

The vegetated 80% organic plot had more roots present than the 40% organic vegetated plot, again with a large number being fine vertical roots in the top 20 cm, many from the ryegrass ([Figure 14](#page-81-0)). Medium roots were observed lower down in the profile, though few were present under 50 cm. The variation with depth was like that of the other profiles: the top was slightly lighter in colour than the bottom and contained fewer fines. The granular structure was more pronounced in this profile than any of the other Technosol profiles. Large wood pieces were removed from this profile, including one which approached 60 cm in length.

Figure 14. 80% organic, 60 cm deep, vegetated Technosol profile. Many fine roots visible; little colour or structure variation with depth.

The field pit was dug under tamarack, willow, and birch; the ground was covered by grass and moss. The profile was only able to be dug to 42 cm due to numerous large rocks at the base ([Figure 15](#page-83-0)); many rocks were also removed during the digging of the pit. In general the soil was quite compact and rocky, with an overall sandy texture. As this soil is composed entirely of materials moved to the site by human activity during the reclamation process, it too is considered a Technosol.

There was a thin LFH layer at the top of the profile mainly composed of decomposing moss and grasses. There was a sharp boundary between the LFH horizon and the dark brown Ahj horizon below, which varied from about $2 - 4$ cm in thickness. The Ahj horizon was granular and dominated by fine sand sized particles. There were many fine vertical and subvertical roots present, along with some horizontal roots near the top of the horizon. Coarse vertical roots were also observed.

The Ahj was separated from the underlying Bm horizon by another sharp boundary. The Bm horizon was a lighter brown-yellow colour than the above, and consisted of fine- to mediumsized sand particles with some silt. Pebbles were common throughout. Many fine roots were present, running vertical to subvertical. The Bm horizon averaged about 8 cm in thickness and had a subhorizontal boundary with the B horizon, which was 19 cm in thickness and was a darker colour than the Bm but had the same brown-yellow tone. Mottles were seen in this horizon, and pebbles were again common. The B horizon was coarser than the Bm, being mostly sand with a small amount of silt. More horizontal coarse roots were present in this horizon, though few fine roots were still observed.

The B horizon shared a distinct boundary with the C horizon, which was a light brownish-grey colour and had few roots within it. It had a finer silty sand texture than the B horizon, but not as fine as the Bm horizon. It had a subangular blocky structure and 8 cm of it was visible in the pit dug.

Figure 15. Successional field soil profile, with Ah and Bm horizons visible.

The forest soil is a Podzol, and the profile was 30 cm deep over bedrock ([Figure 16](#page-84-0)). There was a 4 cm thick LF horizon overlaying a 2 cm thick H horizon which was dark in colour and high in organics. There were many fine to coarse roots in this horizon, mostly horizontal to subhorizontal, and fungal hyphae were also visible. The H horizon was granular and had a fine sand texture.

A sharp wavy boundary separated the H and Ae horizons. The Ae horizon was around 11 cm thick on average, and was bleached a light- to medium-grey colour. It was composed of a mixture of sand- and silt-sized particles. Many fine roots were observed, as were some coarse and medium roots, again in a mostly horizontal to subhorizontal pattern.

The Bf horizon lay directly on the bedrock and was separated from the Ae horizon by a sharp subhorizontal boundary. It was a reddish-brown colour and had a medium-to-fine subangular blocky structure. The particles varied from coarse sand to silt. Medium subvertical roots were seen in this horizon.

Figure 16. Shallow podzolic profile formed in till over bedrock. Ae and Bf horizons visible.

Profile chemistry

When mixed with deionized water, the pH of the intermediate volcanic and metasedimentary rocks used to create the Technosols was 8.83 and 8.88, respectively, while the pH of the woody residuals was 6.53. Four years later, the high pH of the mine rock was reflected in the pH of the Technosol profiles, which was above 7 at all depths ([Figure 17](#page-85-0)). The pH of the

80% organic Technosol was approximately an order of magnitude less than the 40% organic (8.0 vs. 9.4). Vegetated plot pH was higher at the surface in both the 40% and 80% Technosols, but became lower at $4 - 6$ cm in the 40% Technosol and $18 - 20$ cm in the 80%. The forest soil behaved as expected of a Podzol, starting at 6.2 in the LFH horizon before dropping to around 4.7 at lower depths. The field soil pH was more similar to the our younger Technosols; in the upper layers it was around 7 before increasing to over 9 at depth.

Figure 17. pH_w of 40% and 80% organic Technosol profiles, successional field, and forest profiles.

The pH_{Ca} of the parent materials was an order of magnitude lower than the pH_w for both rock types: 7.51 for intermediate volcanics and 7.44 for metasedimentary. The woody residuals were slightly lower as well, at 6.31. Profile pH measurements were also consistently lower, as is common when comparing pH_{Ca} to pH_{w} . Again, the forest soil had a much lower pH than the field or any of the Technosol profiles ([Figure 18](#page-86-0)). The 40% organic vegetated and non-vegetated

profiles had similar pH and were consistent throughout the profiles. The 80% organic profiles had a slightly lower pH on average than the 40% , but also contained a high peak over 9 at $16 -$ 18 cm in the vegetated plot and 25 – 30 cm in the non-vegetated. The field pH is similar to the 80% organic, with a smaller peak at $16 - 18$ cm, and another peak at the base of the profile.

Figure 18. pH $_{Ca}$ of 40% and 80% organic Technosol profiles, successional field, and forest profiles.

Electrical conductivity in the 40% organic Technosol profiles was on average about 40 μ S/cm higher than in the 80% organic Technosol, around 100 μ S/cm ([Figure 19](#page-87-0)). There was little difference between the vegetated and non-vegetated profiles; the non-vegetated high organic typically had slightly higher EC but there was no clear pattern between the two low organic profiles. Both forest and field had high EC in the $0 - 2$ cm layer, but quickly dropped to below the Technosol levels by 6 cm in the forest soil and even with the 80% organic by 8 cm in the field. Unlike the forest and the field profiles, the EC was consistent throughout the Technosols.

Figure 19. Electrical conductivity of 40% and 80% organic Technosol profiles, successional field, and forest profiles.

Soil temperature

Soil temperatures in Technosol plots were measured from Sept 20, 2012 to Nov 4, 2016. Comparisons between the two vegetated and one non-vegetated plot for each treatment showed minimal differences between the two at all depths, therefore the three were averaged into one daily temperature reading per treatment. The difference between the 30 cm and 60 cm plots was also minimal for both the high and low organic Technosols, so it was decided to display only the 60 cm thick plot temperatures to see the full range in depths.

The largest differences in temperature with depth are seen in the 80% organic soil ([Figure](#page-89-0) [20](#page-89-0)). There is much greater variation in the shallow depths than at 60 cm, and the temperature is more extreme. In all Technosol plots it appears that the winter temperatures are increasing with

time while the summer temperatures remain consistent. The plots also all had a 'shoulder' at 60 cm and to a lesser extent at 30 cm during spring: temperatures were flat around zero and then sharply increased until summer temperatures are reached. This was seen in the field and forest soils as well; however, it was visible at all depths although it was most pronounced at the base of the profiles.

Differences were seen between the two levels of organic content, particularly at 60 cm during the winter ([Figure 21](#page-90-0)). The 40% organic Technosol froze earlier and was a few degrees colder than the 80% organic. It also thawed earlier and was a few degrees warmer throughout the summer. Both showed similar summer temperatures to the field soil at this depth, but were noticeably colder in the winter; the field soil remained several degrees above freezing while the Technosols dropped to 0°C and below for the duration of the winter.

The temperature of all four soils (Technosols, field, and forest) became more variable at shallower depths and reaches greater extremes. This was most evident at 5 cm, but was also visible at 10 cm ([Figure 22](#page-91-0)), where daily temperatures in the Technosol plots during the summer were approximately equal to the air temperature. The difference between the two organic contents was minimal at these shallow depths but there was a large difference in the winter temperatures of the Technosols and the comparison soils. Again, the field soil remained several degrees above freezing while the forest soil remained at or slightly below 0°C, in contrast to the Technosols which in 2015 reached -10°C. In summer, the Technosols were around the same temperature as the field soil but with more variation. However, they are 5°C warmer on average than the forest soil.

Figure 21. Mean daily soil temperature at 60 cm depth in 40% and 80% organic 60 cm plots (n = 3) and successional field from Aug 1, 2014 – Nov 4, 2016. Air temperature at 2 m is also shown.

Figure 22. Mean daily soil temperature at 10 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil from Aug 1, 2014 – Nov 4, 2016. Air temperature at 2 m is also shown.

Soil moisture

Soil moisture was measured as volumetric water content (m^3/m^3) within the plots over the same period as the soil temperature (Sept 20, 2012 – Nov 4, 2016), though only data from May 1 to Dec 1 each year was kept, as moisture cannot be measured when water is in a solid state. The moisture was typically higher in the plots with vegetation than the plots without, though the magnitude of the difference varied. The only exceptions occurred at 10 cm in the 40% organic plots; in both the 30 cm and the 60 cm plots the moisture was higher in the non-vegetated plots, though the difference was greater in the thin plot. The 40% organic, 30 cm plot also showed no difference between vegetated and non-vegetated plot moisture at 30 cm, unlike the other three treatments which had higher moisture in the vegetated plots.

In the 40% Technosol plots, the moisture in the thin plot at 5 cm was lower than the thick plot, but higher at 30 cm. The opposite trend was seen in the 80% organic plots over the same period. For both 40% and 80% organic Technosols at 10 cm, the moisture was initially higher in the thin plots but around July 2014 it was equal in the thick plots, and by 2015 the moisture at 30 cm was higher in the thick plots.

In terms of differences between the two organic contents, in plots of either depth the higher organic Technosol had higher moisture content at 5 and 10 cm, but lower or equal at 30 cm and 60 cm.

Overall the moisture at 5 cm was the most variable and lowest, except for the 80% organic 30 cm plots where it was largely overlapping the other depths. In the thick plots the moisture was highest at 60 cm and either the same or higher at 10 cm than at 30 cm ([Figure 23](#page-94-0)). The moisture in the plots roughly tracked precipitation, with factors such as temperature and wind speed likely the cause of the differences observed.

The soil moisture in the field was consistently higher than either the low or high organic Technosol at all depths other than 60 cm, where it was approximately the same. Field moisture was more variable than the moisture in the test plots, though not as variable as the moisture in the forest. Forest soil moisture initially started higher than the Technosol plots, around the same as the field, but dropped throughout the first several months of the growing season until it was lower than the Technosol soil moisture at 5, 10, and 30 cm in August. Forest moisture then rose until it reached levels similar to the Technosols by the end of November ([Figure 24](#page-95-0)). The forest moisture reaches field levels by the end of the season, noticeably higher than the moisture of the Technosols.

Figure 23. Mean daily volumetric water content in 40% organic, 60 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3) with total daily precipitation. Data from winter months (Dec 1 – May 1) excluded.

Figure 24. Mean daily volumetric water content at 5 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil with total daily precipitation from Aug 1, 2014 – Nov 4, 2016. Data from winter months (Dec 1 – May 1) excluded.

Microbial functional diversity

The average well colour development (AWCD) of most depths in all profiles showed an initial lag phase from inoculation to 24h; between 24h and 72h there was a sudden increase in OD590, with a tapering off observed from 72h to 96h. In Technosol profiles the maximum absorbance was just over 1.00, while in the comparison forest and field profiles the top layers in the profile had absorbances over 1.50. The comparison forest profile had the most variation in AWCD with depth, with some of the middle layers of the profile having absorbances less than 0.50 after 96h. The field profile showed less variation, and had a general pattern of higher AWCD in the upper soil layers [\(Figure 25\)](#page-97-0). The high organic profiles had least variation and no pattern with depth. The unvegetated high organic profile had an OD_{590} between 0.75 and 1.00 at 96h in all depths. The low organic Technosol profiles had more variation and lower AWCD in the top layer of soil. This is particularly clear in the vegetated plot, which had very little colour development in the top 8 cm of soil $(OD_{590} < 0.25$; [Figure 26](#page-97-1)).

ANOVAs comparing the AWCD, richness (*R)*, evenness (*E*), and Shannon diversity (*H)* at 48h between profiles and depths revealed that while there were no depth differences for any of the four variables, there were significant differences between profiles. The AWCD was significantly higher in the successional field than the vegetated low organic Technosol and forest profiles. *R* was significantly higher in the field and non-vegetated, high organic Technosol plots than in the vegetated low organic Technosol and forest profiles [\(Figure 27\)](#page-98-0). The forest profile also had significantly lower *R* than the non-vegetated low organic and vegetated high organic Technosol profiles. *H* was significantly lower in the vegetated high organic Technosol than the unvegetated low organic one. *E* was not significantly different between any of the six profiles.

Figure 25. Average well colour development (AWCD; 590 nm) of the successional field profile over 96 hours post-inoculation.

Figure 26. Average well colour development (AWCD; 590 nm) of the 40% organic vegetated Technosol profile over 96 hours post-inoculation.

Figure 27. Richness (*R*) of soil profiles at 48h measured as number of substrates with $OD_{590} > 0.1$; letters indicate significant differences as determined by Tukey HSD. Profiles are 3A: low organic, unvegetated; 3C: low organic, vegetated; 4B: high organic, unvegetated; 4C: high organic, vegetated; FI: field; FO: forest.

In total six PCAs were performed using colour development at 48 h (all samples, no forest samples, no forest or field, samples with $AWCD > 0.1$, samples with $AWCD > 0.1$ and no forest, samples with $AWCD > 0.1$ and no forest or field) with the aim of increasing the separation between explanatory variables and seeing site differences. However, there was no substantial difference between the PCA with all samples and any of the PCAs lacking samples, so only the full PCA will be discussed.

PC1 and PC2 explained 38.4% and 15.7% of the variance respectively. The forest profile samples separated out with only a slight overlap with some of the vegetated low organic Technosol samples [\(Figure 28\)](#page-99-0). The Technosol profiles were largely overlapping, and while some of the successional field samples separated, most were in the same area as the Technosols.

Figure 28. Principal component analysis (PCA) for all soil profile samples at 48h post-inoculation with 95% confidence ellipses. Profiles include 40% organic Technosols with and without vegetation, 80% organic Technosols with and without vegetation, the successional field, and forest soil.

The majority of substrates were positively correlated with PC1 while about half were with PC2. Seven of the ten highest-loading substrates on PC2 were carbohydrates while the highest loaded onto PC1 were more diverse, including three carbohydrates, three amino acids, two amines, and one each of phenolic compounds and carboxylic acids ([Table 5](#page-100-0)). N-acetyl-Dglucosamine, putrescine, and L-arginine had loadings with a magnitude > 0.2 on both PC1 and PC2. In general substrates were positively correlated with PC1, while on PC2 about half of the substrates had a negative correlation.

	Substrate	Guild	Loading
PC1	D-Mannitol	Carbohydrate	0.256
	4-Hydroxybenzoic Acid	Phenolic compound	0.252
	L-Asparagine	Amino acid	0.248
	L-Serine	Amino acid	0.248
	D-Galacturonic Acid	Carboxylic acid	0.236
	Putrescine	Amine	0.232
	D-Galactonic Acid-y- Lactone	Carbohydrate	0.228
	L-Arginine	Amino acid	0.228
	Phenylethyl-amine	Amine	0.202
	N-Acetyl-D-Glucosamine	Carbohydrate	0.200
PC ₂	β -Methyl-D-Glucoside	Carbohydrate	-0.332
	D-Cellobiose	Carbohydrate	-0.318
	D-Xylose	Carbohydrate	-0.295
	<i>i</i> -Erythritol	Carbohydrate	-0.268
	Glucose-1-Phosphate	Carbohydrate	-0.259
	D-Glucosaminic Acid	Carboxylic acid	-0.257
	N-Acetyl-D-Glucosamine	Carbohydrate	-0.236
	α -D-Lactose	Carbohydrate	-0.236
	Putrescine	Amine	0.232
	L-Arginine	Amino acid	0.216

Table 5. Substrates with the ten highest magnitude loadings on PC1 and PC2 from the soil profile comparison PCA.

Six substrates were selected for an examination of their colour development with time: the amine putrescine, the carbohydrates D-mannitol and β-methyl-D-glucoside, the carboxylic acids α-ketobutyric acid and D-glucosaminic acid, and the phenolic compound 4 hydroxybenzoic acid. The development of 4-hydroxybenzoic acid, D-mannitol, and Dglucosaminic acid in the six profiles was similar to the average well colour development, with increases beginning between 24 and 48 hours post-inoculation for most samples and the forest profile having the most spread. Putrescine was also similar until 72h, after which there was a levelling off in most samples. 4-hydroxybenzoic acid, D-mannitol, and putrescine were over 65% correlated with each other, while D-mannitol and D-glucosaminic acid are 68% correlated.

β-methyl-D-glucoside was also correlated with D-mannitol (54%) but had a very different pattern of colour development. In the Technosol profiles most of the samples remained at or below an absorbance of 0.05, while in the field and forest a few samples, typically those from shallow depths, reached absorbances around 0.1 or greater. The trend in α-ketobutyric acid, which was not highly correlated with any other substrates, was similar in that many samples remained below 0.1 in the Technosol profiles, with only a few increasing from 72 to 96 hours, and with the forest and field having more samples which increased to between 0.2 and 0.4 in the same time period.

Discussion

Profile development

Unlike the comparison forest and reclaimed successional field profiles, there was little to no evidence of horizon development within any of the four Technosol pits. Between the 40% and 80% organic Technosols the main difference was unsurprisingly the amount of woody debris and the colour of the soil; the 40% organic Technosol was a greyish colour while the 80% was more

a brown-grey. The surface of the Technosols was different as well; though both types had a coarse layer at the top with the fines washed out to several cm below, the remaining coarse fraction on the 40% organic was mostly mine rocks, while on the 80% organic it was mostly wood chips.

The abundance of roots in the vegetated Technosols, particularly the numerous fine roots in the upper 20 cm, was an immediate obvious difference between vegetated and unvegetated plots. Upper layers in vegetated plots with ryegrass roots held together better while the profiles were dug than the unvegetated plots lacking these roots. Roots are known to form dense networks and stabilize profiles (Angers & Caron, 1998), so this result was expected. The fact that effects were seen though the ryegrass lived only a few months is also not surprising; grasses typically have been seen to have a rapid stabilization effect (Angers & Caron, 1998).

Closer inspection revealed that the vegetated Technosols also had more structure, being granular rather than fine granular. These findings indicate that the vegetation sped the Technosol development. Plant roots are known to improve aggregation by directly binding smaller particles with fine roots or by the release of organic compounds which act as binding agents (Angers $\&$ Caron, 1998), and this was probably the cause of the change seen in the vegetated plots. Plant roots also affect soil porosity; the compressive and shear stresses roots apply to the soil result in pores being created and enlarged, increasing the conductivity of the soil (Angers & Caron, 1998). This becomes more pronounced when plant roots decay and leave pore space behind, increasing infiltration (Angers & Caron, 1998). Because the ryegrass roots were so numerous throughout the upper 20 cm and because none survived the first year, it is possible these decaying roots might have a large effect on the infiltration rate in the top layers of the vegetated Technosols.

The accumulation of organic material at the surface is often one of the earliest processes which occurs during Technosol pedogenesis (Huot, Simonnot, et al., 2015), but no evidence of an organic layer forming was found on the vegetated Technosols. However, at the end of the growing season litter accumulation was observed under large alders on all vegetated plots (Chapter 4), so it is possible that a re-examination of the profiles in several years would reveal the beginnings of an LFH horizon. It is also possible that if the profiles had been dug closer to the alders, more changes to both soil structure and horizon development would have been seen. However, in this instance the preservation of the alders was important for other aspects of the study and therefore the areas immediately under the alders was not dug, meaning that any very local effects would have been missed by this assessment.

pH changes in the upper profile layers are also typically present in the early stages of Technosol pedogenesis (Scholtus et al., 2015). However, our analysis of pH and conductivity through the Technosol profiles did not show a clear pattern of change in either direction for any. The pH of the soil throughout the profiles was quite high, averaging around 8 for high organic Technosols and 9.5 for the low organic when measured in water. While these results are somewhat consistent with the pH of the water samples collected from the Technosols over the past four years, which ranged from $7 - 8.3$ (Chapter 2), it is quite high in comparison to most soils, particularly those in the boreal shield. Similar soil pH results have been found on sites in Alberta, where the natural soil pH is normally below 6.0 but on reclaimed oil sand sites can exceed 8.0 (Calvo-Polanco, Zhang, Macdonald, Señorans, & Zwiazek, 2017), likely due to calcareous parent material. Local natural soils, such as the forest soil used as a comparison in this study, are mainly podzols and have a pH typically closer to $4 - 5$ than $8 - 9$. As discussed in Chapter 2, the mine rocks used to manufacture the Technosol have a high acid neutralization

potential which was clearly enough to raise the pH despite the addition of large amounts of boreal coniferous woody residuals with a pH of 6.5. The fact the pH of the 80% organic Technosol was approximately an order of magnitude lower than the 40% organic Technosol means the increasing organics did have an effect on the pH, but not a large enough one to bring it down to slightly acidic or even neutral levels. This may pose problems as it could exceed the pH limits of native boreal vegetation, many of which are not well known (Calvo-Polanco et al., 2017). High pH may change the nutrient accumulation and growth patterns of plants on the Technosols (Calvo-Polanco et al., 2017). It could also result in microbial communities which are very different from those in the surrounding natural soils, which again could influence the vegetation as well as nutrient cycling within the Technosols.

The pH at the surface of the vegetated plots unexpectedly appeared to have increased compared to the unvegetated plots, a result possibly due to natural variation in pH through the Technosols. The litter of green alders is known to decrease surface pH, as was seen a study on succession in Alaska where pH decreased from 8 to 5 under alder litter. However, the sites in this study were 35 – 50 years old (Crocker & Major, 1955), unlike our four-year-old Technosols. The Technosols may not have been mature enough for the alder to have had a large influence on the soil pH. Many of our alders were also fairly small, so the observed litter accumulation (Chapter 4) probably had a very limited area of influence.

 pH_{Ca} is generally considered a more consistent measurement than pH_w because it is less affected by the soil electrolyte concentration (Minasny, McBratney, Brough, & Jacquier, 2011). In this study, the difference between mean pH_w and pH_{Ca} was 1.2 in 80% organic Technosols (8) to 6.8) and 2 in 40% organic (9.5 to 7.5). The differences between the two are due to soil exchangeable cation concentrations, as the Ca^{2+} exchange with H^+ and Al^{3+} on soil particles

reduces the pH of the solution (Minasny et al., 2011). The drop in pH is less as electrical conductivity (EC) increases (Minasny et al., 2011). The relative low EC throughout the profiles, under $150 \mu S/cm$, can therefore explain the large differences between the two.

EC is an indirect measurement of the total dissolved ion concentration or ionic strength of the soil solution (Brady $\&$ Weil, 2010), thus the low values in this study make sense considering the high amount of organics in the Technosols. The effect of increasing organics induces the approximately 40 µS/cm drop from the 40% organic Technosol profiles to the 80% organic Technosol profiles, from 100 μ S/cm to 60 μ S/cm; the decreasing mineral content results in decreased ion content in the soil solution.

In Chapter 2 the conductivity of the water percolating through the plots and equilibrated at the base of the Technosol layer within the plots was measured. The values obtained were substantially higher than the EC values found in the soil water extract solutions, particularly in the gravity through-flow samples which frequently reached an EC of $1000 \mu S/cm$. These higher values were likely due to the transmission of the water, which first flowed through the Technosols, then through a layer of crushed mine rock, then coarse mine rock before entering the collection barrel. This flow path exposed the water to a much larger amount of rock and new sources of ions, allowing dissolution of larger concentrations of dissolved ions.

The tension water also had noticeably higher EC than the 1:4 (m/v) equilibrium water, typically $400 - 500 \mu S/cm$. Unlike the through-flow, this is water that remained within the Technosol pedonmatrix. The higher values here were probably due to the length of time the water was held in the Technosol between sampling periods. Tension water was sampled approximately once a month during the growing season, giving the water ample time to equilibrate with the Technosols and react with the surrounding material, accumulating more

dissolved ions than the soil profile samples which were equilibrated only for 30 min – 1 hour before being read.

Soil microclimate

Continuous monitoring of temperature and moisture within all Technosol plots from 2012 to 2016 gave an understanding of the year-to-year temporal variability of the soil microclimate at various depths as a response to changing environmental conditions, as well as changes due to maturation of the Technosols. There is a noticeable difference in the extent of temperature variation at the different depths in all Technosol plots, with lower depths exhibiting far fewer peaks and lesser extremes. The 60 cm depth in particular does not show the diurnal temperature spikes observed in the 5 and 10 cm temperature data. This observation suggests the insulating effect of soil material increased with depth.

The increased moisture in lower depths of the Technosol is also consistent with the initial hypotheses. The 60 cm plots were constructed to test the hypothesis that thicker plots would have increased moisture at depth (Watkinson, 2014), and the recorded data generally indicated this in both Technosols. There was a general relationship between precipitation and soil moisture content, particularly in the 5 cm layer. The increases or decreases in moisture did not always directly correlate, likely due to temperature. The decreased temperature in the fall resulted in decreased water loss, so despite no increases in precipitation the soil moisture could increase, particularly in the upper soil layers. The lower layers also display less moisture content variability, similar to soil temperatures.

There is large seasonal variability in the Technosol soil temperatures, with the upper layers being over 20 °C in summer and decreasing nearly to -20 °C the first two winters. Soil temperatures in the following two winters were not so low, with around -10 °C being the

minimum. The weather station on site was not installed until 2015, so the air temperature comparison at the site does not show whether the difference was due to increasing insulating effects of the soil as the Technosols mature, or whether the first winters were simply colder than the previous two. However, the winter of 2015-2016 was not as cold as 2014-2015, explaining some of the difference in soil temperature. Summer soil temperatures over the monitoring period are relatively stable, with maxima between $20 - 25$ °C. This trend of increasing winter soil temperatures and unchanging summer ones was observed in all plots regardless of which Technosol was used to build them, though the higher organic content of the 80% organic Technosol did appear to have a moderating effect on the soil temperatures. This was particularly noticeable at the 60 cm depth sensor: in summer, the 80% organic tended to be a couple degrees cooler at 60 cm and a couple degrees warmer through most of the winter.

Large differences in the winter temperatures between the comparison field and forest and the Technosol plots were obvious. The field soil matrix remained above 0 °C at all times, while the forest soil was at or just below 0 °C. The difference in soil temperatures was probably due in part to the increased exposure of the Technosols plots, which may prevent the formation of a large insulating snow pack on the plots. The raised structure may also have left parts of the sides exposed, allowing colder air to enter laterally at lower depths, something not possible in soil forming the ground surface.

Technosol summer soil temperatures are much warmer than the forest soil temperatures, probably simply due to the amount of solar radiation the Technosols receive compared to the solum in th shaded forest site. The Technosols are similar to the successional field site, which is covered mainly with grasses and herbaceous plants in the vicinity of the sensors and scattered immature spruce, poplar, and tamarack further back. Field soil temperature peaks did not quite
reach the maxima of Technosol peaks, but were typically one or two degrees lower. The temperature data implied summer soil temperatures at least should not prevent the growth of vegetation on the Technosols, though the freezing winter temperatures may pose more of a challenge for the survival of plant roots. The cold winter temperatures could also impact the microbial community present within the Technosols. As the natural soils in the region never go below freezing, the low thermal regime of the Technosols may contribute to differences between their microbial communities and those typical of the natural soils of the area.

Previous research determined the field capacity of the 40% organic Technosol was 0.14 m^3/m^3 , while the 80% organic was 0.17 m^3/m^3 (Watkinson, 2014). The moisture in the high organic Technosol plots was therefore expected to be higher throughout all depths (Watkinson, 2014). Monitoring over the four years revealed this was partially true. Moisture content of the high and low organic Technosols is similar, with the high organic having higher moisture at 5 cm, 10 cm, and about half the time at 30 cm. However, at 60 cm the low organic matrix appeared to have higher moisture. The lower moisture at 60 cm in the 80% organic could be due to more water being retained in the upper layers of the soil, with less percolation reaching the base of the Technosol layer.

Both Technosols have lower moisture than the successional field, and they do not experience the high spikes that are seen at various times at 5 cm in the field soil which are likely caused by runoff after heavy or repeated rain events. The forest soil has a very different pattern from either the Technosols or the field; though it starts at comparable moisture levels to the field, by mid-summer it has dropped below the Technosols and only begins to climb again in fall. This is probably due to uptake and transpiration by the surrounding trees; the trees in the forest are more mature and much closer to the sensors than the trees in the successional field.

The effects of vegetation establishment on the Technosol microclimate were also examined as part of the monitoring plan. No differences were seen in the soil temperature of unvegetated and vegetated plots, which is not surprising as the vegetation cover on the plots is still sparse and large areas are not shaded during the summer, or covered with insulating litter in the winter. Many of the plants are also still small, so large drifts and piles of snow cannot be accumulated around them, and they will not prevent snow from reaching the ground in fall or from melting in the spring.

The effect of vegetation on moisture is more interesting. In all treatments, moisture was increased at 5 cm by the presence of vegetation, particularly after summer 2015 when bearberries and additional green alders were planted. As previously discussed, plant roots can increase the conductivity and infiltration of the soil (Angers & Caron, 1998) and it seems likely that is the cause of the increased moisture. Higher moisture levels in vegetated plots were seen at all depths in the 80% organic Technosols, though the difference was small at 30 cm in the 60 cm thick plot. This suggests that despite the water being removed from the soil by transpiration, the increased infiltration from roots and increased holding capacity due to high levels of organics is enough to keep the volumetric water content of vegetated plots higher than unvegetated.

In the 40% organic, 30 cm Technosol plots, vegetated plots had higher moisture at 5 cm, lower moisture at 10 cm, and equal moisture at 30 cm. The 60 cm plots had higher moisture in vegetated plots at all depths except 10 cm, where unvegetated were higher. The 10 cm sensor is within the main rooting zone of the plants and this is likely the depth where they remove the most water. The lower holding capacity of this Technosol could mean that it is unable hold enough water for the increased infiltration into vegetated plots to mask the effects of increased water loss from evapotranspiration at this depth.

The permanent wilting points of the Technosols were previously found to be $0.03 \text{ m}^3/\text{m}^3$ soil moisture for the 40% organic and 0.05 m^3/m^3 soil moisture for the 80% organic (Watkinson, 2014). In all plots, only the 5 cm layer ever approaches or falls below these levels. This means that vegetation with mainly shallow roots may struggle to obtain adequate moisture, particularly during summer months. However, plants with deeper roots should be able to survive these drier periods.

Microbial functional diversity

Differences between the Technosol plot microbial communities and the two comparison soil communities are expected based on the differences in properties observed above and differences in the age of the soils. The Technosols on the plots are thought to have had little to no biological activity after their initial construction four years ago, with other researchers documenting that soil microbial community development in manufactured soils can take many years to reach levels resembling those of natural communities (Dimitriu et al., 2010). Soil microbial communities are affected by the mineral composition and the quality and quantity of organic material in the soils (Ditterich et al., 2016). In the case of the current Technosols there were clearly differences in the amount and distribution of organic material throughout the profiles when compared to that observed in the natural forest profile. The mineral composition may also have been different, as the Technosols were composed of crushed rock which would usually only have provided available nutrients through the slow weathering of bedrock underlying the soil. This low nutrient supply rate was in addition to the differences in pH, EC, and soil microclimate observed earlier in this chapter, which are also likely to impact community composition.

The average well colour development (AWCD) of the six profiles shows large differences between the Technosols and the comparison sites. Technosol plot profiles all had similar AWCD and had the highest AWCD in the lower depths, while the field Technosol profile AWCD was highest in the surface layers. The forest profile had much more variation in AWCD between depths than all other profiles. The fact that the Technosol plots had similar AWCD patterns means they probably have communities and quantities of microorganisms more similar to each other than to the comparison communities. This suggestion fits with the differences observed between the Technosol properties and those of the more natural soils, specifically pH and microclimate. Soil matrix pH is known to affect microbial community structure in the boreal forest, as does the concentration of nutrients and contaminants within the soil (Pennanen, 2001). These changes can alter the proportions of bacteria and fungi within the soil profiles (Pennanen, 2001), which would influence the results of the Biolog® procedure. As fungi are not able to reduce the dye used on the plates, any shifts in their abundance or diversity will not be detected using this method (Preston-Mafham et al., 2002). This could explain why the forest depths had such low AWCD, as fungi are an important part of the boreal forest ecosystem and form mycorrhizal associations, particularly with conifers (Price et al., 2013). In any event, this preliminary examination of the microbial community functional diversity can confirm that there are differences between communities in natural soils and communities in the Technosols, even if the nature of those differences cannot be described in detail.

ANOVA results for AWCD confirmed the visual impression of differences between profiles, as the forest soil horizon samples had significantly lower AWCD than the field Technosol profile. The forest pedon samples also had significantly different richness (*R*) and evenness (*E*) from all other profiles except for the vegetated 40% organic Technosol pedon. This

pedon was the one most similar the forest pedon. The low colour development in the top 10 cm of the vegetated 40% organic Technosol profile resembled the low colour development at many depths in the forest profile. This low colour development could be due to the sample collection and storage procedures; with transport and storage causing lower viability in the bacterial communities. However, since the samples were not all processed in the same batch the possibility an inoculation error occurred on three separate occasions is low.

Richness (*R*) was significantly lower in the forest profile than in all other profiles except the vegetated low organic Technosol, the same as the AWCD. This indicates that fewer substrates were used in the forest and vegetated low organic Technosol profiles, possibly because of increased fungal activity, which is not measured by EcoPlates™. Only the vegetated high organic and non-vegetated low organic profiles had significant differences in Shannon Diversity (*H*): substrate usage was significantly less diverse in the vegetated high organic Technosol profile. The higher content of woody organics may have meant an abundance of certain substrates in this Technosol, promoting the growth of bacteria specialized in their consumption and leaving the community function skewed towards the metabolizing of those types of carbon sources. Vegetation can also impact microbial communities, and again may have promoted the growth of a set of bacteria specializing in a less diverse set of substrates. There were no significant differences in the evenness (E) of the six profiles. In this case, E is measuring whether the activity levels in the substrates which were utilized by the microbial communities were approximately equal (utilization being defined as an $OD_{590} > 0.1$). All of the six profiles in this study had even substrate usage 48 hours after inoculation, as all had *E* close to 1.

The separation of the forest profile from the Technosol profiles in the PCA provided further evidence of differences between the natural microbial communities and the Technosol

communities. The successional field Technosol profile overlapped with the plot Technosol profiles to a much greater extent, though it did extend further along the PC1 axis than the plot Technosols. This Technosol is older than the plot Technosols and may not contain mine rock, but rather is composed of stockpiled soil. Few differences were seen between it and the plot Technosol profiles, consistent with the fact that chemical properties of this profile such as pH and EC were similar to those of our plot Technosols. The largest difference between the pedons was in the soil microclimate data; unlike the Technosol plots, the successional field pedon temperatures remained above 0 °C at all times and the soil moisture was consistently greater. However, overall it appears that the young age of the successional field Technosol and its similar chemical properties to our mine rock-based Technosols have resulted in the development of similar microbial communities.

PC1 had a diversity of substrates with positive loadings, including amino acids, carbohydrates, amines, a carboxylic acid, and a phenolic compound. Other studies have found that PC1 reflects variation in inoculum density (Glimm, Heuer, Engelen, Smalla, & Backhaus, 1997; Lawley & Bell, 1998), which could explain the separation of the forest profile from the other profiles on the PC1 axis. The substrates that are more highly loaded on PC1 are those with higher colour development, suggestive of greater utilization by the community present, therefore inoculum density has a higher impact. In contrast, PC2 had negative loadings of carbohydrates and positive loadings of nitrogen-containing compounds. Carbohydrates are typically considered energy storage compounds, so their preferential metabolization suggests the bacteria are taking advantage of the available nutrients and experiencing a period of rapid growth. Research has suggested that EcoPlates™ favour fast growing bacteria that thrive under high nutrient conditions (Preston-Mafham et al., 2002). The differences in communities along PC2 may

indicate population size variability of the generalists able to take advantage of the high nutrient conditions.

4-hydroxybenzoic acid, D-mannitol, and D-glucosaminic acid all had the same pattern of colour development. 4-hydroxybenzoic acid is a phenolic derivative of benzoic acid, and among other things is formed during ubiquinone synthesis in Gram-negative bacteria. D-mannitol is one of the most common energy and carbon storage molecules, and D-glucosaminic acid is a component of bacterial lipopolysaccharides, which are found in the cell membrane of Gramnegative bacteria. The other compound which follows a similar utilization pattern over 72 h is putrescine, an amine formed during decomposition by the breakdown of amino acids. The fact that these compounds are all commonly produced by bacterial metabolic processes could indicate that the microbial communities in the Technosols are dominated more by bacteria than other microorganisms such as fungi, and are therefore more able to degrade these compounds.

β-methyl-D-glucoside, a monosaccharide derived from glucose, and α-ketobutyric acid, a carboxylic acid product of cystathionine lysis and threonine degradation, had little colour development in the Technosol profiles in comparison to the field and forest profiles. This utilization profile indicates differences in the capacities of the microbial communities to metabolize these compounds, although the nature of these differences is not known.

Conclusion

An examination of the Technosol profiles revealed few signs of early pedogenesis four years after construction. Increased aggregation was observed in the vegetated profiles, but little variation in colour, pH, or EC was seen with depth. Organic matter accumulation is occurring immediately beneath the large alders on the vegetated plots, but the area of influence remains quite small. Comparisons of the soil microclimate between the high organic and low organic

Technosols showed the high organic system had less extreme temperatures and higher moisture content overall, particularly at 60 cm in the thick plots. Vegetated plots appeared to have increased infiltration in the top 5 cm of both Technosols. Finally, differences between the Technosol soil microbial functional diversity and the natural forest soil community were observed, though the nature of these differences remains uncertain. These results indicate that development of the Technosol profiles is occurring slowly, and that increased vegetation establishment on the Technosols would likely contribute to an increasing rate of pedogenesis.

Chapter 4. Vegetation growth on Technosols designed for mine reclamation in the Boreal Shield

Introduction

Revegetation is a crucial component of mine reclamation and is important for long-term ecosystem stability (MacKenzie & Quideau, 2010). Vegetation establishment prevents site erosion, improves soil properties such as organic matter content, accelerates soil formation, increases nutrient cycling, promotes fungal and microbial community development, and improves aesthetics (Mukhopadhyay et al., 2016). As mining involves the removal of the natural soils and vegetation of an area along with the subsurface geologic material, revegetation often requires first an addition of soil materials to the site. As importing and spreading topsoil is an expensive option for remote mines in the Boreal Shield, the soil materials used are often the stockpiled soils and overburden materials removed during mine construction (MacKenzie & Quideau, 2010). These materials are often low in organic matter and nutrients, making the addition of amendments necessary to ensure successful plant growth (Young et al., 2015).

Often the preference is to use amendments with a local origin, such as peat salvaged from initial construction phase (MacKenzie & Quideau, 2010; Pinno & Hawkes, 2015). To reduce costs, when organic material from the site is not available, local industry waste products can also be used as amendments, including paper mill sludge and wood chips (Young et al., 2015) or sewage sludge (Bradshaw, 1997).

The resulting manufactured soils, classified as Technosols under the World Reference Base for Soil Resources (IUSS Working Group WRB, 2014), can have properties which limit plant growth such as compaction (Young et al., 2015), coarse textures and low moisture retention (Macdonald et al., 2015), and unfavourable chemical conditions (Mukhopadhyay et al., 2016). A common industry practice is to further modify the Technosols through the addition of

commercial fertilizers in order to improve their properties, another process which can be both economically and environmentally costly (Sloan et al., 2016).

Although there are numerous studies on the reclamation of sites in the Alberta oil sands region using Technosols manufactured from local waste products (e.g. Pinno & Hawkes, 2015; Sorenson, Quideau, MacKenzie, Landhäusser, & Oh, 2011), there are fewer studies on the reclamation of other types of mine sites with Technosols. Knowledge of how well native vegetation will establish and grow upon Technosols on the Boreal Shield would be beneficial, particularly as mining development in the north is likely to increase. To this end, a field study was conducted on a gold mine in Northern Ontario designed to assess the ability of Technosols of different depths and organic matter contents to support vegetation, and more specifically the survival, growth, overall health, and nutrient status of the selected species.

Methods

Site description

The study site was located at Barrick Gold Corporation's Williams Mine in Hemlo ON, north of Lake Superior and approximately 350 km east of Thunder Bay. This site is on the Canadian Shield, within the boreal forest ecosystem. Mean monthly temperatures range from 15°C in July and August to -14°C in January; precipitation varies from 122 mm in September to 47 mm in February (Environment Canada, 2017). The forest is dominated by a mixture of coniferous species including black spruce (*Picea mariana* (Mill.) B.S.P.), jack pine (*Pinus banksiana* Lamb.), tamarack (*Larix laricina* (Du Roi) Koch), and balsam fir (*Abies balsamea* (L.) Mill.), with deciduous hardwoods such as trembling aspen (*Populus tremuloides* Michx.), white birch (*Betula papyrifera* Marsh.), willow (*Salix* sp.), and alder (*Alnus* sp.) (Sims et al., 1996; Zoladeski & Maycock, 1990). Podzols, the most common soils in the region, have a

characteristic reddish illuvial (Bhf/Bf) horizon high in aluminum and iron often overlain by a grey eluvial (Ae) horizon from which these elements and soluble organic matter have been leached (IUSS Working Group WRB, 2014).

Plot design

Technosols were constructed in summer 2012 using non-acid generating metasedimentary and intermediate volcanic rocks, which were excavated as part of the open pit mining operations at the Williams mine, in combination with woody residuals from the White River Forest Products sawmill. The mill is located approximately 60 km from the site in White River; the woody residuals are primarily boreal coniferous sawdust, bark, and off-cuttings (Watkinson, 2014).

The woody residuals were combined with the rock in either an 80:20 or 40:60 ratio by volume to obtain high organic and low organic Technosols. These Technosols were layered in 30 cm or 60 cm depths over lysimeters constructed from coarse mine rock and capped with crushed mine rock. Each Technosol-depth combination was replicated three times for a total of twelve plots (Watkinson, 2014). Plots were then left to equilibrate for one year before vegetation planting.

Vegetation species

Native vegetation species selected as candidates for establishment on the Technosol plots were early successional species considered capable of living on dry soils with low nutrients, as the Technosols were not expected to have high quantities of either available nitrogen or phosphorus, and the water holding capacities of the plots are expected to be limited at least initially due to exposure to sun and wind, as well as the lack of silt and clay sized particles within the soil matrix.

The first species selected for establishment on the plots was green alder (*Alnus viridus* subsp. *crispa* (Aiton) Turrill). Green alders are native to the area and are an early successional species which can be found growing on disturbed sites under harsh conditions (Lefrançois et al., 2010). Alders are capable of nitrogen fixation due to their symbiosis with nitrogen-fixing actinobacteria *Frankia,* and so have been used on many reclamation sites in the hope that they will increase the access of neighbouring plants to nitrogen through time (Densmore, 2005). Though alders have been found to at times inhibit natural seedling establishment, their presence can promote the growth of established seedlings by improving soil quality and acting as a nurse crop (Densmore, 2005).

The second species selected for planting was common bearberry (*Arctostaphylos uva-ursi* (L.) Spreng.), also native to the area. Bearberry is a low-growing evergreen shrub which forms a ground cover and is commonly found on dry soils low in nutrients (Krpata et al., 2007). The shrub has a high capacity to regrow after damage and colonize nearby areas (Salemaa & Sievänen, 2002), making it a good candidate for reclamation activities (Krpata et al., 2007).

Vegetation planting

Green alders collected from a recently disturbed site along Highway 17 in August 2013 had a mean height of 17 cm. The roots were cleaned of soil before planting. Sixteen alders were planted on two of the three replicates of each plot type. In August 2015, additional alder seedlings of approximately the same size were removed from a roadside within the mine property and soaked in water for several hours before being planted on the plots to bring the number on each up to at least nine. At this time twelve bearberry plants (Connon Nurseries, Waterdown, ON) were also planted on each of the vegetated plots.

In April 2016 a mixture of annual and perennial ryegrass seed (Home Gardener Overseed Premium Grass Seed Mix with Kickstart™ Technology) was hand seeded over the vegetated plots in an attempt to establish a nurse crop to promote and protect bearberry growth.

Vegetation survival and health

Vegetation surveys were conducted approximately once per month throughout the 2016 growing season (May to September) to assess the survival and health of the vegetation on the plots. Bearberry health was assessed by colour: green, a mixture of green and red or yellow, and red or black. Bearberry plants which was entirely black were considered dead. Alders were assessed by percent leaf cover: 100, 75, 50, 25, or 0 %; alders with a leaf cover of 0% were assumed to be no longer living. The ryegrass was assessed solely by colour and did not survive long enough for either sampling or other measurements to be taken.

The final health survey of the year (September 2016) also included measurements of the maximum diameter and diameter at right angles to the maximum for bearberries, and the height and diameter for the alders. In addition, alder root development and nodulation was examined through the partial excavation of a large alder growing on a 30 cm thick, low organic Technosol plot.

Two-way ANOVAs with Tukey HSD tests were performed to determine significance of organic content and depth. One-way ANOVAs with Tukey HSD were also performed to examine treatment differences. The nonparametric Kruskal Wallis test with Dunn's post-hoc was used to examine alder height differences by treatment. Data were log-transformed when appropriate.

Repeated measure ANOVAs were performed on the health survey data for both alders and bearberries using the 'ez' package in R (Lawrence, 2016). One- and two-way ANOVAs with

Tukey HSD were also performed to examine differences. All statistical analysis was performed using R 3.3.2 (R Core Team, 2016).

Nutrient and trace element analysis

Samples were collected in September 2016 to assess the nutrient content of the vegetation. One alder sample was composited from each vegetated plot, consisting of 3 leaves from each plant (when possible). In addition, four alder samples were composited from the 2015 source site and three from nursery-stock green alder (St Williams Nursery & Ecology Centre, St Williams, ON) for comparison. One root sample was also taken from the partially excavated alder described above.

One bearberry sample was collected from each plot, consisting of one runner from each plant (when possible). For comparison, two bearberry samples were taken from a healthy stand in the same manner from alongside an old logging road off Highway 17, east of Hemlo.

Vegetation samples were dried overnight at 60°C and ground using a Thomas Wiley® Mini-Mill Cutting Mill. Samples (0.5 g) were weighed into 50 mL Teflon™ tubes, followed by the addition of 7.5 mL of trace metal grade HNO₃ and 2.5 mL of trace metal grade HCl for digestion at 110° for 240 minutes. Cooled samples were diluted with deionized water to 50 mL prior to analysis by ICP-MS. The QA/QC protocol included Certified Reference Materials, method blanks, and sample duplicates.

Fourteen elements were selected for examination: the macronutrients phosphorus, potassium, magnesium, and calcium, and the micronutrients boron, copper, iron, manganese, molybdenum, nickel, and zinc. Arsenic, antimony, and cadmium, which along with molybdenum are elements of environmental interest to the mine due to the presence of molybdenite, arsenopyrite, realgar, and stibnite in the deposit (Muir, 2002), were also quantified.

Results

Mortality

Mortality in the initial alder planting in 2013 was high, with a maximum survival rate of 50.0% at the end of the summer and a minimum survival rate of 6.25% per vegetated plot (Watkinson, 2014). Only 18 of these alders survived to 2015. Mortality after the 2015 planting was much lower; the lowest survival rate observed was 66.7%, while five of the eight plots had 100% survival. It should be noted that these survival numbers include both the alders planted in 2015 and the remaining alders planted in 2013.

The survival rate of bearberry planted in 2015 was also high: five plots had 100% survival, and the lowest survival rate was 83.3%. Plots 4A, 2B, 2C, and 1C had 100% survival of both bearberry and alder.

The ryegrass planted in April was on average 5 cm high in June, and germination appeared to have been in the previous week following a large rain event. The shoots were thin but green and healthy in appearance. However, by July the shoots were reddening, with no further growth observed. By the end of the season the ryegrass had completely died off, without further growth.

Plant size

Alder height and diameter were highly variable across the four treatments, particularly in the 30 cm, 40% organic treatment ([Figure 29](#page-123-0)). A visual assessment indicated lower median values of both height and diameter for the low organic (40%) soils; this was confirmed by the two-way ANOVA results (height: $F(1, 63) = 11.62$, $p = 0.001$; diameter: $F(1, 63) = 6.00$, $p =$ 0.017). Depth also had a significant effect on diameter $(F(1, 63) = 4.89, p = 0.031)$.

The Tukey HSD test following one-way ANOVAs comparing treatments revealed that the 60 cm, 40% organic treatment was significantly different from both the high organic treatments ($p = 0.007 \& p = 0.005$). The only significant difference between treatments for diameter was between the 30 cm, 80% organic and 60 cm, 40% organic treatments ($p = 0.008$).

Figure 29. (a) Height and (b) diameter of green alders on the Technosol plots in Sept 2016 (n = 19, 17, 16, 15).

Bearberry maximum diameters and diameters at 90° from maximum showed few treatment differences [\(Figure 30\)](#page-124-0). No significant differences were found through the two-way ANOVAs of organic content and depth or the one-way ANOVAs of treatment for either of the bearberry size measurements.

Figure 30. (a) Maximum diameter and (b) diameter at 90° from maximum of bearberries on the Technosol plots in Sept 2016 (n = 22, 24, 24, 24).

The root development of a large alder on a low organic, 30 cm Technosol plot was examined through partial excavation of the alder. The roots were numerous and extensive throughout the surrounding soil, with nitrogen-fixing nodules clearly visible ([Figure 31](#page-125-0)). At the end of the growing season leaf litter accumulation was observed under the large alders on all plots ([Figure 32](#page-126-0)).

Figure 31. (Left) Partially excavated alder from low organic, 30 cm thick Technosol plot with root structure visible. (Right) Green alder root with nitrogen fixing nodules.

Figure 32. Litter build-up under alders (November 2016).

Health

Alder health appeared to vary with treatment; the alders growing on the thick, low organic Technosol plots had noticeably fewer with full leaf coverage than those growing on other plots ([Figure 33](#page-127-0)). The percentage of alder with full leaf coverage increased slowly throughout the growing season for all treatments. The 30 cm, high organic treatment had no alders without leaf coverage by the end of the season, and the highest percentage with full coverage.

A repeated measures ANOVA on the percentage of alders with full leaf coverage found significant differences between months (F(3, 12) = 7.68, $p = 0.004$), and Tukey's HSD following the fitting of a linear mixed effect model revealed the differences were between June/July and August/September. ANOVAs by month revealed that for full leaf coverage, organic content was barely significant in September ($p = 0.049$).

For partial leaf coverage, both month and organic content were significant. Organic content was significant every month, while the difference in months was again between June/July and August/September. There was no significance found in the repeated measures ANOVA for alders with no leaf coverage.

Figure 33. Percentage of alders with each leaf coverage measure on each Technosol treatment throughout the summer of 2016. Treatments are 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80%, 60 cm.

Bearberry health was unlike alder health as it had an obvious time-of-year dependence [\(Figure 34\)](#page-128-0). There were no significant differences found between organic content or depths for bearberry plants of any colour, but the percentage of green bearberry in June was significantly higher than in any other month, while the percentage of plants which were both green and another colour was significantly less in April and June than in July, August, or September. Finally, red or black bearberry were significantly more common in April than in June, July, or August.

Figure 34. Percentage of bearberries within each colour category on each Technosol treatment throughout the summer of 2016. Treatments are 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80%, 60 cm.

Nutrients and trace elements

Macronutrients

The macronutrient content of the alders and bearberries grown on the Technosol plots appeared similar to those of the reference alder and bearberry plants. Phosphorus levels in the Technosol plot alder were lower than in the source site alder but close to nursery alder leaf concentrations, though some of the low organic Technosol samples contained less ([Figure 35](#page-129-0) upper left). The bearberry had a similar trend, with some of the low organic plots having lower P content than the comparison site bearberry. Potassium levels of both plot alder and bearberry were close to those of the reference vegetation ([Figure 35](#page-129-0) upper right).

Alder Ca concentrations in the Technosol plots were lower than the nursery alder, excluding the root sample, but approximately the same as in the alder source site samples. The bearberry Ca levels were generally higher on the plots than for the comparison site, with the

exception of two of the low organic Technosol plots ([Figure 35](#page-129-0) bottom left). Magnesium alder content was much higher in the root and nursery alder samples than in either the plot or source site alder samples, which had similar values ([Figure 35](#page-129-0) bottom right). The bearberry Mg was higher in the Technosol plots than the comparison site.

Figure 35. Macronutrient concentrations (phosphorus, potassium, calcium, magnesium) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Micronutrients

Concentrations of B in the Technosol plot alder leaves ranged from 17 mg/kg in two of the high organic plots to 35 mg/kg in a low organic plot. This was comparable to the B content of the nursery alders (25 mg/kg) but higher than measured in the source alders, which ranged from 3 to 13 mg/kg. Bearberry concentrations on the Technosol plots averaged around 14 mg/kg, which was slightly higher than the comparison site, at around 8 mg/kg.

Copper content in Technosol plot alders was around $3 - 6$ mg/kg, similar to the nursery alder concentrations but slightly lower than source site alder concentrations, which were around 10 mg/kg. The alder root sample had a higher Cu level of 18 mg/kg, while all bearberry samples contained approximately $1 - 3$ mg/kg, including the comparison bearberry.

The Fe content in the alder root sample was substantially higher than any of the leaf samples, being 7540 mg/kg. Technosol plot alder Fe concentrations ranged from 83 to 385 mg/kg, with the low organic Technosol plots typically having the higher concentrations. The nursery alder had concentrations equal to the lower end of this range, while the source site alder had concentrations similar to those of the higher end (up to 345 mg/kg). The bearberry Fe concentrations from the Technosol plots are much higher than in the comparison bearberry; plot concentrations range from around $75 - 100$ mg/kg in the high organic Technosols to 200 mg/kg in the low organic Technosols, while the comparison bearberries have an Fe content of only 3 – 17 mg/kg.

Manganese concentrations were much higher in Technosol alder than nursery alder (approx. $100 - 200$ mg/kg compared to 25 mg/kg), and also higher than most of the source alder samples (average 70 mg/kg). The high organic Technosols had lower concentrations than the low organic Technosols, more comparable to the source site alders. Root alder concentrations were

equal to the leaf concentrations in the low organic plots and one of the 30 cm high organic plots (over 150 mg/kg). Bearberry had much lower concentrations of Mn and appeared quite similar to reference bearberries, with an average of 20 mg/kg.

Nickel concentrations were much higher in the alder root sample than the leaf samples, at 20 mg/kg. Leaf concentrations appeared similar to comparison samples for both bearberry and alder, and in all cases are below 5 mg/kg.

The Zn content of the bearberries in the treatments was $100 - 140$ mg/kg, higher than in alder leaves, which ranged from 40 to 60 mg/kg, and slightly higher than the alder root which was 90 mg/kg. Zinc in Technosol plot bearberry was substantially higher than the Zn in the comparison bearberry, which is around 30 mg/kg.

Molybdenum

Molybdenum was present in all alder samples but only on two bearberry samples from low organic Technosol plots at approx. 3.3 mg/kg ([Figure 36](#page-132-0)), with all other samples being below the detection limit of 3 mg/kg. In alders, the root sample had noticeably higher Mo concentrations than the leaf samples (60.3 mg/kg). Alder leaves from the source site had higher concentrations than nursery alders. Alders from the high organic Technosol plots had lower concentrations than those in the low organic Technosol plots, which had concentrations comparable to the source site. High organic Technosol alders were close to or below nursery alder levels.

Figure 36. Molybdenum concentrations in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Elements of environmental interest

Arsenic was found only in the alder samples; all bearberry samples were below the detection limit of 0.2 mg/kg. There was no As detectable in the nursery alder samples, though it was seen in the alder samples from the source site at approximately 0.3 mg/kg. The alder from the high organic Technosol plots had As concentrations similar to those in the source alders, while the low organic Technosol samples were slightly higher (approx. 0.6 mg/kg). All alder leaf samples from both the plots and the source sites were below 1 mg/kg As, while the alder root sample contained 3.2 mg/kg.

Antimony was detected only in the alder root sample, at a concentration of 0.368 mg/kg; it was below the 0.07 mg/kg detection limit in all of the alder and bearberry leaf samples either from the Technosol plots or from the comparison vegetation.

Cadmium was not detected in any of the vegetation samples on the Technosol plots other than the alder root sample, at a concentration of 0.402 mg/kg. Cadmium was also found in two of the alder source samples, at concentrations of 0.0512 and 0.053 mg/kg, just above the detection limit (0.05 mg/kg).

Discussion

Mortality

The bare root transplant method, high August temperatures, and low moisture were thought to be the cause of the high mortality rates of green alder from the August 2013 planting (Watkinson, 2014). The planting method was therefore modified slightly for the 2015 planting, although planting still occurred in August. The soil was not deliberately removed from the roots in the hope this would preserve more of the fine root system. The alders were also soaked in water for several hours before planting to reduce wilting and stress to the plant while waiting for transplant. Once planted they were thoroughly watered again to provide easily accessible water to sustain them while their root systems became established in the soil. These measures appear to have been successful in reducing mortality, as 100% of the alders survived to the next spring on five of the eight vegetated plots. Therefore, if planting during spring when moisture is higher and temperatures are lower is not possible, survival can be dramatically increased by soaking and one watering event after transplanting. The bearberry also had high transplant survival rates with watering after planting; the only difference in methods was the bearberry were planted with the potting soil intact around their roots. Complete survival of alder and bearberry was seen in both

the thin 80% organic plots, as well as one thick 80% organic and one thin 40% organic plot. These success rates are also reflected in the size and health of plants; the thin 80% organic plots consistently had the best scores in terms of plant health.

The annual ryegrass had a good germination success rate with the April planting and light raking into the soil. The complete die-off of the ryegrass which occurred several months later appeared to be due to problems with moisture or nutrient deficiencies in the soil rather than the seeding method.

Plant size

The highly variable alder sizes, particularly on the thin 40% organic plots, were due in part to the presence of alder of different ages on the plots. Many of the 18 alders surviving since 2013 had grown fairly large by 2016, much larger than those planted in 2015. The new alders tended to die back during the months following transplant, losing leaves likely due to the sudden increase in exposure they experienced on the plots. Alder seedlings that survived the winter grew back with stronger stems and smaller leaves. Some alders appeared to die back, being only a few cm in size in April 2016. However, by the end of the season growth was observed on almost all of them.

Alders were significantly larger on the high organic Technosol plots. The high organic Technosol typically has higher moisture than the low organic, specifically in the upper 30 cm of the soil (Chapter 3). As this area of soil also has the largest number of roots (Chapter 3), it can be assumed that the vegetation is obtaining a large amount of its moisture requirements from these depths and therefore the increased amount available in the high organic could be the cause of the increased growth.

The depth of the plot was also important for the diameter of the alder; however, in this case it appears that thicker plots had alders with smaller diameters. This is contrary to expectations, as thicker plots were thought to hold more moisture and therefore promote growth (Chapter 3). The smaller sizes could have been due to the increased exposure of the thick plots. The extra 30 cm of Technosol on these plots raises them above the thin plots, which then have the extra benefit of increased shelter from the wind. The thinner plots do have higher moisture than the thick ones at 5 cm, though this changes at 10 cm (Chapter 3). The moisture difference could be the cause of the decreasing diameters, as the alders may need to develop a more extensive root system before they can take advantage of the higher moisture at lower depths.

The bearberry did not show any differences in growth habit. The lack of difference could indicate that the bearberries had not sufficiently established for the treatment differences to have affected their growth, but what is more likely is that the bearberry were already struggling on the Technosol plots and so very little growth was occurring.

The partial excavation of one of the large alders on a thin, low organic plot confirmed the roots were extensive throughout the 30 cm layer of the Technosol. It also confirmed what was suspected due to the deep green leaves and general health of the alders: nitrogen fixation nodules were plentiful on the root system. The nitrogen-fixing abilities of alders were one of the reasons they were chosen for the plots, in the hope that they would be able to survive the low nutrients and over time improve the soil quality, so the confirmed presence of nodules is encouraging.

The other effect of the large size of some of the alders was the build-up of fresh and decomposing leaf litter at the end of the growing season. As the alders grew, it was hoped they would break the wind over the plots to help promote the growth of other vegetation and allow the

accumulation of organic matter and snow. These litter mounds hopefully represent the beginning of humus form development critical for nutrient cycling.

Health

There were significantly fewer alders with full leaf coverage the thick, low organic Technosol plots than the other three plot types. Both time in the season and organic content of the Technosol influenced the number of alders with only partial leaf coverage, as alders continued to grow and leaf coverage increased throughout the growing season.

The high organic Technosol tended to have higher levels of moisture in the upper layers of soil where the roots are more concentrated. This Technosol also had a slightly narrower range between minimum and maximum temperatures (Chapter 3). Finally, concentrations of bioavailable macronutrients in the high organic Technosol were significantly higher than in the low organic Technosol. The high organic Technosol also had lower bioavailable Mo and Cd (Chapter 2) and a lower percentage of As which was bioavailable (Chapter 2). The more favourable nutrient conditions and lower amounts of potentially harmful elements may also have been a factor in the improved plant health on the high organic Technosol.

The reason the thin, low organic Technosol plots had comparable plant health results to the high organic plots is likely because these plots were less exposed to weather than either of the thick plots. Although the moisture in the top 10 cm was lower than any of the other plots, the moisture at 30 cm was higher (Chapter 3), a property which seems to indicate that the low organic Technosol could hold sufficient moisture for plants with deeper roots to thrive as long as the soil was not highly exposed. When exposure increases, as in the thick low organic plots, the soil appeared to be unable to hold enough moisture to compensate for the increased evaporation resulting from increased wind, and plant growth suffers as a result.

Bearberry again showed no differences between treatments, but season had a strong effect. At the end of April a large number of bearberry were in the red or black health category, which would normally be thought to indicate the plant was struggling. However, as bearberry is an evergreen shrub whose leaves turn a red colour during winter (USDA NRCS Plant Materials Program, 2006), the bearberry were probably still emerging from winter dormancy. In June 75% of bearberry plants were green, but that number decreased dramatically in the following three months. From July on, most bearberry had leaves which were partially yellow or red. Only 11 of the 98 bearberries showing signs of life by August 2017.This high mortality could be a consequence of an unsuitable soil habitat, or a result of poor growing conditions in the latter part of the growth season. As alder did well, the cause of poor bearberry growth is probably the nature of the soil matrix.

In the first case, the most obvious factor which could cause difficulties is moisture. Although we have seen the lower levels of the soil never fall below the permanent wilting point, the 5 cm layers may have done so during dry periods (Chapter 3). The bearberry rooting system also may not have been as extensive as the alders, preventing the roots from reaching the moisture zone at depth within the plots. The bearberry plants were also planted with their original potting soil intact around their roots in the hope this would increase their survival by providing a soil richer in nutrients and moisture during establishment. However, retention of the potting soil may have prevented the bearberries from sending out large numbers of roots into the less favourable Technosol surrounding them. This would in turn inhibit their ability to obtain the moisture required. Excavation of several bearberries in summer 2017 revealed that there were some roots leaving the potting soil ball, and that the roots were more extensive in the high organic Technosol. Overall though the bearberry root system appeared less well developed than

the alder root system, indicating that the bearberry may have had more difficulty obtaining water than alders. The lack of moisture at times during the summer and fall may have stressed the bearberries to the point where they could not survive the freezing temperatures of winter, causing the large die-off seen in 2017. The difficulty with this theory is that bearberry are known to survive on dry sites (Krpata et al., 2007), one of the reasons they were chosen as a reclamation species. The bearberry plants were nursery-stock grown in southern Ontario, with the genotype being less adapted to dry conditions and northern temperatures. The use of local bearberry could remove this possibility, however, bearberry germination success rates are low and cuttings may require additional treatment before they can be planted successfully (Smreciu, Gould, & Wood, 2013; USDA NRCS Plant Materials Program, 2006).

The second scenario is that environmental conditions were not the problem, but rather the chemical and nutrient conditions of the Technosols themselves. The nutrient status of the bearberries will be discussed in more detail in the next section, but generally the Technosols are not nutrient rich and thus nutrient deficiencies possibly contributed to the bearberry mortality. Bearberry are typically able to survive in low nutrient soils (Krpata et al., 2007), so it would be expected that any deficiencies resulting in plant death would be fairly obvious, which was not the case. It also appears that toxicity from the elements of environmental interest (Mo, As, Cd, Sb) was not a likely cause of bearberry mortality.

The high pH of the Technosols is a potential issue for plant growth. With a pH of $8.0 -$ 9.4, the Technosols are strongly alkaline, and the pH tolerance limits of bearberry are not well defined. Plant guides suggest bearberry require acid soils with pH limits such as $4.0 - 6.0$ (Prairie Nusery, 2017), under 6.8 (Wildflower Centre, 2013), or with a 5.5 to 8.0 range (Smreciu et al., 2013). One study documented high mortality at $pH 7.3 - 7.7$ (Howat, 2000). An in-depth

study of the effects of pH on boreal plants found that bearberry shoot:root ratio and transpiration rates declined slightly as pH increased, but that overall bearberry were not highly responsive to pH and could be a good candidate for revegetation on sites with a wide range in pH (Calvo-Polanco et al., 2017). However, this was a only short-term study, and that foliar concentrations of some nutrients decreased with increasing pH (Calvo-Polanco et al., 2017). This study suggests long-term exposure to high pH may still be a contributor to the die-off observed. The pH of the tension water samples taken from the base of the Technosol soil layer was typically $7 - 8.5$ (Chapter 2). Though this water may be below the reach of the bearberry root zone, particularly in the 60 cm thick plots, this is still an indicator of the pH of the pore water available to the plants.

The EC is less of a concern than the pH for the plant survival. Bearberry EC limits have been reported as $\langle 730 \mu S/cm$, much higher than the values $\langle 200 \mu S/cm$ reported in the Technosol profiles (Chapter 3), and also higher than most of the tension water samples from the plots, which were typically 500 µS/cm or lower (Chapter 2).

Ryegrass survived only one month before turning red and dying off. Like bearberry, this could reflect a lack of moisture, nutrients, or problems with other soil chemistry variables.

Nutrients

The macronutrient concentrations within the plant leaves suggested that only P could be potentially limiting, as K, Ca, and Mg concentrations in plants from Technosol plots were generally consistent with comparison vegetation samples analyzed during this study. Phosphorus deficiencies are typically associated with red or purple colours developing on plant stems and leaves (Barker & Pilbeam, 2007). Stunting, resulting in small, dark green leaves and short, slender stems, and chlorosis have also been observed (Barker & Pilbeam, 2007). Both the general appearance of the alder and the levels of P measured in leaves indicate that P is likely not

limiting alder growth. However, the bearberries did redden and very little growth was seen over the 2016 growing season, suggestive of a P deficiency. The ryegrass also turned red and died about a month after its germination, which was also suggestive of a P deficiency.

Data on P requirements for bearberry is extremely limited; other than the general statement that the species nutrient requirements are low. The closest related species in many cases was high bush blueberry (*Vaccinium corymbosum* L.), with documented sufficiency ranges of 0.10 – 0.32% dry weight (Barker & Pilbeam, 2007) or 0.20 – 0.50% (Jones Jr., Wolf, & Mills, 1991). Blueberry is also a member of Ericaceae and grows on dry, sandy, nutrient-poor soils with a similar documented pH tolerance (Calvo-Polanco et al., 2017). If we accept blueberry as a proxy for bearberry requirements, the Technosol bearberry leaves fell into or just below the low end of the blueberry sufficiency ranges discussed above. Overall, with only three samples having foliar P concentrations noticeably lower than the comparison plants, a P deficiency was probably not the prime factor which caused the bearberry die-off.

Micronutrient concentrations indicated no deficiency issues in the alders, with ranges similar to either the reference source site or nursery alders. This observation indicates that the Technosols were likely able to meet the nutritional requirements of the alders and the species is a good choice for reclamation with the Technosols. The generally healthy appearance and growth of the alder support this assertion.

Micronutrient deficiencies were also not likely to be affecting the bearberry, as B, Cu, Mn, and Ni foliar concentrations were similar for both the Technosol bearberry and the comparison bearberry. Of note, Fe and Zn were both higher in the Technosol bearberry than in the reference bearberry by at least 100 mg/kg.

In high bush blueberry, elevated levels of Fe are > 200 mg/kg (Jones Jr. et al., 1991). The bearberry samples from the Technosols ranged from about 75 mg/kg in the high organic Technosols to 200 mg/kg in the low organic Technosols, falling just below the blueberry range. Iron toxicity is typically only seen in flooded conditions when Fe^{3+} is reduced to Fe^{2+} (Nagajyoti et al., 2010). Iron can accumulate in plants to several hundred mg/kg without toxicity occurring (Jones Jr. et al., 1991), indicating Fe levels are an unlikely cause of the mortality observed.

In blueberry, Zn is variously reported as being high when > 100 mg/kg (Jones Jr. et al., 1991), or with > 80 mg/kg causing toxicity symptoms (Barker & Pilbeam, 2007). Levels of Zn in the range of 100 – 140 mg/kg were reported in the Technosol plot bearberry, higher than both reported limits for the blueberry. Zinc toxicity can limit the growth of roots and shoots as well as cause chlorosis (Nagajyoti et al., 2010; Rout & Das, 2009). Chlorosis is sometimes caused by a Zn-induced Fe deficiency (Nagajyoti et al., 2010) but Fe concentrations in young chlorotic leaves have also been measured at much higher levels than usually required, over 100 mg/kg (Foy, Chaney, & White, 1978; Rout & Das, 2009). Zinc can inhibit nutrient transfer within the plant, causing Cu and Mn deficiencies in leaves (Nagajyoti et al., 2010) and accumulation of Zn, Fe, Mg, K, P, and Ca in roots (Rout & Das, 2009). A purplish-red colour in leaves is also reported as a possible symptom of Zn toxicity (Nagajyoti et al., 2010), as is Zn-induced P deficiency (Foy et al., 1978).

In the Technosol bearberry leaf samples all nutrients appeared to be present in sufficient concentrations to support healthy growth, with the possible exception of P, which does not support the theory of Zn toxicity. While the high Fe levels and reddening of leaves were partially consistent with Zn toxicity, increases in Fe could just as easily have been caused by higher bioavailability of Fe in the soil allowing more uptake. The reddening of bearberry leaves could

have been due to low P levels or an early senescence caused by other factors such as low moisture and high pH. Thus the suggestion that Zn toxicity was a problem for the bearberries cannot be confirmed or refuted without more research data.

It should be noted that nitrogen content in the leaves was not measured as part of this analysis; therefore it is possible that the health of the bearberries was adversely affected by a nitrogen deficiency. However, bearberry normally thrive in N-poor sandy soils of Boreal regions.

Elements of environmental interest

Molybdenum is the primary element of concern on the site, being present throughout the deposit (Pan & Fleet, 1995). Mo is also an essential micronutrient because of its crucial role in nitrate reduction and in symbiotic nitrogen fixation (Barker & Pilbeam, 2007). Molybdenum a cofactor in two proteins found in nearly all nitrogeness, enzymes responsible for N_2 reduction (Barker & Pilbeam, 2007). Molybdenum levels are thus particularly important for alders, which form a symbiotic relationship with the actinobacteria *Frankia* and as a result have additional Mo requirements to sustain the nitrogenase enzyme within the bacteria (Bélanger, Bellenger, & Roy, 2013). This symbiosis can provide a significant advantage to alders when N is limiting, as *Frankia* can meet up to 90% of the N demand of the plant (Bélanger et al., 2013).

Our data revealed that Mo concentrations in alder were higher than in the bearberry grown on the same treatment. Molybdenum was detected in all alder samples but was only detected in bearberry on two of the low organic Technosol plots. Both of these samples had Mo concentrations of 3.3 mg/kg, which is slightly higher than the commonly found value of $0.1 - 2$ mg/kg (Oustriere et al., 2016). The higher concentrations in alder, averaging 18 mg/kg in leaves, are consistent with their increased need for the element but still less than reported for stressed plants (Oustriere et al., 2016). The alder root sample contained more Mo than the leaves (60

mg/kg); alder roots have been found to contain more Mo than shoots because the nodules where N-fixation occurs act as a Mo sink (Bélanger et al., 2013). These concentrations were consistent with the comparison alders and are likely quite safe for the plants, particularly as Mo has a relatively low toxicity for plants (Bélanger et al., 2013).

None of the additional elements of environmental concern (As, Cd, and Sb), having low bioavailability, appeared to be present in the vegetation in sufficient quantities to decrease growth. All three elements were below the detection limit in bearberry, and though all were found in small quantities in the alder root, only As was detectable in the leaves of alders from the Technosol plots. This indicates that little uptake of these elements is occurring. The amount of bioavailable Cd and Sb in the Technosols was low. Cadmium was detected in only two 40% organic plots and Sb was below detection limit in all (Chapter 2), so the low concentrations in the vegetation were unsurprising.

Arsenic was more bioavailable, a potential concern as the arsenate ion, $AsO₄³$, is an analog for phosphate and can therefore be taken up in its place (Nagajyoti et al., 2010). Uncontaminated terrestrial plants typically report $0.2 - 0.4$ mg/kg of arsenic (Cullen & Reimer, 1989). The alder leaves on the high organic Technosol had As concentrations within this range and alders on low organic Technosols were slightly higher, with a maximum of 0.7 mg/kg observed. Similar results have been seen in *Alnus incana* leaves from As-contaminated soils; leaf As concentrations were measured at 0.4 mg/kg (Kuehnelt, Lintschinger, & Goessler, 2000). This suggests the alders do not accumulate large amounts of As, and that the As that is contained within the leaves is not present in high enough concentrations to harm the plants.
None of the elements of environmental interest are likely to be harmful to vegetation growth on the Technosols, and none of the four appear responsible for the lack of success with bearberry or ryegrass grown on the Technosol plots.

Conclusion

Green alders appeared to thrive on the Technosol plots, particularly on the high organic plots which provided more moisture and higher levels of bioavailable nutrients. Low rates of mortality and large amounts of growth were observed on many of the test plots, indicating that green alders are a good choice of species for initial revegetation on the Technosols.

Bearberries were not successful on the Technosols, struggling throughout the growing season. A combination of factors, including low moisture in upper soil layers, less extensive root systems, high soil pH, low phosphorus, and possibly zinc toxicity, probably contributed to their decline. If bearberry were to be used on these Technosols, amendments would need to be added to increase survival rates, or local varieties tolerant of neutral to alkaline pH must be selected. Otherwise an alternative species needs to be selected for revegetation to be successful on the Technosols.

Chapter 5. Summary, Implications, and Recommendations

Summary and Implications

This study was designed to test whether a Technosol constructed from local waste products, specifically mine rock and lumber mill woody residuals, is an effective medium for use in site reclamation. The study was located at Barrick Gold's Hemlo Operations, which are just north of Lake Superior and consist of an underground and open pit mine. Though the original soil material removed when operations began was stockpiled and saved, the mine rock pile generated by the open pit is so large that alternative material will be needed to complete reclamation and revegetation. Using local waste products such as the mine rock and woody residuals reduces operational costs and the carbon footprint associated with transporting large amounts of alternative materials from long distances.

Due to the varied nature of Technosols formed either intentionally or not from mine wastes, studying the development of one specifically designed for reclamation purposes was one of the main objectives of this study, as was assessing the elemental content of water samples released from the Technosols and flowing over mine rock. Their ability to support native vegetation was also critical. The Technosols were manufactured and placed into plots in summer 2012, providing four years to develop *in situ* as the study continued. Two Technosols were created, one with 40% woody residuals and one with 80% woody residuals, to examine the effects of higher organic content on the physical and chemical properties of the Technosol and its ability to support vegetation. Finally, two depths of Technosol application (30 cm and 60 cm) were compared to see whether a thicker layer of soil would provide a better environment for plant growth.

The first thing examined was the initial soil chemistry and the water chemistry over the four years since the plots were constructed (Chapter 2). The higher organic Technosol was found

129

to have significantly more C and N, as well as Ca, Mg, K, P, and Mn, available for plant uptake. Of interest, the high organic Technosol also had significantly lower amounts of bioavailable Mo. Water samples from all Technosols, particularly the high organic Technosols, had high concentrations of DOC, much higher than concentrations in most natural waters in the region, throughout the four years they were measured. The pH of both pore and through-flow water samples from both Technosols was $7 - 8$, with the alkalinity attributed to the presence of carbonates within the mine rock. Electrical conductivity was lower in the soil pore water than in the through-flow samples, with chemical analysis these water samples indicating this conductivity may be due to SO_4^2 ⁻, Ca^{2+} , K⁺, and Mg²⁺ concentrations.

There are several elements of environmental interest on the Hemlo site, namely Mo, As, Cd, and Sb. Analysis of water samples indicated that Mo and Cd exceeded PWQO on occasion, though Cd did not exceed by a large amount. Antimony concentrations were never above PWQO, and As concentrations were below the PWQO but above the interim PWQO in many pore water samples, though not in through-flow samples. This means that ongoing monitoring of the through-flow from the Technosols, particularly for Mo and Cd, will likely be required.

The development of the Technosol profiles and the variations in their properties with depth was examined in Chapter 3. Little profile development, in terms of variations in colour, pH, EC, and structure, appears to have occurred in the four years the Technosols have been in place. Vegetated plots were found to have a more granular structure than non-vegetated ones. When soil microclimates were compared, vegetated plots were found to have higher levels of moisture in the upper layers of soil, likely due to root-induced structure and porosity development increasing infiltration.

The pH of around 8 in the high organic Technosol profiles and 9.5 in the low organic profiles, was much higher than the natural soils of the area and may impact successful establishment of some vegetation species. The EC of the profiles was low throughout, under 150 µS/cm, likely as a result of the high percentage of organics within the Technosols.

In terms of the soil microclimate, the high organic Technosols had less extreme temperatures in both summer and winter, though the surface layers of soil still reached above 20 °C in summer and below -10 °C in winter. There was less variation in both temperature and moisture the lower layers of the Technosols, particularly at 60 cm depth, with moisture levels at lower depths always higher overall than the upper layers. The high organic Technosols had more moisture in the upper 30 cm of soil, but less at 60 cm, possibly due to the increased retention and slow percolation through the upper layers. Occasionally the surface 5 cm of soil fell below the permanent wilting point of the Technosols, so for plants to survive dry periods deeper root systems would be required.

A basic comparison of the microbial functional diversity of the Technosols to the comparison forest and successional field soils revealed differences between the communities do exist. Using EcoPlates™, richness and diversity were compared. The forest soil had significantly lower richness than the Technosol profiles, and exhibited lower average well colour development, a measure of substrate usage. Substrates common in bacterial metabolisms were more actively utilized and displayed a more steady colour increase than others, indicating that the community may have contained more bacteria than fungi or other microorganisms.

The survival and health of vegetation on the Technosols was the last area of study (Chapter 4). When green alders and bearberries were planted on the Technosols in 2015 after being soaked and were then watered once, survival rates in 2016 were high, unlike the low

131

survival following bare-root transplants in 2013. Alders continued to thrive on the Technosols through 2016; the vibrant green colour and nodulation observed during the excavation of an alder root system indicate that nitrogen fixation is occurring. Alders on the low organic, 60 cm thick plots were significantly smaller than alders on any other plots, probably due to increased exposure and lower moisture in these plots.

The bearberries did not survive as well as the alders; despite an initial low mortality, almost complete mortality was observed in 2017. Throughout 2016 bearberries showed signs of distress, with red and yellow leaves appearing in early July and very little evidence of extension measured. No differences in size or health were seen between the low and high organic Technosols, or the thick and thin plots. An examination of the nutrient and trace elements of interest in the vegetation leaves revealed that, while alders appear to have sufficiency in all circumstances, bearberries could potentially be experiencing either a P deficiency or Zn toxicity, or both. Nitrogen deficiency is another possibility which should be investigated, as are a lack of moisture in the upper soil layers where the bearberry roots are and high soil pH.

The overall conclusions of this study are that the Technosols can be used to reclaim the mine rock pile. The high organic Technosol is recommended, due to higher nutrient content, reduced availability of elements of environmental interest such as Mo and Cd, less extreme soil temperatures, increased soil moisture, and reduced soil pH. Green alders are an ideal species for use on the Technosol and appeared to be thriving several years after planting. However, bearberries should not be used unless amendments are added to make the Technosol more suitable for their growth or local bearberry varieties are used. Finally, monitoring of water percolating through the Technosol will be needed to ensure concentrations of elements such as Mo and Cd do not exceed regulatory limits.

132

Recommendations for future work

The high pH of the soil is one of the main concerns for vegetation survival, but at the same time it is a good sign for the mining company in terms of the acid generation potential of the Technosol on the mine rock pile. Acidity will not be a major concern when the Technosols are in place. As a result, vegetation species with a broad range in pH tolerance will have to be chosen for revegetation unless some amendment such as sulphur prills or crushed acid generating mine rock is added to lower the pH. This may limit the species available, as most boreal soils are acidic and therefore most vegetation may be adapted for lower pH conditions. Species like green alder, which gradually acidify soils, may slowly change surface soil conditions and provide habitats for these native species as their influence on the soil increases.

The low nutrient content of the soil is another potential issue for vegetation growth, as it is a possible factor in the failure of the bearberries. While P content was measured in plant leaves and found to be a potential limiting nutrient, N in leaves was not measured. In future studies, an examination of the N content of the leaves of vegetation such as bearberries on the Technosol will be critical for determining whether N or P are limiting nutrients.

The shallower observed root systems of the bearberries preventing access to the higher moisture held deeper in the soil is another potential problem for growth. The planting with their potting soil intact may have slowed their root expansion into the less hospitable environment of the Technosols, limiting their potential for moisture uptake. Bearberries transplanted without the potting soil buffer could be monitored to learn which did better or worse in the Technosol. The performance of locally sourced bearberry should be investigated as they may grow better than nursery stock, being already adapted to the climate of the region. Though bearberry can be difficult to germinate, the use of local cuttings may also have more successful growth if such cuttings were obtained.

The monitoring of the microclimate on the surface of the plots should be more precisely measured to see what the bare soil conditions are so that the impacts of vegetation establishment can be described. Although we have soil temperature and moisture throughout the plot, a more in-depth examination of the surface and the upper 20 cm where the majority of rooting occurs would be interesting. Samples of soil pore water from 10 cm rather than from 30 or 60 cm, as is currently obtained, would give a better idea of the quantity and content of the water and nutrients the plants are most exposed to. Plot surface temperatures and wind conditions would give more information on the amount of exposure the plants are experiencing, and how that is changing as the alders increase in size. Further studies on the infiltration rates and bulk density of the soil would give insight into the porosity and water storage of in the Technosols. Measurements of transpiration rates would also be valuable, as they would provide more information about the water cycling and requirements on the plot, particularly as plant size and number increases.

The completion of the soil profile chemistry, including how nutrient and trace element concentrations and bioavailability change with depth, will give further insight into how the Technosols have changed over the four years since their creation and whether the nutrients plants require are present in the areas of the soil where they are able to access them.

Finally, looking at other vegetation species on the Technosol will be beneficial and critical. Though bearberry and ryegrass did not succeed, other species such as field strawberry have been observed colonizing the Technosol materials with some success. Trials with seeding tickle grass in 2013 were unsuccessful (Watkinson, 2014) but these occurred when a hardened crust was observed on the surface of the plots, something no longer present by 2015 when the Technosols had matured more. Tickle grass would now possibly be able to germinate and grow on the plots.

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Appendix A: Supplemental Figures for Chapter 2

Soil chemistry

Soil organics

		Air dry			Bone dry			
Plot ^a	$C\%$	$N\%$	$S\%$	$C\%$	$N\%$	$S\%$	C/N	C/S
1A	3.77	0.038	0.237	3.80	0.05	0.24	75.4	15.9
1B	3.88	0.054	0.534	3.90	0.07	0.53	55.4	7.3
1 ^C	5.19	0.046	0.356	5.20	0.06	0.36	86.5	14.6
Mean	4.28	0.046	0.376	4.30	0.06	0.377	72.4	12.6
$\pm SD$	± 0.79	± 0.008	± 0.149	± 0.78	± 0.01	± 0.146	± 15.8	± 4.6
2A	14.35	0.131	0.360	14.4	0.17	0.36	84.4	39.9
2B	18.42	0.169	0.685	18.5	0.22	0.69	83.7	26.9
2C	13.92	0.123	0.439	14.0	0.16	0.44	87.0	31.7
Mean	15.56	0.141	0.495	15.6	0.18	0.50	85.0	32.8
$\pm SD$	± 2.48	± 0.025	± 0.170	± 2.5	± 0.03	± 0.17	± 1.7	± 6.6
3A	4.26	0.046	0.221	4.30	0.06	0.22	71.0	19.3
3B	4.86	0.031	0.516	4.90	0.04	0.52	121.5	9.4
3C	4.19	0.031	0.393	4.20	0.04	0.39	104.6	10.7
Mean	4.44	0.036	0.377	4.47	0.05	0.38	99.0	13.1
$\pm SD$	± 0.37	± 0.009	± 0.149	± 0.38	± 0.01	± 0.15	± 25.7	± 5.4
4A	17.20	0.138	0.361	17.4	0.18	0.36	95.6	47.6
4B	15.21	0.108	0.426	15.3	0.14	0.43	108.7	35.7
4C	13.31	0.100	0.526	13.4	0.13	0.53	102.4	25.3
Mean	15.24	0.115	0.438	15.4	0.15	0.44	102.2	36.2
$\pm SD$	±1.95	± 0.020	± 0.083	± 2.0	± 0.03	± 0.09	± 6.6	± 11.2

Table A7. CNS results (% w/w), C/N, and C/S ratios for all Technosol soil samples (air and bone dry). Ratios calculated from bone dry samples.

 a 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm; letters indicate replicates

Total and bioavailable ions

		Table A8. Total soil macro and micronutrient concentrations in all Technosol samples.									
Plot ^a	B	Ca	Cu	Fe	$\bf K$	Mg	Mn	Mo	Ni	\mathbf{P}	Zn
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
1A	19.8	30800	30.6	17800	17200	14500	353	53.1	19.3	427	106
1B	26.3	33500	31.0	21100	15900	12900	403	47.6	21.6	482	100
1 ^C	23.2	37900	25.7	25500	16000	11800	455	84.0	26.9	566	107
Mean	23.1	34066.7	29.1	21466.7	16367.7	13066.7	403.7	61.6	22.6	491.7	104.3
$\pm SD$	± 3.3	± 3583.8	± 3.0	± 3863.1	± 723.4	± 1357.7	± 51.0	± 19.6	± 3.9	±70.0	± 3.8
2A	40.7	29900	45.8	20500	16300	12900	388	50.8	19.3	432	113
2B	35.6	30200	21.8	16500	16200	12600	406	45.2	17.3	494	164
2C	21.2	31300	19.0	19700	14900	11300	436	45.9	16.5	486	105
Mean	32.5	30466.7	28.9	18900.0	15800.0	12266.7	410.0	47.3	17.7	470.7	127.3
$\pm SD$	± 10.1	± 737.1	± 14.7	± 2116.6	± 781.0	± 850.5	± 24.2	± 3.1	±1.4	± 33.7	\pm 32.0
3A	24.3	32600	19.4	19700	16600	12800	378	51.8	18.7	464	112
3B	25.6	35400	24.6	21600	16900	11600	420	45.6	25.1	495	98.2
3C	32.2	32600	26.0	22700	17300	11800	436	56.5	23.6	487	105
Mean	27.4	33533.3	23.3	21333.3	16933.3	12066.7	411.3	51.3	22.5	482.0	105.1
$\pm SD$	± 4.2	± 1616.6	± 3.5	± 1517.7	± 351.2	± 642.9	± 30.0	± 5.5	± 3.3	± 16.1	± 6.9
4A	19.9	26900	19.4	16500	15400	11100	408	43.2	15.8	427	103
4B	19.7	27200	22.2	17100	14700	10400	355	33.5	17.0	433	98
4C	21.9	28000	19.2	19100	16500	10700	360	64.8	19.3	439	105
Mean	20.5	27366.7	20.3	17566.7	15533.3	10733.3	374.3	47.2	17.4	433.0	102.0
\pm SD	± 1.2	± 568.6	± 1.7	± 1361.4	± 907.4	± 351.2	± 29.3	±16.0	±1.8	± 6.0	± 3.6

Table A8. Total soil macro and micronutrient concentrations in all Technosol sample

 a_1 : 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm; letters indicate replicates

			Total son concentrations of cientents of chynomnemal is
Plot ^a	As	Cd	Sb
	mg/kg	mg/kg	mg/kg
1A	6.34	0.284	0.484
1B	5.80	0.291	0.385
1 ^C	6.64	0.308	0.426
Mean	6.26	0.294	0.432
\pm SD	± 0.43	± 0.012	± 0.050
2A	6.77	0.371	0.35
2B	4.38	0.533	0.368
2C	4.68	0.297	0.305
Mean	5.27	0.400	0.341
\pm SD	±1.30	± 0.121	± 0.032
3A	5.40	0.295	0.511
3B	6.04	0.290	0.363
3C	6.11	0.288	0.352
Mean	5.85	0.291	0.409
\pm SD	± 0.39	± 0.004	± 0.089
4A	6.53	0.334	0.333
4B	4.51	0.313	0.341
4C	5.35	0.320	0.296
Mean	5.46	0.322	0.323
\pm SD	± 1.01	± 0.011	± 0.024

Table A9. Total soil concentrations of elements of environmental interest in all Technosol samples.

 a 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm; letters indicate replicates

Plot ^b	As	$\, {\bf B}$	Ca	Cd	Cu	Fe	K	Mg	Mn	Mo	\mathbf{P}
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
DL	0.002	0.004	0.009	0.0003	0.001	0.001	0.002	0.0008	0.0004	0.0008	0.002
1A	0.288	0.339	126	\triangle DL	0.0524	0.243	43.1	18.2	\triangle DL	0.2	0.195
1B	0.0995	0.0224	121	0.00223	0.00413	0.12	45.1	16.9	$<\!\!DL$	0.161	0.376
1 ^C	$\langle DL$	\triangle DL	137	\triangle DL	0.122	0.725	49.2	21.4	0.759	0.187	\triangle DL
Mean ^c $\pm SD$	0.194 ± 0.13	0.181 ± 0.22	128.0 \pm 8.2	0.00223	0.0595 ± 0.06	0.363 ± 0.32	45.8 ± 3.1	18.83 ± 2.3	0.759	0.183 ± 0.02	0.286 ± 0.13
2A	0.0466	0.211	167	\triangle DL	0.00621	0.0726	55.6	23.2	\bigtriangleup DL	0.0825	1.08
2B	\triangle DL	0.0523	204	\triangle DL	0.0335	0.297	57.2	28.6	1.75	0.067	2.01
2C	0.0925	\triangle DL	174	\triangle DL	\triangle DL	0.476	58.8	26.9	1.47	0.1	0.545
Mean ^c	0.070	0.132	181.7		0.0199	0.282	57.2	26.23	1.61	0.083	1.212
$\pm SD$	± 0.03	± 0.11	± 19.7	\triangle DL	± 0.02	± 0.20	± 1.6	± 2.8	± 0.2	± 0.02	± 0.74
3A	$<$ DL	0.213	124	\triangle DL	$<\!\!DL$	0.315	51.3	15.1	0.412	0.267	0.0831
3B	$<$ DL	$<\!\!DL$	118	0.00323	0.00519	0.391	51.5	15.4	0.412	0.193	$\langle DL$
3C	0.119	$\langle DL$	151	\triangle DL	0.0279	0.49	51.5	19.4	0.708	0.163	\triangle DL
Mean ^c $\pm SD$	0.119	0.213	131.0 ± 17.6	0.00323	0.0165 ± 0.02	0.399 ± 0.09	51.4 ± 0.1	16.63 ± 2.4	0.51 ± 0.2	0.208 ± 0.05	0.0831
4A	0.141	0.0422	191	\triangle DL	\triangle DL	0.13	54.8	25.2	\triangle DL	0.0354	1.43
4B	0.0535	0.0354	201	\triangle DL	$<\!\!DL$	0.433	61.5	35	$<\!\!DL$	0.0648	1.85
4C	$<$ DL	$\langle DL$	195	\triangle DL	0.0177	0.145	63.7	24	$<\!\!DL$	0.107	1.04
Mean ^c $\pm SD$	0.097 ± 0.06	0.039 ± 0.004	195.7 ± 5.0	\triangle DL	0.0177	0.236 ± 0.17	60.0 ± 4.6	28.07 ± 6.0	\triangle DL	0.069 ± 0.04	1.440 ± 0.41

Table A10. Concentrations of bioavailable soil nutrients and elements of environmental concern in all plots^a

^a All Ni, Sb, and Zn concentrations below detection limits (0.0003 mg/L, 0.018 mg/L, and 0.001 mg/L respectively)

 b 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm; letters indicate replicates

^c Means and SD were calculated using only samples above detection limits

Table A11. Results of bioavailable ion t-tests comparing 40% and 80% organic Technosols

racio Tri Propano of croavamacio fon è todo comparing 10% and 00% organic recimentos										
					Fе		Μg	Mn	Mo	
a. t.					10					
	-1.518	-1.159	7.469	-0.783	-1.104	4.855	4.658	6.316	-6.428	3.330
	0.189	0.299	2.14 e^{-5} *	0.464	0.296	6.66 e^{-4} *	8.98 e^{-4} *	$0.003*$	9.53 e^{-5} *	$0.013*$

* Significant (α = 0.05)

Table A12. Percentage of total element concentration which is bioavailable for low and high organic Technosols

Technosol	As		Ca	Cd	Cu	Fe	л	Mg	Mn	Mo	
Low org (40%)	2.79	0.76	0.38	0.94	0.16	0.0018	0.29	0.14	0.14	0.35	0.04
High org (80%)	1.55	0.32	0.65	$N\!/\!A$	$0.08\,$	0.0014	0.37	0.24	0.41	0.16	0.29

Water chemistry

Basic chemistry

Figure A37. Redox potential (rel mV) of Technosol gravity through-flow (GT) and tension water (TW), and Williams mine rock test cell (MR) water samples from Oct 2012 to Nov 2016 with loess curve and 95% CI.

Figure A39. Concentrations of $NO₃$ (mg/L) from Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 197 samples < DL (0.01 mg/L), 126 of which are TW samples.

Macronutrients

Figure A40. Concentrations (mg/L) of K in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 1 sample < DL (13.5 μ g/L).

Figure A41. Concentrations (μ g/L) of P in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 334 samples < DL (0.45 µg/L).

Micronutrients

Figure A43. Concentrations (μ g/L) of Mn in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 18 samples < DL (0.1 µg/L).

Figure A44. Concentrations (µg/L) of Ni in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 45 samples < DL (0.15 µg/L).

Figure A45. Concentrations (μ g/L) of Zn in Technosol gravity through-flow (GT) and tension water (TW), Williams mine rock test cell water (MR), natural water (NW), and rainwater (RW) samples from July 2013 to Nov 2016. 7 samples < DL $(0.2 \mu g/L)$.

Elements of environmental interest

Figure 46. Cd concentrations (µg/L) of a) all samples including Williams mine rock test cell water (MR) and b) Technosol gravity through-flow (GT), Technosol tension water (TW), natural water (NW), and rainwater (RW) from July 2013 to Nov 2016. 58 samples < DL (0.03 μ g/L).

UIUIIUII U Element	2013	2014	2015	2016	Total <dl< th=""></dl<>
$(DL, \mu g/L)$	$(158$ obs.)	$(106$ obs.)	$(83$ obs.)	$(232$ obs.)	$(579$ obs.)
As	10	$\overline{4}$	$\overline{7}$	43	64
(0.35)	6.3 %	3.8 %	8.4 %	18.5 %	11.1 %
\mathbf{B}	τ	18	6	10	41
(0.06)	4.4 %	$17.0\ \%$	7.2 %	4.3 %	7.1%
Ca	$\mathbf{1}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\mathbf{1}$
(20)	$0.6\:\%$				0.2 %
Cd	15	11	5	27	58
(0.03)	9.5%	10.4 %	$6.0\ \%$	$11.6\,\%$	10.0%
Cu	$\boldsymbol{0}$	11	$\boldsymbol{0}$	9	20
(0.01)		10.4 %	\equiv	3.9%	3.5 %
Fe	$\boldsymbol{0}$	60	28	67	155
(I)		56.6%	33.7 %	28.9%	26.8%
$\bf K$	$\mathbf{1}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\mathbf{1}$
(13.5)	$0.6\,\%$				0.2 %
Mg	$\mathbf{1}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\mathbf{1}$
(1)	$0.6\,\%$				$0.2~\%$
Mn	$\mathbf{1}$	$\boldsymbol{0}$	13	$\overline{4}$	18
(0.1)	0.6%		15.7 %	1.7 %	3.1 %
Mo	6	$\boldsymbol{0}$	$\mathbf{1}$	$\mathbf{1}$	8
(0.13)	3.8 %		1.2 %	$0.4~\%$	1.4 %
Ni	$\boldsymbol{0}$	34	$\mathbf{1}$	$10\,$	45
(0.15)		32.1 %	1.2 %	4.3 %	7.8 %
\mathbf{P}	92	86	33	123	334
(0.45)	58.2 %	$81.1\ \%$	39.8%	53.0%	57.7 %
Sb	$\mathbf{1}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\mathbf{1}$
(0.01)	$0.6\,\%$				$0.2~\%$
Zn	τ	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$	τ
(0.01)	4.4 %				1.2 %

Table A13. Number and % of water samples with concentrations below detection limit for each measured element by year. -

Appendix B: Supplemental figures for Chapter 3

Profile chemistry

Figure B47. pH of 40% organic Technosol profiles with and without vegetation.

Figure B48. pH of 80% organic Technosol profiles with and without vegetation.

Figure B49. pH of successional field Technosol and forest Podzol reference sites.

Figure B50. Mean daily soil temperature in 40% organic, 30 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3).

Figure B51. Mean daily soil temperature in 80% organic, 30 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3).

Figure B52. Mean daily soil temperature in 40% organic, 60 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3).

Figure B53. Mean daily soil temperature at 5 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil Aug 1, 2014 – Nov 4, 2016. Air temperature at 2 m is also shown.

Figure B54. Mean daily soil temperature at 30 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil Aug 1, 2014 – Nov 4, 2016. Air temperature at 2 m is also shown.
Soil moisture

Figure B55. Mean daily volumetric water content in 40% organic, 30 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3) with total daily precipitation. Data from winter months (Dec 1 – May 1) excluded.

Figure B56. Mean daily volumetric water content in 80% organic, 30 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3) with total daily precipitation. Data from winter months (Dec 1 – May 1) excluded.

Figure 57. Mean daily volumetric water content in 80% organic, 60 cm deep Technosol plots from Sept 20, 2012 to Nov 4, 2016 (n = 3) with total daily precipitation. Data from winter months (Dec $1 - May 1$) excluded.

Figure B58. Mean daily volumetric water content at 10 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil with total daily precipitation from Aug 1, 2014 – Nov 4, 2016. Data from winter months (Dec 1 – May 1) excluded.

Figure B59. Mean daily volumetric water content at 30 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil with total daily precipitation from Aug 1, 2014 – Nov 4, 2016. Data from winter months (Dec 1 – May 1) excluded.

Figure B60. Mean daily volumetric water content at 60 cm depth in 40% and 80% organic 60 cm plots (n = 3), successional field, and forest soil with total daily precipitation from Aug 1, 2014 – Nov 4, 2016. Data from winter months (Dec 1 – May 1) excluded.

Microbial functional diversity

Figure B61. Average well colour development (AWCD) at 590 nm of 80% organic Technosols (no vegetation and vegetated), 40% organic Technosol, and reference forest profiles over 96h period after inoculation.

		F	df	
AWCD	Profile	3.648	5,62	0.00588
	Depth	0.856	16, 62	0.620
R	Profile	5.333	5, 62	0.000386
	Depth	0.503	16, 62	0.936
H	Profile	2.658	5, 62	0.0306
	Depth	0.629	16, 62	0.847
E	Profile	3.178	5, 62	0.0129
	Depth	0.570	16, 62	0.894

Table B14. Summary of two-way ANOVA results comparing average well colour development (AWCD), richness (*R*), Shannon-Weaver Index (*H*), and evenness (*E*) by profiles and minimum sample depth.

Figure B62. Average well colour development (AWCD) of soil profiles at 48h post-inoculation; letters indicate significant differences as determined by Tukey HSD. Profiles are 3A: low organic, unvegetated; 3C: low organic, vegetated; 4B: high organic, unvegetated; 4C: high organic, vegetated; FI: field; FO: forest.

Figure B63. Shannon-Weaver Index (*H*) of soil profiles at 48h calculated from substrates $OD_{590} > 0.1$; letters indicate significant differences as determined by Tukey HSD. Profiles are 3A: low organic, unvegetated; 3C: low organic, vegetated; 4B: high organic, unvegetated; 4C: high organic, vegetated; FI: field; FO: forest.

Figure B64. Evenness (*E*) of soil profiles at 48h calculated with substrates $OD_{590} > 0.1$; letters indicate significant differences as determined by Tukey HSD. Profiles are 3A: low organic, unvegetated; 3C: low organic, vegetated; 4B: high organic, unvegetated; 4C: high organic, vegetated; FI: field; FO: forest.

Appendix C: Supplemental Figures for Chapter 4

Mortality

Figure C65. Annual and perennial ryegrass blades upon Technosol plots in (top) June 2016 and (bottom) July 2016.

Health

Table C15. Repeated measure ANOVA results comparing green alders in each leaf coverage category.

Alder leaf coverage	Effect	d. f. num.	d.f. denom.	\boldsymbol{F}	\boldsymbol{p}
Full	Organic content		4	4.819	0.093
	Technosol depth	1	$\overline{4}$	2.222	0.210
	Month	3	12	7.682	$0.004 *$
	Org: Depth	1	$\overline{4}$	0.540	0.503
	Org: Month	3	12	0.545	0.661
	Depth: Month	3	12	0.146	0.930
	Org: Depth: Month	3	12	0.187	0.903
Partial	Organic content	1	4	18.39	$0.013 *$
	Technosol depth	1	4	2.235	0.209
	Month	3	12	5.953	$0.010 *$
	Org: Depth	1	$\overline{4}$	3.446	0.137
	Org: Month	3	12	0.573	0.644
	Depth: Month	3	12	0.061	0.980
	Org: Depth: Month	3	12	0.061	0.980
None	Organic content	$\mathbf{1}$	4	0.005	0.949
	Technosol depth	1	$\overline{4}$	1.056	0.362
	Month	3	12	1.571	0.248
	Org: Depth	1	$\overline{4}$	0.117	0.749
	Org: Month	\mathfrak{Z}	12	0.429	0.736
	Depth: Month	3	12	0.429	0.736
	Org: Depth: Month	3	12	1.571	0.248

* Significant (α = 0.05)

Bearberry colour	Effect	d. f. num.	$d. f.$ denom.	\boldsymbol{F}	\boldsymbol{p}
Green	Organic content	1	4	0.005	0.947
	Technosol depth	1	$\overline{4}$	0.005	0.947
	Month	4	16	29.21	3.56 e^{-7} *
	Org: Depth	1	$\overline{4}$	0.234	0.654
	Org: Month	4	16	0.452	0.770
	Depth: Month	4	16	1.334	0.300
	Org: Depth: Month	4	16	0.204	0.933
Green	Organic content	1	$\overline{4}$	0.163	0.707
mixed	Technosol depth	1	$\overline{4}$	$9.47 e^{-4}$	0.977
	Month	4	16	15.74	$2.10 e^{-5}$ *
	Org: Depth	1	$\overline{4}$	0.111	0.756
	Org: Month	4	16	1.376	0.286
	Depth: Month	4	16	0.779	0.555
	Org: Depth: Month	4	16	0.304	0.871
Red/Black	Organic content	1	$\overline{4}$	0.145	0.723
	Technosol depth	1	$\overline{4}$	0.005	0.948
	Month	4	16	7.298	$0.002 *$
	Org: Depth	1	$\overline{4}$	1.278	0.322
	Org: Month	4	16	1.894	0.161
	Depth: Month	4	16	0.525	0.719
	Org: Depth: Month	4	16	0.934	0.469

Table C16. Repeated measure ANOVA results comparing bearberries in each health colour category.

* Significant (α = 0.05)

Nutrients and trace elements

Macronutrients

Sample ^a	% dry weight									
	Ca	K	Mg	P						
Alder 1A	1.37	1.12	0.113	0.0958						
Alder 1C	1.24	1.04	0.147	0.107						
Alder 2B	1.37	0.923	0.156	0.121						
Alder _{2C}	1.03	1.06	0.136	0.14						
Alder 3B	1.21	1.17	0.14	0.105						
Alder 3C	1.09	1.06	0.121	0.1						
Alder _{4A}	1.07	1.09	0.117	0.166						
Alder _{4C}	1.28	0.931	0.156	0.134						
Alder Root	1.93	0.775	0.791	0.103						
Nursery Alder 1	1.69	0.912	0.424	0.114						
Nursery Alder 2	1.78	1.09	0.409	0.135						
Alder Source 1	1.31	1.05	0.132	0.173						
Alder Source 2	1.5	1.03	0.136	0.165						
Alder Source 3	1.16	0.917	0.148	0.176						
Alder Source 4	1.21	0.988	0.12	0.182						
Bearberry 1A	1.22	0.587	0.22	0.127						
Bearberry 1C	0.893	0.449	0.202	0.0916						
Bearberry 2B	0.976	0.525	0.211	0.11						
Bearberry 2C	1.08	0.444	0.218	0.102						
Bearberry 3B	1.09	0.516	0.211	0.105						
Bearberry 3C	0.887	0.411	0.173	0.0902						
Bearberry 4A	1.07	0.442	0.203	0.0913						
Bearberry 4C	0.954	0.537	0.23	0.169						
Bearberry Comp 1	0.892	0.471	0.134	0.133						
Bearberry Comp 2	0.876	0.528	0.116	0.112						

Table C17. % dry weight of macronutrients in green alder and bearberry samples from each vegetated Technosol plot and comparison vegetation.

^a1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm; letters indicate replicates

Figure C66. Concentrations of B (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Figure C67. Concentrations of Cu in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Figure C68. Concentrations of Fe (mg/kg) in vegetation samples with alder root sample excluded (7540 mg/kg Fe). Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Figure C69. Concentrations of Mn (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Figure C70. Concentrations of Ni (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).1 alder Technosol sample < DL (0.2 mg/kg).

Figure C71. Concentrations of Zn (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Elements of environmental interest

Figure C72. Concentrations of As (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Figure C73. Concentrations of Sb (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Figure C74. Concentrations of Cd (mg/kg) in vegetation samples. Source sites are Technosol plots (1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm) and comparison vegetation (AN: nursery alder, AR: alder root, AS: source alder; BC: comparison bearberry).

Source	As	\bf{B}	Ca	Cd	Cu	Fe	K	Mg	Mn	Mo	Ni	${\bf P}$	Sb	Zn
DL	0.2	1	5	0.05	0.05	\boldsymbol{l}	6	0.1	0.02	\mathfrak{Z}	0.2	\boldsymbol{l}	0.07	0.06
Alder														
1A	0.704	31.3	13700	$<$ DL	4.72	207	11200	1130	189	26.9	3.13	958	$<$ DL	52.5
1 _C	0.613	26.3	12400	$<$ DL	3.77	216	10400	1470	176	25.2	0.654	1070	$<\!\!DL$	39.2
2B	0.375	27.6	13700	$<$ DL	6.23	163	9230	1560	178	16.8	0.922	1210	$\langle DL$	82
2C	0.201	18.2	10300	$<$ DL	5.19	114	10600	1360	97.6	8.15	0.593	1400	$<$ DL	47.9
3B	0.415	34.7	12100	$<$ DL	5.06	385	11700	1400	159	31.5	0.621	1050	$<$ DL	55.5
3C	0.565	31.7	10900	$<$ DL	4.76	290	10600	1210	189	24.2	2.06	1000	$<$ DL	45.8
4A	$<$ DL	26.1	10700	$<$ DL	8.22	83.6	10900	1170	105	3.99	$<$ DL	1660	$<\!\!DL$	48.7
4C	0.244	17.8	12800	$<$ DL	5.31	202	9310	1560	88.1	8.23	0.533	1340	$<$ DL	61.2
Root	3.2	12.6	19300	0.402	18.1	7540	7750	7910	162	60.3	19.9	1030	0.368	91.1
Nursery 1	$<\!\!DL$	23.8	16900	$<$ DL	2.47	92.3	9120	4240	25.2	10.8	1.01	1140	$<$ DL	18.1
Nursery 2	$\langle DL$	25.7	17800	$<$ DL	2.37	83.2	10900	4090	23.8	13.2	1.57	1350	$<$ DL	20.3
Source 1	0.297	13.4	13100	0.0512	11	302	10500	1320	104	33.9	2.5	1730	$<$ DL	43.3
Source 2	0.259	11	15000	0.053	11	345	10300	1360	69.2	31.5	1.11	1650	$\langle DL$	45.2
Source 3	$<$ DL	4.87	11600	$<$ DL	9.1	195	9170	1480	44.1	21	1.04	1760	$\langle DL$	20.3
Source 4	\bigtriangleup DL	3.35	12100	$<$ DL	10.6	211	9880	1200	68.4	25.7	1.89	1820	$<$ DL	32.2
Bearberry														
1A	\bigtriangleup DL	15.2	12200	$<$ DL	2.4	200	5870	2200	27.8	3.3	0.8	1270	$\langle DL$	143
1 _C	\bigtriangleup DL	12.9	8930	$<$ DL	1.43	121	4490	2020	20.2	$\langle DL$	0.57	916	$<$ DL	131
2B	$\langle DL$	14.4	9760	$<$ DL	1.23	75.8	5250	2110	19.6	$\langle DL$	0.484	1100	$<$ DL	121
2C	$<$ DL	12.4	10800	$<$ DL	0.957	75.2	4440	2180	18.2	$\langle DL$	0.478	1020	$<$ DL	117
3B	$\langle DL$	16.8	10900	$<$ DL	1.72	184	5160	2110	23.6	3.24	0.905	1050	$<$ DL	120
3C	$\langle DL$	12.4	8870	$<$ DL	1.43	201	4110	1730	24.5	$\langle DL$	0.853	902	$<$ DL	123
4A	\bigtriangleup DL	11.8	10700	$<$ DL	1.19	87.1	4420	2030	26.1	\bigtriangleup DL	0.563	913	$<$ DL	118
4C	$<\!\!DL$	14.3	9540	$<$ DL	2.06	97.3	5370	2300	19.8	$\langle DL$	0.366	1690	$<$ DL	101
Comp. 1	\bigtriangleup DL	8.75	8920	$<$ DL	3.28	3.69	4710	1340	17.9	\bigtriangleup DL	0.477	1330	$<$ DL	37.8
Comp. 2	$<$ DL	9.71	8760	$<$ DL	2.65	17.9	5280	1160	14.8	\langle DL	0.466	1120	$<$ DL	24.5

Table C18. Concentrations (mg/kg) of nutrients and select trace elements in vegetation from Technosol plots and reference vegetation.

Technosol treatments are 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm; reference vegetation is Nursery alder, Source alder, and Comparison bearberry; letters (A, B, C) indicate replicates

Appendix D. Soil profile chemistry

Figure D75. % moisture of soil profile samples collected Sept 27th and 28th, 2017 from Technosol and comparison profiles.

Figure D76. The % organic matter in the fraction of soil < 2 mm as determined by LOI (650°C, < 12 h)

Figure D77. Concentrations of sulphate in Technosol and comparison soil profiles (Sept 2017).

Profile	Depth	As	$\mathbf B$	Ca	Cd	Cu	Fe	$\bf K$	Mg	Mn	Mo	Ni	${\bf P}$	Sb	Zn
	(cm from	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
DL	surface)	0.2	\boldsymbol{l}	5	0.05	0.05	1	6	0.1	0.02	\mathfrak{Z}	0.2	\boldsymbol{l}	0.07	0.06
	$0 - 2$	15.6	68.3	1650	1.23	100	37300	42900	753	549	109	45.5	1060	1.47	374
	$2 - 4$	17.8	64.5	3040	1.96	68.6	37800	49900	57	676	142	46.4	1170	1.45	492
	$4 - 6$	23.7	55.5	26400	1.46	102	65800	70800	1510	1090	153	78.1	1560	1.9	507
	$6 - 8$	15.7	76.6	3350	1.22	97.9	36400	45600	1370	589	197	49.4	867	1.72	327
	$8 - 10$	17.9	59.4	2940	1.23	90.5	40400	52500	89.7	732	151	51.2	1190	2.05	417
	$10 - 12$	21.1	52.3	5610	0.914	97.3	48900	58700	349	819	130	71.1	1170	1.46	438
40% organic	$12 - 14$	18.4	64.3	7500	1.02	91.2	47000	54700	409	876	153	73.4	1250	1.46	444
Technosol,	$14 - 16$	19.1	50.3	7470	$<$ DL $\,$	96.1	43100	50300	355	863	138	70.3	1080	1.49	410
unvegetated	$16 - 18$	23.8	81.8	7570	1.18	107	54200	64300	481	996	246	81.9	1390	1.49	723
	$18 - 20$	35.2	49.2	5200	0.991	76.5	47600	53400	404	847	148	74.2	1140	1.56	426
	$20 - 25$	21.6	77.9	5160	0.618	97.5	49100	54900	1640	671	182	71.9	1190	1.73	446
	$25 - 30$	17.9	61.2	4510	0.645	112	45400	52600	324	812	121	65.5	1230	1.35	466
	$30 - 40$	16.9	77.4	4560	$<$ DL	86.1	40800	57600	308	802	121	63	1230	1.38	530
	$40 - 50$	20.5	71.6	6490	1.26	92.6	49200	62600	419	1020	206	72.8	1450	1.66	734
	$50 - 60$	24.5	70.4	5730	0.347	104	52600	62300	402	1040	144	77.9	1520	1.85	575
	$0 - 2$	21	73.9	3860	1.31	90	47400	52200	287	925	123	68.4	1570	1.68	516
	$2 - 4$	21.9	60.2	7210	0.415	117	58500	61700	476	1180	130	80.1	1700	1.55	623
	$4 - 6$	24.3	76.2	9490	0.689	110	58100	64500	452	1210	128	82.1	1620	1.69	554
	$6 - 8$	23.7	64.9	10300	1.12	106	57600	64700	595	1130	176	76.4	1720	1.66	566
	$8 - 10$	28.4	75.9	8040	2.04	175	62600	63400	503	1150	169	90.1	1890	1.94	768
	$10 - 12$	29.6	78.8	4520	0.717	110	51100	62400	1030	843	136	77.8	1530	1.8	483
40% organic	$12 - 14$	15.4	32.2	10500	0.631	68.7	41800	39000	593	938	88.6	57.3	848	0.816	374
Technosol,	$14 - 16$	11.5	22.2	7800	$<$ DL $\,$	204	36700	31300	782	739	88.3	54.6	576	0.683	382
vegetated	$16 - 18$	10.2	31.3	3340	$<$ DL $\,$	64.5	35200	32200	299	698	103	48.1	773	0.81	291
	$18 - 20$	14.8	29.7	1250	1.37	63.8	33300	31300	368	577	78.4	46	732	0.757	418
	$20 - 25$	14.1	28.6	1150	1.01	68.4	45000	34700	525	586	67.2	46.8	849	0.805	453
	$25 - 30$	14.4	36.9	916	0.427	79.9	42400	40300	349	586	103	54.5	1030	0.884	301
	$30 - 40$	12.9	32.9	6410	0.345	79	36500	34200	618	632	107	50.4	988	0.837	278
	$40 - 50$	10.3	28.2	669	0.849	54.3	32400	29400	274	526	88.5	39.8	717	0.677	255
	$50 - 60$	12.4	27.6	3030	0.655	79.8	38500	34000	340	684	80.2	51.4	888	0.764	326

Table D19. Total concentrations of nutrients and elements of environmental interest in Technosol and comparison soil profiles.

Profile	Depth	As	\bf{B}	Ca	Cd	Cu	Fe	$\bf K$	Mg	Mn	Mo	Ni	\mathbf{P}	Sb	Zn
	(cm from	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
DL	surface)	0.2		5	0.05	0.05	\boldsymbol{l}	6	0.1	0.02	\mathfrak{Z}	0.2	1	0.07	0.06
	$0 - 2$	0.015	0.026	109	$\langle DL$	0.0009	2.82	33	3.63	0.013	0.296	\bigtriangleup DL	\triangle DL	0.0015	0.081
	$2 - 4$	0.017	0.044	69.4	0.0014	$<\!\!DL$	1.2	28.8	3.13	0.008	0.239	\langle DL	$<\!\!DL$	0.0013	0.078
	$4 - 6$	0.019	0.015	65.3	$<$ DL	0.0003	0.801	29.1	3.33	$<$ DL	0.176	\bigtriangleup DL	$<$ DL	0.0012	0.059
	$6 - 8$	0.024	0.02	61.1	$<\!\!DL$	$<\!\!DL$	0.783	29.4	3.84	0.002	0.271	\bigtriangleup DL	$<$ DL $\,$	0.0014	0.063
	$8 - 10$	0.027	0.034	74	$<$ DL	0.0053	2.28	33.4	5.8	0.066	0.269	\bigtriangleup DL	$<$ DL $\,$	0.0018	0.102
	$10 - 12$	0.035	0.02	56.2	$<$ DL	0.0099	3.46	29.4	6.47	0.132	0.181	\bigtriangleup DL	$<$ DL	0.0019	0.104
40% organic	$12 - 14$	0.029	0.024	55.7	$\langle DL$	0.0036	1.29	30.3	5.31	0.042	0.214	\langle DL	$<$ DL $\,$	0.0016	0.063
Technosol,	$14 - 16$	0.034	0.027	62.9	$\langle DL$	0.0055	3.11	29.8	7.69	0.121	0.199	\triangle DL	$<$ DL $\,$	0.0021	0.107
unvegetated	$16 - 18$	0.033	0.023	52.7	$<$ DL	0.0048	2.87	28.3	6.98	0.113	0.187	\bigtriangleup DL	$<$ DL $\,$	0.0015	0.1
	$18 - 20$	0.032	0.026	54	$<$ DL	0.0039	2.91	29.8	7.38	0.123	0.178	\langle DL	$<$ DL	0.0016	0.093
	$20 - 25$	0.031	0.027	55.4	$<$ DL	0.0011	1.08	30.5	6.17	0.031	0.188	\triangle DL	$<$ DL $\,$	0.0014	0.062
	$25 - 30$	0.034	0.036	59.2	$<$ DL	0.0034	2.82	31.3	7.74	0.121	0.239	\langle DL	\bigtriangleup DL	0.0017	0.101
	$30 - 40$	0.037	0.029	55.8	$<$ DL	0.0062	2.92	33	8.04	0.111	0.225	\langle DL	$<$ DL $\,$	0.0017	0.094
	$40 - 50$	0.038	0.043	66.7	$<\!\!DL$	0.007	3.42	34.7	9.31	0.125	0.275	\langle DL	\triangle DL	0.0021	0.113
	$50 - 60$	0.042	0.048	67.8	0.0003	0.0074	2.97	46	8.56	0.053	0.253	\langle DL	$<$ DL $\,$	0.0016	0.069
	$0 - 2$	0.016	0.056	107	$<\!\!DL$	0.0061	3.63	39.3	5.96	0.026	0.229	\triangle DL	$<$ DL $\,$	0.0011	0.089
	$2 - 4$	0.022	0.061	80.4	0.0003	0.0057	2.44	48.1	6.53	0.016	0.347	\bigtriangleup DL	$<$ DL $\,$	0.0015	0.064
	$4 - 6$	0.023	0.095	87.5	0.0002	0.0107	4.38	50.4	8.8	0.092	0.224	\bigtriangleup DL	$<$ DL	0.0019	0.102
	$6 - 8$	0.033	0.098	99.1	0.0005	0.0143	6.41	51.8	11.8	0.169	0.287	\triangle DL	$<$ DL	0.0016	0.166
	$8 - 10$	0.032	0.16	99.7	0.0004	0.0108	6.1	54.8	12.2	0.154	0.282	\langle DL	$<$ DL	0.0016	0.151
	$10 - 12$	0.032	0.069	85.7	0.0004	0.0101	5.56	51.4	11.7	0.16	0.294	\bigtriangleup DL	$<$ DL	0.0017	0.13
40% organic	$12 - 14$	0.027	0.071	83.7	0.0005	0.0049	2.36	54.9	9.36	0.015	0.351	\langle DL	$<$ DL $\,$	0.0016	0.073
Technosol,	$14 - 16$	0.035	0.096	100	0.0007	0.0077	3.77	59	12.5	0.048	0.363	\bigtriangleup DL	$<$ DL $\,$	0.0019	0.121
vegetated	$16 - 18$	0.025	0.07	87.2	0.0002	0.0082	5.58	53.5	11.9	0.148	0.237	\triangle DL	$<$ DL $\,$	0.0019	0.129
	$18 - 20$	0.025	0.055	89.3	$\langle DL$	0.0047	2.61	54.6	10.1	0.018	0.19	\bigtriangleup DL	$<$ DL	0.0018	0.076
	$20 - 25$	0.023	0.076	93.8	0.0003	0.0052	3.28	55.4	10.9	0.052	0.264	\langle DL	$<$ DL $\,$	0.0014	0.074
	$25 - 30$	0.02	0.068	107	0.0003	0.0043	3.29	59.1	11.7	0.022	0.266	\langle DL	$<$ DL	0.0018	0.065
	$30 - 40$	0.031	0.071	87.4	0.0003	0.008	4.09	55.9	10.7	0.083	0.268	\bigtriangleup DL	$<$ DL $\,$	0.0018	0.134
	$40 - 50$	0.026	0.082	90.7	$<$ DL	0.0055	2.59	59	10.5	0.015	0.236	\bigtriangleup DL	$<$ DL	0.0016	0.069
	$50 - 60$	0.029	0.059	79.4	$\langle DL$	0.005	2.14	56.3	9.38	0.011	0.122	\langle DL	\bigtriangleup DL	0.0015	0.057

Table D20. Water extractable concentrations of nutrients and elements of environmental interest in Technosol and comparison soil profiles.

Appendix E: Plot design and site layout

Not to scale

Figure E78. Internal structure of the thick Technosol plots, including sensor and leachate sampling plate placement. In thin Technosol plots, the leachate sampling plate and MPS-2 sensor are located at the 30 cm depth, and there is no 60 cm 5TM sensor.

Figure E79. Layout of the Technosol plot study site at Barrick Hemlo. Plot treatments are 1: 40% organic, 30 cm; 2: 80% organic, 30 cm; 3: 40% organic, 60 cm; 4: 80% organic, 60 cm. Plots without vegetation are 2A, 3A, 4B, and 1B.