Influence of Amendments and Soil Depth on Available Nutrients and Microbial Dynamics in Contrasting Topsoil Materials Used for Oil Sands Reclamation

by

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Abstract

As of December 2013, the cumulative area disturbed by oil sands mining in NE Alberta was 896 km^2 out of an estimated final footprint of 4,800 km^2 – all of which will require reclamation. Expensive handling costs and scarce soil resources necessitate judicious management and application of salvaged topsoils and soil amendments such that the post-mining landscape is re-established to an "equivalent land capability" of pre-disturbance conditions. Soil microbial communities and microbially mediated nutrient availability are largely overlooked in reclamation analyses, despite their potential in providing a sensitive measurement of ecosystem processes. This study measured nutrient availability and microbiological parameters in directly-placed forest floor mix (FFM) and peat mix (PM), which were compared to natural reference sites. The study was divided into two components: 1) assessing fertilizer and charcoal amendments (reclamation to d ecosite); and 2) assessing topsoil application depths (reclamation to a/b ecosite).

1) The principal study on CNRL's Reclamation Area-1 (RA-1) compares a fertilizer amendment on PM and FFM. I added a charcoal amendment to simulate natural additions to soil from wildfire; and compared reclaimed treatments to recently burned and unburned natural reference sites. Microbial biomass-carbon was greatest in natural and reclaimed organic soils. Burning and charcoal amendments tended to increase metabolic quotient, indicating potential nutrient stress or decomposition inefficiency. Nutrient profiles differed mostly between natural and reclaimed sites, followed by sites receiving fertilizer. Fertilization increased TIN availability by two orders of magnitude above unfertilized treatments, while P and K availability were below natural variation.

2) Syncrude Canada's Aurora Soil Capping Study provided Shallow and Deep topsoil application depths of PM and FFM which were compared to a control receiving no topsoil and a harvested analogue (Harvest). Soil respiration rates were greater in FFM and Harvest than in PM treatments, with no difference attributable to subsoil type or placement depth. Phospholipid fatty acid analysis (PLFA) and community level physiological profiles (CLPP) measured microbial community structure and function, respectively. Non-metric multidimensional scaling ordinations revealed the greatest similarity between FFM and Harvest for available nutrients, PLFA and CLPP analyses. Deep FFM application shared greatest PLFA similarity to Harvest, but Shallow FFM was more similar in CLPP. Shallow PM was more similar than Deep for all parameters measured. PM indicated greater TIN and S availability, and deficiencies in P and K compared to FFM and Harvest.

Preface

The following thesis is composed of original data generated and analyzed by D. Mark Howell, with no data having been published at the time of submission. Data from Chapter 2 "Implications of topsoil application depths to microbial communities and nutrient availability" was presented at the following conferences: poster at the 2014 Alberta Soil Science Workshop, Calgary, Alberta, Canada; poster at the 20th World Congress of Soil Science, Seogwipo, Jeju-do, South Korea; oral presentation at the 2014 Annual Meeting for the Soil Science Society of America, Long Beach, California, USA; poster presentation at the 2015 Alberta Soil Science Workshop, Edmonton, Alberta, Canada.

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She has seen me through this degree, gratuitously providing me with sustenance during long days, and keeping me company during mundane work. I would therefore like to dedicate this work to my fiancée Heather, and to our future family.

~

Acknowledgements

I have several people and organizations I would like to thank for their contribution to this project and to my education. This work, and my degree, would not have been possible without your inspiration and help.

My program leads for my diploma from Lethbridge College (Ken Perl), bachelor’s degree from the U of A (Dr. Anne Naeth) and master’s degree at the U of A (Dr. Derek MacKenzie) have all provided excellent supervision and created a positive atmosphere conducive to learning and self-improvement. My colleagues at the U of A have also helped me on innumerable occasions with help in the field (Sawyer Desaulniers, Nicole Filipow, Sanatan Das Gupta), and lab (Jamal Taghavimehr, Arezoo Amini, Dr. Nduka Ikpo, Dr. Mathew Swallow, Dr. Sylvie Quideau).

Working at Canadian Natural Resources Ltd.’s Horizon Project over the course of three summers gave me valuable insight into what reclamation looks like on the ground. This experience ultimately began my graduate studies. The reclamation team provided formidable guidance (Steve Tuttle), soils expertise (Dr. Benjamin Sey), proficiency in vegetation management (Scott Johnson, Ira Sherr) and supervision (Krista Shea, Meghan Perry, Sherri Hanlon). I would also like to thank Syncrude Canada Ltd. for providing financial support and staff to help with sampling, plot setup and logistics (Richard Kao, Ryan Cameron, Lori Cyprien); and research information (Marty Yarmuch).

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List of Abbreviations

Al	Aluminum
AMF	Arbuscular mycorrhizal fungi
AOSR	Athabasca Oil Sands Region
ASCS	Aurora Soil Capping Study
B	Boron
B/C	Salvaged subsoil blend of B and C horizons from a/b ecosites
$BaCl_2$	Barium chloride
C:N	Carbon to nitrogen ratio
Ca	Calcium
$CaCl_2$	Calcium Chloride
Cd	Cadmium
CEC	Cation exchange capacity
$CHCl_3$	Chloroform
CLPP	Community level physiological profile
CNRL	Canadian Natural Resources Ltd.
CO_2	Carbon dioxide
Cu	Copper
CWD	Coarse woody debris
DOC	Dissolved organic carbon
EC	Electrical conductivity
EIA	Environmental Impact Assessment
F:B	Fungi to bacteria ratio

FAME	Fatty acid methyl ester
Fe	Iron
FFM	Forest floor mix
FMA	Forest Management Agreement
H_2O	Water
Harvest	Harvested reference site (a/b ecosite)
HCl^-	Hydrochloric acid
K	Potassium
LCCS	Land Capability Classification System
LFH	Refers to organic soil horizons in natural upland forest floor
LOS	Lean oil sand
MASL	Meters above sea level
Mg	Magnesium
Mn	Manganese
MRPP	Multiple response permutation procedures
N	Nitrogen
NaOH	Sodium hydroxide
NH_4^+	Ammonium
NMS	Non-metric multidimensional scaling
NO_3^-	Nitrate
NRAL	Natural Resources Analytical Laboratory
OC	Organic carbon
P	Phosphorous

Pb	Lead
PLFA	Phospholipid fatty acid analysis
PM	Peat mix
qCO_2	Metabolic quotient
RA-1	Reclamation Area-1
S	Sulfur
S:M	Saturated to monounsaturated fatty acid ratio
SAGD	Steam-assisted gravity drainage
SAR	Sodium adsorption ratio
SCO	Synthetic crude oil
SO_4^-	Sulfate
SOM	Soil organic matter
SS	Subsoil salvaged from > 1 m depth
SSIR	Sum of substrate induced respiration from CLPP analysis
TIN	Total inorganic nitrogen
TOC	Total organic carbon
VWC	Volumetric water content
Zn	Zinc

1.0 Reclamation in the Athabasca Oil Sands Region

1.1 - Economy and Ecology

Commodity markets driven by global economies are potentially lucrative investments for resource extraction companies. Extraction and manufacturing exploits have improved living standards and human wellbeing however many consequences of these activities remain unseen by the general public due to the large disconnect between consumption and manufacturing. The culmination of these disturbances on natural landscapes can be deleterious to surrounding ecosystems and human health. Due to increasing population size and improved technology, humans are able to modify their environment at unprecedented rates. Degraded lands impart costs in the form of reduced land productivity and marginalized ecosystem services. The arising social consequences establish both moral and economic grounds to mitigate adverse environmental impacts.

Major anthropogenic disturbances range in size and severity, but share similarities in their capacity to reduce ecosystem function. This means an overall disruption in the ecological capacity of lands to provide the hydrology, geology, climatic conditions, decomposition and nutrients that are necessary for their ability to support diverse biotic communities. Open-pit mining for commodities such as precious metals, minerals and oil, is arguably the apex of disturbance severity. Technological advancements have made this technique economically viable, replacing historical subsurface mining in many cases. Open-pit mining requires the complete removal of vegetation, soil and overburden to expose ore-bearing geologic strata. Land is degraded to a state of primary succession upon completion of mining activities, since all biological legacies are removed. If left alone, vegetation would eventually encroach and soils develop, similar to post-glacial landscapes. However sub-surface geologic materials are generally unsuitable for plant growth and would require a massive temporal gap to restore ecosystem function (Bradshaw 1997). Additionally, deleterious environmental impacts would threaten public interests, for which resource extraction companies would be liable. Land reclamation mitigates these negative impacts and expedites ecosystem re-establishment by returning landscapes closer to a state of secondary succession. Therefore reclamation is a crucial response to major land disturbances. Rowland et al. (2009) suggests that site-specific reclamation practices will set a disturbed ecosystem on a trajectory towards its pre-disturbance state (Figure 1.1).

Land reclamation is a multi-faceted procedure requiring appropriate management of available biotic and abiotic inputs to re-establish an ecosystem following a disturbance. Soil management plays a critical role in this procedure. Soils are a theater hosting a variety of essential ecosystem processes, and providing the foundation for terrestrial biota (Bradshaw 2000). From the initial state of primary succession, soils develop over several millennia as a result of parent geological materials, climate, topography and biota, affecting processes of input, loss, transformation and translocation of materials (Jenny 1941). These combine to provide essential ecosystem processes in an interface between the lithosphere, hydrosphere, atmosphere and biosphere. Among the most important services provided are organic matter decomposition, water retention and filtration, habitat for edaphic organisms and nutrient cycling (Sourkova et al. 2005, Sere et al. 2008) – all of which work in concert to enable and regulate vegetative growth. Without developed soil materials, plants would not be able to meet their nutrient or water requirements. Suitable materials for reclamation are undeniably pivotal for re-establishing ecological function following large disturbances.

1.2 - Oil Sands Mining

Extracting oil from bituminous ore deposits in the Athabasca Oil Sands Region (AOSR) of northeastern Alberta has led to significant perturbations of natural boreal landscapes, and is currently a contentious environmental issue debated in the media. Deposits of bitumen near the soil surface were first extracted on a commercial scale in 1967 with the opening of the Great Canadian Oil Sands Company - now Suncor Energy. Increases in the market value of oil, coupled with technological advancements, resulted in greater economic viability for bitumen mining. This created incentive for rapid expansion and lease development by a myriad of national and international energy companies. The oil sands industry is currently the economic mainstay of the AOSR and constitutes a large proportion of the provincial revenue - with \$3.56 billion in royalties for the 2012/2013 fiscal year (Government of Alberta 2013a). Local, provincial and national economic incentives in the oil sand industry promote a rapid expansion trajectory set by industry. The AOSR is now home to 40 operating bitumen extraction projects, 6 of which are open-pit mines and the rest are in-situ extraction operations, such as steam assisted gravity drainage (SAGD)(Government of Alberta 2015). Both the intensity and scale of this disturbance present major difficulties to mine closure planners. The approximate footprint of mining disturbance is estimated to be 896 km^2, with a total projection of 4,800 km^2. Of this, roughly 82 km^2 are at some stage of reclamation, and only 104 ha has received reclamation certification (Figure 1.2). This is partly because reclamation objectives are poorly defined, and therefore it is difficult to conclusively determine whether a release of liability is acceptable.

1.3 - Oil Sands Extraction Process

Resource extraction companies mine bitumen where it is economically viable. This limits mining activities within close proximity to the Athabasca River, north of Fort McMurray, Alberta. Briefly, oil sands extraction begins with vegetation removal, where merchantable timber is logged by forest management agreement (FMA) holders, while un-merchantable products are typically piled into slash piles and burnt, or salvaged for coarse woody debris (CWD) amendments on reclaimed sites (Alberta Environment and Water 2012). Cleared land is then dewatered and soils are salvaged as per the terms arranged in the mineral lease agreement between proponent and regulator. Geologic materials between the soil surface and ore bodies are termed overburden, and are placed in overburden dumps or engineered into structures such as roads, ramps and dykes. Much of these materials contain highly saline strata, or strata with low concentrations of bituminous hydrocarbons. Due to the high costs associated with material transport, the ratio of overburden to ore to must not surpass 12:1, which typically precludes mine pits depths exceeding 75 m (Government of Alberta 2009). Ore is classified as material containing > 6 % bitumen by weight, with lower bitumen concentrations classified as overburden, inter-burden and lean oil sand (LOS). On average, 2 tonnes of ore is required to produce 1 barrel of synthetic crude oil. Deposits in excess of mining depths are typically extracted using SAGD technology. Hydraulic or cable shovels, capable of excavating up to 150 tonnes per pass, load 400 tonne haul trucks for a continuous feed from the pit face to the crusher units. Here, ore is broken down into smaller aggregates, mixed in a slurry with hot water and hydro-transported to upgrading facilities. Naphtha is added to the slurry as a diluent to help separate hydrocarbons from mineral geologic material. Hydrocarbons float to the surface as a froth in separation vessels, which is removed for refining. The remainder of the slurry includes fresh water, residual naptha and mineral solids. These are hydro-transported to large tailings retention facilities where water is recycled and sediments are left to settle. Hydrocarbons separated from the slurry are cracked at high temperatures in a coking unit, converting long chain hydrocarbons into lighter fractions and producing a petroleum coke by-product. Distillates are segregated and hydrotreated (saturated with hydrogen), which results in more stable chemical structure. This process removes sulfur (S), nitrogen (N) and trace metals. Coke and S are produced in large quantities and stockpiled. Finally, distillates are mixed to produce synthetic crude oil (SCO) which is shipped via pipeline to southern refineries.

1.4 - Federal and Provincial Regulations

Canada's economy has been largely dependent on its vast and diverse stores of natural resources, from fur trading in early colonialization, to mineral mining and oil extraction in the present day. The abundance of natural resources has created economic incentive for resource extraction disturbances. Canada is now an industry leader in mining activities with more than 50 % of the total number of mining companies globally having headquarters in Canada (Foreign Affairs, Trade and Development Canada 2015). The federal government regulates certain components of major industrial disturbances within its borders; however these are usually confined to controlled substances or issues where impacts may extend across political boundaries. Environment Canada, the federal regulating body, may require an environmental impact assessment (EIA) for large projects, or regulate projects that impact navigable or fish-bearing waters.

Regulation of resource extraction activities lies within provincial jurisdiction as a result of the *Natural Resources Transfer Agreements*, which bestowed natural resource governance in western Canadian provinces to provincial regulatory authorities in 1930. The Alberta government recognized the need to regulate such activities to protect the public against environmental degradation, and in 1973 the first reclamation legislation was introduced as the *Surface Reclamation Act* (Powter et al. 2012). Over time, this legislation evolved to encompass ecologically relevant requirements and increased environmental protection under several acts. Environmental legislation was compiled in 1993 to form the *Environmental Protection and Enhancement Act*; under which the *Conservation and Reclamation Regulation* requires operators to return land to an "equivalent land capability" before mineral leases can be returned to public ownership. Regulations specific to the disturbance in question have evolved from this legislation; including soil salvage requirements, segregated soil stockpiles and capping soil placement depths. Proponents of projects incurring major disturbances must first perform assessments of potential ecological and socio-economic impacts arising from their projects. Mine closure plans are developed in accordance with this legislation and are submitted to the appropriate regulatory body for approval. Lease agreements between government and project proponents are developed to further specify operating limitations specific to the area of disturbance.

1.5 - The Boreal Region

Oil sand mining disturbances are mostly found in the Boreal Forest Natural Region – Central Mixedwood Subregion (Natural Regions Committee 2006). Annual averages (1981-2010) for Fort McMurray list 418.6 mm of precipitation with 316.3 mm falling as rain; and a daily average temperature of 1 °C (Environment Canada 2015). Summer temperatures are hottest in July and August with an average daytime temperature of 17.1 °C and 15.4 °C, respectively. Trees native to well drained upland positions include jackpine (*Pinus banksiana* Lamb.) and some trembling aspen (*Populus tremuloides* Michx.) in dry stands on Brunisolic soils; and trembling aspen and white spruce (*Picea glauca* Moench) in mesic stands on Luvisolic soils (Beckingham and Archibald 1996). Highly variable drainage patterns intersperse uplands with complex hydrologic networks of wetlands. Beaver (*Castor Canadensis* Kuhl) activity adds to this by restricting flow and creating bogs and fens. Saturated lowlands possess thick organic layers of peat mosses (*Sphagnum spp.*) under various stages of decomposition, and support black spruce (*Picea mariana* Mill.), birch (*Betula spp*.) and tamarak (*Larix laricina* Du Roi) in saturated lowland Organic and Gleysolic soils. High salinity is naturally occurring in some soils in this region as a result of geologic inputs. This is expressed in vegetative community composition and tree rooting depths (Purdy et al. 2005).

In addition to variable landscape positions and soil types, periodic natural disturbances shape the heterogeneous structure of boreal vegetative communities. On a small scale, disturbance from pathogens, beaver dams and windfall add to the boreal's structural mosaic. However, fire is the most influential natural disturbance in boreal regions due to its capacity to impose landscape-scale effects. Typically 5 – 10 % of boreal landscapes remain unburnt past stand ages > 200 years; however fire frequency varies greatly (Johnson et al. 1995). Vegetative communities recovering from wildfire have adapted to conditions characteristic of post-fire environments including modified temperature ranges, increased photosynthetically active radiation at the soil surface, charred organic matter on the soil surface, and altered soil nutrient dynamics (Zackrisson et al. 1996).

Resource extraction is increasingly becoming a major additional disturbance to boreal landscapes in the form of timber harvesting, oil exploration and mining operations. These are evolutionarily unprecedented disturbances, which hold the potential of developing novel ecosystems. However, anthropogenic disturbances can use the aforementioned natural disturbances as benchmarks for reclamation success.

1.6 - Reclamation Practices

1.6.1 - Topsoil types used in oil sands reclamation

Following vegetation clearing, lease holders are required to salvage suitable topsoils for later use in reclamation. Provincial authorities mandate soil salvage and tailor requirements to individual lease agreements. Salvaged soils are segregated based on topographic position in their natural location and texture. There are two main categories for reclamation topsoils.

Firstly, a peat mixture (PM) consisting of decomposing sphagnum peat deposits and surrounding mineral soils salvaged at a ratio of 60:40. This is a general ratio and varies greatly since some PMs lack mineral components. Additionally, peat and mineral materials may not thoroughly mix – this is especially evident in fine-textured mineral soils with strong aggregation. Historically, PM has been the preferred topsoil used in reclamation due to its abundance on mineral leases. It is salvaged using a shovel/haul truck combination after dewatering, typically in winter months.

Secondly, government regulations have recently mandated salvage of upland forest floor mixture (FFM). This mixture typically includes the LFH and the A horizon in an approximate 10 – 20 cm lift of natural forest soils. Dozer operators windrow FFM so it can be transferred into haul trucks using excavators. Salvaging too deep can admix soil horizons and dilute beneficial soil properties characteristic of the upper solum (Rokich et al. 2000). Quality of this material varies greatly depending on texture, and therefore operators segregate FFM between fine and coarse textured mineral soil. Forest floor mix is the preferred topsoil for use in reclamation due to its inherent propagule bank and similar soil characteristics to what existed prior to disturbance (Mackenzie and Naeth 2010, MacKenzie 2013). Industry sometimes refers to this material simply as LFH, however in its purest definition this is a misnomer since LFH refers to the surficial organic horizon in natural upland soils. Once salvaged, it becomes a reclamation material and no longer has a distinguishable LFH horizon.

Finally, transitional soils are salvaged from forested areas with imperfect drainage and an organic horizon not exceeding 40 cm. Suitable subsoil is also salvaged and stockpiled for later use. All soil types must be salvaged and stockpiled separately as to avoid admixing and potential contamination.

1.6.2 - Direct placement

The greatest benefit to reclamation is derived from directly placed topsoils following salvage (MacKenzie 2013). This approach has less impact on soil biogeochemical conditions and maintains propagule viability when compared with stockpiling. Additionally, material handling is the greatest cost in mining operations. Progressive reclamation is a mining model used to mitigate these costs by initiating reclamation immediately following mine landform decommissioning. These new landforms are then contoured and capped with subsoils and topsoils salvaged from the mine advance, and are immediately placed and spread to the desired thicknesses. This practice not only provides better reclamation outcomes, but also reduces handling costs and space requirements for stockpiles. However, this is not always possible due to timing of donor site salvage with an available receptor site. Most of the soils used for reclamation in the final landscape will come from stockpiles.

1.7 - Soil Preparation

1.7.1 - Application Depth

Soil depth varies greatly in natural systems due to topographic position, annual precipitation and vegetation type. Therefore, fixing a standard application depth for all reclamation scenarios is not practical. Capping depth requirements issued by the regulator are determined on a case-by-case basis and by the current legislation at the time of mineral lease approval. As a result, soil depths vary between and within mine sites. Depth recommendations are intended to isolate the rooting zone from adverse materials and provide sufficient nutrient and water holding capabilities to support forest vegetation of the desired ecosite. In general, cover soil material must be applied to a minimum of 0.2 or 0.5 m over tailings sand or suitable overburden, respectively. A minimum of 1 m capping depth is mandated over adverse materials such as LOS and saline/sodic overburden. Abrupt textural differences at the interface between soil layers is another important consideration to avoid impeding root penetration and restricting vertical water movement (Naeth et al. 2011).

1.7.2 - Roughness

Surficial microtopographic variability provides greater site heterogeneity for plant establishment (Alberta Environment and Water 2012). This practice improves soil water conditions by trapping blowing snow, and providing shelter for establishing vegetation. Overland flow is slowed, promoting infiltration and reducing erosion. Species that disperse seed by wind will have a greater chance at establishing on rough surfaces due to slower wind speeds at the soil surface.

1.7.3 - Amendments

Amendments are often used to promote initial vegetation establishment and to counteract soil nutrient deficiencies. Fertilizer (N-P-K-S) applications are targeted to provide an initial pulse of nutrition for establishing vegetation. The greatest application rate is typically in the first growing season; however it may be applied for up to 5 years following reclamation (Pinno et al. 2012). This is accomplished using farming implements where accessible, or by aerial application. Concerns with fertilizer application include over-use resulting in promotion of weedy species establishment, leaching and atmospheric losses.

Coarse woody debris applications are becoming increasingly common (Alberta Environment and Water 2012). This technique resembles naturally disturbed stands where woody debris remains on-site. Benefits of using CWD include additional soil carbon and nutrients from decomposition, microsites for establishing vegetation, erosion control and increased moisture retention (Brown 2010).

1.7.4 - Soil Stabilization

Agricultural barley (*Hordeum vulgare*) is often applied as a nurse crop in the first growing season which serves to stabilize soil and initiate soil-plant interactions. It may be seeded with farming implements or aerially broadcast. Reclaimed sites are seeded late in the growing season such that plants do not reach maturity prior to frost kill. Since it is an annual species and the seed is not viable, barley will not regenerate the following year.

1.8 - Planting

Following site preparation, tree seedlings grown in a nursery are planted as per recommendations provided in *Guidelines for Reclamation to Forest Vegetation in the Athabasca Oil Sands Region* (Alberta Environment 2010). The type of species planted depends on the target ecosite for the reclaimed area. The most common species are white spruce, jack pine and trembling aspen. High-value shrubs species may also be included in planting prescriptions. Seedlings are typically rooted in a plug, but may also be bare-root stock. Other species migrate to reclaimed sites without human assistance through seed dispersal using wind and animal transport from natural areas.

1.9 - Difficulties with Reclaimed Soils

1.9.1 - Site Assessment Tools

Assessing success of reclamation on landscapes is challenging due to the complexity of natural systems. The Land Capability Classification System (LCCS) provided an operational model for companies to assess site productivity based on soil moisture and nutrient regimes, and included useful measurements of soil quality (Cumulative Environmental Management Association 2006). Yet these assessments oversimplified soil health and did not provide adequate discriminatory power, therefore the LCCS was discontinued. Operating over such a vast area means that a diversity of challenges will be encountered and developing a tool that will satisfy demands will be challenging, but necessary.

1.9.2 - Salinity

Due to naturally occurring salinity in the region, some soils possess greater electrical conductivity (EC) and sodium adsorption ratios (SAR) than regulatory guidelines permit. Additionally, some shales found in the overburden are highly saline. Purdy et al. (2005) measured different plant communities in naturally saline soils than non-saline soils and suggested that it may be unreasonable to expect vegetative communities similar to non-saline soils on reclaimed lands.

1.9.3 - Stockplies

A major impact of mining will become apparent in the near future as stockpiled soils are used for reclamation. Due to incongruent timing of soil salvage with landform reclamation, direct placement is usually not achievable and large quantities of soil materials must be stockpiled for decades, prior to application. Stockpiling affects soil temperature, water content, gasses, redox conditions and incurs losses due to erosion (MacKenzie 2013). Some of these issues can be mitigated by storing topsoils in smaller stockpiles and planting them with desirable species. Soil propagule banks are mostly lost within the first 16 months after stockpiling.

1.9.4 - Biodiversity

Recent practices using directly placed FFM for topsoil and applying CWD to sites have helped in achieving greater aboveground biodiversity. Yet traditional practices of fertilization and nurse crops can sometimes prove counter-productive. Fertilizer overuse can promote weedy species establishment which can out-compete native vegetation (Pinno and Errington 2015). This also poses concerns for nutrient runoff and atmospheric losses of N from denitrification. While nurse crops may initially stabilized soils, mitigate nutrient losses and provide organic matter to newly reclaimed soils, they are often possess vigorous growth rates and may out-compete other vegetation in the first growing season.

1.9.5 - Scarcity and Depth

Arguably the greatest challenge of oil sands reclamation is the size of the disturbance. This creates a large demand for already scarce soil materials and invokes economic restrictions associated with material handling costs. Most of the final mined landscape will be upland topography, but a large component of mineral leases are naturally covered by wetlands. Therefore access to topsoils formed in upland positions (like FFM) are in short supply. Material balances must be accurately calculated from the beginning of the mining process to ensure soil coverage of the final landscape. If insufficient quantities of soil material were salvaged during land clearing, the final mine footprint will not possess sufficient quantities for final closure.

The Best Management Practices publication (Alberta Environment and Water 2012) acknowledges a knowledge gap in determining minimum capping depths. Typically, the LCCS was employed to help determine requisite depths based on a soil moisture index and a soil nutrient index (Cumulative Environmental Management Association 2006) – however this has recently been discontinued. Currently, suitable soils are applied at pre-determined depths specific to individual lease approvals. Generally, a range of 20 - 50 cm of topsoil is applied to suitable overburden. If the landform to be capped contains deleterious substances, a minimum cap of 100 cm is applied; however companies usually place at least 100 cm subsoil with an additional topsoil application (Alberta Environment and Water 2012). Shallow application depths will permit greater coverage of the final footprint and could be an efficient use of directly placed soils.

1.10 - Research Outline

Soil biogeochemistry is responsible for many of the ecosystem processes needed to return "equivalent land capability" to reclaimed mine sites. Recently, several research programs are focusing on soil biogeochemical assessments in the AOSR, specifically looking at soil organic matter (SOM) quality (Hannam et al. 2004), litter layer genesis (Sorenson et al. 2011), nutrient availability and uptake (MacKenzie and Quideau 2010, Quideau et al. 2013) and microbial community structure and function (Swallow et al. 2009, Dimitriu et al. 2010, MacKenzie et al. 2014). My research intentions are to contribute to existing measures of soil biogeochemical relationships in oil sands mine reclamation. Most criteria used to evaluate soil performance in reclaimed scenarios do not incorporate soil biological measurements, rather chemical and physical parameters are used as proxies. Furthermore, these assessments have a foundation in agricultural methods which aim to optimize soil productivity rather than re-create diverse and functioning ecosystems. Instead, I take a holistic approach to measuring soil nutrition and biological activity, not in the context of productivity, but in establishing soil function similar to natural benchmarks. My first study assesses fertilizer and charcoal amendments on PM and FFM soils to assess if these amendments bring reclaimed soils within the variation observed in soils disturbed by natural wildfire disturbances on Canadian Natural Resources Ltd.'s (CNRL) Horizon Project. This was evaluated in the context of soil microbial biomass and activity, and plant available nutrition. The second study elucidates microbial community structure and function, and nutrient availability in PM and FFM soils in two different topsoil capping depths versus a re-forested analogue on Syncrude Canada's Aurora Soil Capping Study (ASCS). This research could contribute to developing new methodologies for assessing reclamation trajectory, improving upon the LCCS.

Figures

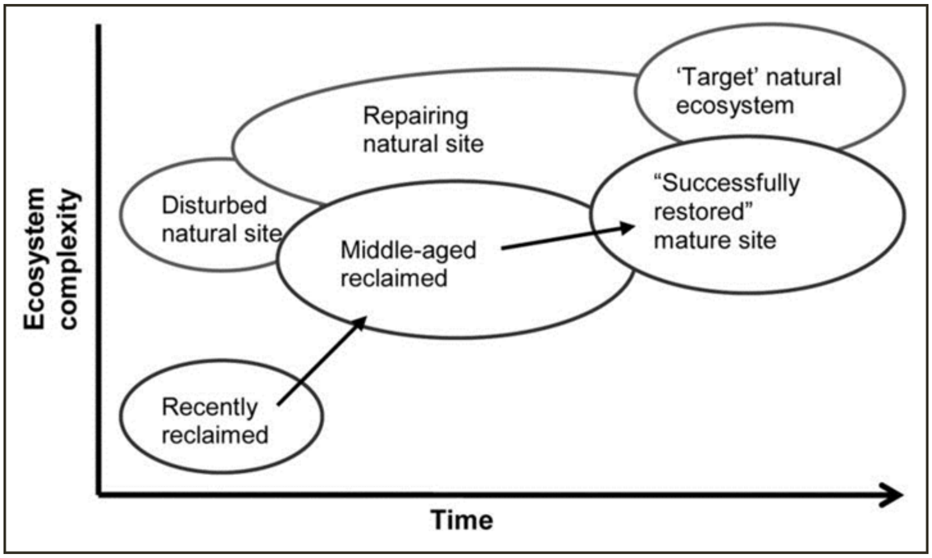

Figure 1.1 – Conceptual model of site restoration following natural and anthropogenic disturbances; modified from Rowland et al. (2009).

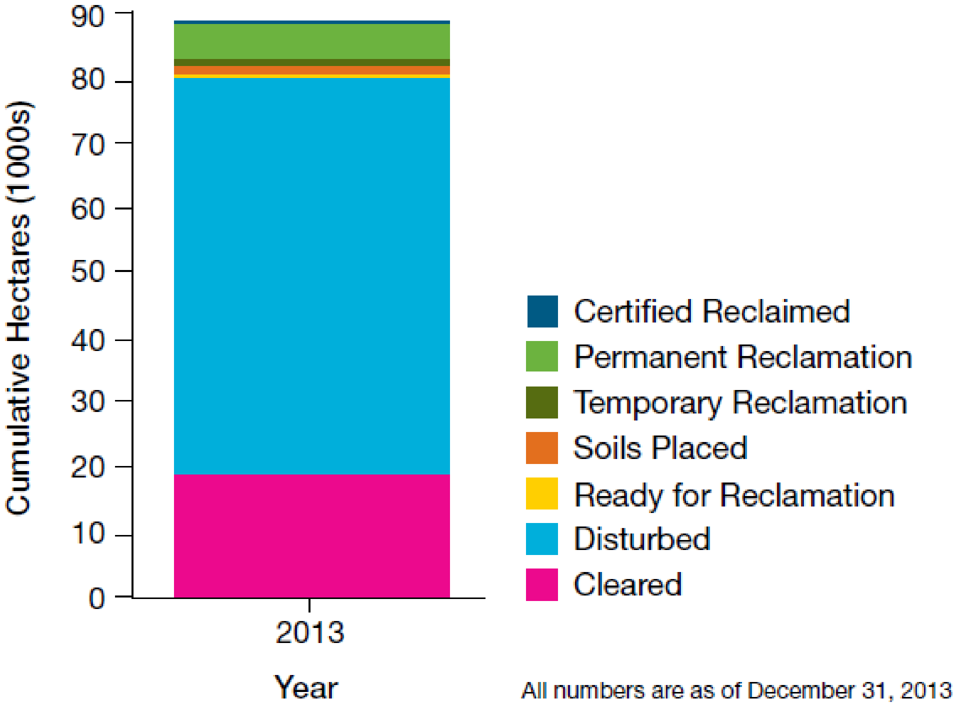

Figure 1.2 – Extent of land disturbed for oil sands mining is estimated to be 896 km^2 as of December 31, 2013 (Government of Alberta 2015).

2.0 Effects of fertilizer and charcoal amendments on PM and FFM nutrient availability and microbial communities

2.1 - Introduction

Reclamation practices must balance operational, technological and economic constraints with environmental variables, resource availability and regulatory requirements. Integrating these limitations with current best management practices and contemporary research assists reclamation practitioners returning land to an "equivalent land capability" of its pre-disturbance state, in compliance with lease approvals (Alberta Environment 2010). Typical measurements of success in the AOSR use standard agricultural and forestry soil assessments which mainly focus on high fertility. In contrast, the primary goal of reclamation is to return ecosystem function and processes to an acceptable condition and mitigate adverse environmental impacts (Gosselin et al. 2010).

Progressive reclamation is a technique where reclamation and site decommissioning occur concurrently with mining disturbances (Alberta Sustainable Resource Development 2010). When mine structures are no longer in use, such as in the case of overburden dumps, materials are contoured to create geotechnical stability, integrated drainage plans and ecologically beneficial topography. Soils are salvaged and applied in the winter to reduce compaction potential. Bare soils are planted with key woody species and are sometimes seeded with an agronomic barley nurse crop to stabilize soils and to begin re-establishing soil processes (Fung and Macyk 2000, Pinno et al. 2012). Disturbance, like in the case of soil salvage, has been shown to increase NO_3^- availability by disrupting plant-microbe interactions and by favouring nitrification (MacKenzie and Quideau 2010). Yet reclamation practitioners frequently amend newly reclaimed sites with fertilizer, sometimes for several seasons, to promote initial vegetative establishment.

In natural boreal forest stands, wildfires have been shown to increase available inorganic N which likely contributes to aggressive vegetative reestablishment following these disturbances. Wildfires also contribute black carbon in the form of charcoal to boreal soils which impacts soil biogeochemistry. Zackrisson et al. (1996) found natural charcoal loads to vary from 984 – 2,074 kg ha^{-1} in boreal soils, which appeared to impact ecological function through sorptive qualities. Depending on the feedstock, black carbon has been shown be influential in boreal soils since it possess a high surface area, contributes nutrients from ash, retains water and immobilizes labile nutrients in the soil, and therefore may prove beneficial in specific reclamation scenarios (Preston and Schmidt 2006).

The first reclaimed mining landform on CNRL's Horizon Project was designed as an operational scale study to compare typical reclamation materials and amendments used in the industry. The principal study assesses FFM compared to PM, both with unfertilized, fertilized and fertilized with CWD application. Reclamation on this site began in the same season that a wildfire burnt within the mine's mineral lease, which created a local comparison of ecosystem restoration following natural and anthropogenic disturbances. This study's aim is to provide ecologically relevant measures of soil quality to determine the benefit of conventional and naturally relevant amendments on two reclamation soil types. Plant available nutrients and basic microbial measures were compared to natural burned and unburned sites in upland and lowland topographic positions. Analyses of in-situ conditions and microbiological assessments of soil materials provided a sensitive analysis of reclamation material quality. Three main hypotheses were generated by this research: 1) an amendment of 2,000 kg ha^{-1} charcoal to reclaimed soils would decrease nitrogen availability and increase microbial activity; 2) FFM would have nutrient availability more akin to naturally disturbed soils than would PM; and 3) fertilizer amendments at 100 kg N ha^{-1} would provide available inorganic nitrogen in concentrations exceeding the observed variation in natural benchmark soils following a wildfire disturbance.

2.2 - Materials and Methods

2.2.1 - Study Area

Canadian Natural Resources Limited's Horizon Oil Sands Project began construction in 2005, with first production of synthetic crude oil (SCO) in early 2009. The Horizon Project is located 70 km north of Fort McMurray, Alberta, Canada in the AOSR. The first phase is expected to produce 110,000 bbl day^{-1} with a planned expansion up to 250,000 bbl day^{-1}, ranking the Horizon Project among the top SCO producers in the region (Canadian Natural Resources Ltd. 2015). CNRL has one of five upgrading facilities in Alberta, exporting SCO to southern refineries via pipeline and stockpiling extraction by-products on-site (petroleum coke, elemental sulfur and tailings) (Government of Alberta 2013b).

Surficial geologic material on this site is morainal to fluvial in origin moving west to east where it borders the Athabasca River (Golder 2002). Upland forest soils are dominated by Orthic Gray Luvisols on fine textured parent material in higher elevations to the west, integrating to Brunisolic Gray Luvisols and Brunisols approaching the Athabasca River. These latter soils are dominated by fine sand and sandy fluvial parent material. Uplands are interspersed by a hydrologically complex network of wetlands, represented by Organic or Gleysolic soil orders.

As a young mine, very few reclamation projects have been completed on site. The first reclamation area surrounds Wãpan Sãkahikan (Horizon Lake), a fish-habitat compensation lake built to offset the destruction of 14 km of the Tar River; however this site was not previously mined. The first mining disturbance to see reclamation was Reclamation Area-1 (RA-1), an 88 ha overburden dump consisting of unsuitable saline/sodic materials derived from the Clearwater Formation (Canadian Natural Resources Ltd. 2013). This site was designed as an operational-scale experiment to compare PM and FFM topsoils, conventional fertilizer amendment and coarse woody debris application on the Horizon Project (Figure 2.1). The research area is divided into a 2 × 2 factorial design of FFM and PM, fertilized and unfertilized. The fertilizer amendment was aerially broadcast at 100 kg N ha^{-1} of 22.9 - 9.1 - 9.1 - 9.1 N-P-K-S in 2011 (date unconfirmed) and on June 18, 2012.

Slated for reclamation in 2011, RA-1 was contoured to gently rolling topography and soils were directly placed from the mine advance in the winter months to maintain native soil characteristics and avoid compaction. Subsoil was placed to an average depth of 1.6 m across all treatments on RA-1, which exceeds minimum regulatory requirements pertaining deleterious overburden (Canadian Natural Resources Ltd. 2013). Forest floor mix was intended to be applied to 20 cm depth, and PM to 40 cm. Due to the scale of operations, large machinery used to execute these operations lead to inaccurate and imprecise placement depths. Actual topsoil depths measured an average of 40 cm and 47 cm for PM and FFM, respectively. Microtopographic variability of soil placement was encouraged since it creates localized environmental conditions that are beneficial to plant recruitment and establishment in accordance with reclamation best management practices in the AOSR (Alberta Environment and Water 2012). A barley nurse crop was seeded via fixed-wing aircraft late in the first growing season such that it was unable to produce viable seed before first frost. Application accuracy with the aircraft is based on pilot skill and GPS navigation and therefore may vary in the order of ± 10s of meters, meaning some overlap and some areas missed. After barley establishment in August, white spruce (*Picea glauca* Moench) seedlings were planted at a density of 2,000 stems ha^{-1}.

In the spring following soil placement, several wildfires spread through forests in the AOSR. The congruent timing of the Richardson Fire with reclamation on RA-1 presents a very unique opportunity to compare reclamation practices with natural disturbance, and to measure the differences between plant available nutrients in soils from naturally disturbed sites and reclaimed sites. The Richardson Fire intruded into the Horizon Project's mineral surface lease providing a study area of close geographical proximity creating optimal conditions for a close comparison chronologically and over environmental variables.

2.2.2 - Plot setup

Plots were delineated to 4 × 4 m in triple replicate for each of the reclamation topsoil types (FFM and PM), in unfertilized and fertilized treatments, with and without charcoal amendment. Charcoal-amended plots were located immediately adjacent to un-amended plots. Reclaimed treatments were compared to natural reference soils (Organic and Luvisolic) that were both unburned and recently burned by wildfire. Plot locations were chosen based on level gradient (less than 2 %) and top or upper slope topographic positions. Replicates were no closer than 100 m apart to minimize the chance that soils from each plot came from the same haul truck when applied.

Burnt slash piles of un-merchantable timber along the mine advance, derived from aspen-white spruce communities, were the source of the charcoal amendment. An axe head was used to shave charred wood from incompletely combusted logs. Charcoal was pulverized to maximize the reactive surface area and subsequently broadcast at a rate of 2,000 kg ha^{-1} dry weight, immediately following fertilizer application on June 19, 2012. This was based on similar loads in boreal soils described by Zackrisson et al. (Zackrisson et al. 1996).

2.2.3 - Field Work

General soil lab analyses are costly and typically do not provide accurate measures of in-situ nutrients available in soil solution. Plant root simulator (PRS™) probes (Western Ag Innovations, Saskatoon, SK) provide a biologically relevant representation of in-situ nutrient availability since probes are incubated in an identical environment to plant roots (Qian and Schoenau 2002). Briefly, cation and anion exchange membranes enclosed in a protective plastic structure provide exchange sites for ions in soil solution. Probes were inserted such that the top of the probe was flush with the soil surface. Four anion/cation probe pair subsamples were installed in each plot from July 17 to August 28, 2012 whereupon they were removed, cleaned with a brush and de-ionized H_2O, and returned to Western Ag Innovations Inc. for elution in 0.5 M HCl. Colourimetry analysis yielded available NH_4^+ and NO_3^- using a segmented flow Autoanalyzer III (Bran and Lubbe, Inc., Buffalo, NY); and P, K, S, Ca, Mg, Mn, Al, Fe, Cu, Zn, B, Cd, and Pb were measured using various methods including inductively-coupled plasma spectrometry (ICP), atomic adsorption spectrometry (AAS) and flame emission spectrometry (FES). Maximum adsorbance values are listed in Table 6.1.

A composite of 4 soil samples were taken immediately adjacent to the burial location of PRS™ probe pairs. Soil aggregates were manually processed to break down structure and mixed in sample bags. Samples were stored in a deep freeze, transported to Edmonton in coolers and kept at 4 °C until laboratory analysis could be performed.

2.2.4 - Lab Work

Field water content was measured by weighing samples before and after drying at 105 °C for 24 hours. Water holding capacity (WHC) was determined for each composite sample. Pressure plates and samples were saturated with distilled water for 24 hours prior to the experiment. Samples were then placed on 0.1 and 0.05 MPa pressure plates, sealed in pressure chambers, and pressurized to 0.1 and 0.03 MPa for 24 hrs for mineral and organic samples, respectively. Samples were removed, weighed, dried at 105 ^{0}C and weighed again to obtain maximum water holding capacity (Kalra and Maynard 1991).

Un-sieved soils were measured to 200 g and 150 g for mineral and organic samples respectively, and were brought to 60 % WHC in 1 L mason jars. Organic soils had much higher water content than 60 % WHC and were not adjusted. Jars were sealed and soil was pre-incubated at room temperature for 40 days to mitigate the impact of freezing the samples during travel, and to avoid measuring a priming effect. During pre-incubation, jars were kept at 22 °C in darkness and vented every 3 days by removing lids for 30 min to release accumulated CO_2. Water loss from evaporation was returned prior to resealing. Scintillation vials containing 20 mL of 1 M sodium hydroxide (NaOH) sample solution were added to mason jars to absorb atmospheric CO_2 during the incubation. Samples were incubated at room temperature (22 °C) for 7 days, whereupon scintillation vials were removed, immediately capped and refrigerated at 4 °C until further analysis. Sample solution (10 mL) with 15 mL of 2 N barium chloride ($BaCl_2$) and 3 drops of phenolphthalein indicator solution, was titrated with 1 M hydrochloric acid (HCl^-) until colour change was apparent (Zibilske 1994, Hopkins 2007).

Microbial biomass-carbon (MB-C) was measured using the chloroform ($CHCl_3$) fumigation-extraction method (Vance et al. 1987). Concurrently with commencement of basal respiration analysis, 25 g mineral soil and 10 g peat soil were weighed into glass beakers. Two jars were used for each sample; one for chloroform ($CHCl_3$) fumigation followed by extraction, the other immediately extracted. Fumigated samples were placed in an evacuated desiccator with 40 mL ethanol-free $CHCl_3$ for 72 hrs. The decrease in partial pressure from the vacuum causes $CHCl_3$ to evaporate and the $CHCl_3$-laden atmosphere fumigates the soil samples. Residual atmospheric and sample-bound $CHCl_3$ was removed by repeatedly de-pressurizing desiccator 5 times. Dissolved organic carbon (DOC) was extracted with a 1:2 and 1:4 ratio of soil:extract solution for mineral and peat soils respectively, using 0.5 M potassium sulfate (K_2SO_4) in a shaker for 1 hr. Sample solutions were then filtered via vacuum filtration through Whatmann #2 filter paper and frozen until analysis. The Natural Resource Analytical Laboratory (NRAL) analyzed samples for total organic carbon (TOC) content using a Shimadzu TOC-V/TN instrument (Mandel Scientific Company Inc., ON, Canada) (Voroney et al. 2006).

Quantification of pH was completed using a 0.01 M $CaCl_2$ solution. Air-dried soil:$CaCl_2$ solution ratio was measured to 1:2 for mineral and 1:4 for organic soils, shaken for 30 minutes and centrifuged for 5 minutes (Kalra and Maynard 1991).

2.2.5 - Statistical Analyses

Using R statistical software, a series of t-tests with Tukey HSD adjustment for multiple comparisons was used to analyze the effect of soil type and treatment on available macronutrients, pH, MB-C, basal respiration and metabolic quotient (qCO_2). Non-metric multidimensional scaling (NMS) was used to display nutrient data in ordination space using PC-ORD v. 6.0 (MjM Software Design, Gleneden Beach, Oregon, USA). Points in close proximity on bi-plots share greater similarity than those far apart. Multiple response permutation procedures (MRPP) is a non-parametric test to assess statistical differences within and between a priori groupings that was used to compare dissimilarities between groups in ordination space. Reported statistics in this analysis include the P-value indicating statistical significance; T-value signifying the strength of the difference between groups with more negative values being more different; and the A-value representing the variation within groups where 1 indicates no variation and 0 indicates completely random associations (Zimmerman et al. 1985). Ordinations were run several times to ensure that low stress outcomes were consistently achieved, and to avoid measuring a local minima instead of the experimental minima.

2.3 - Results

2.3.1 - Biological Measurements

Microbial biomass-carbon and basal respiration were greatest in natural Organic soils, followed by PM, natural Luvisol and finally FFM (Figure 2.2, Figure 2.3). Optimized temperature and water content may have created a pulse of activity that the pre-incubation did not eliminate. Natural soils, on average, had a greater qCO_2 than did reclaimed ($P = 0.0067$). A trend of increased qCO_2 was noted in all char and burnt treatments (Figure 2.4).

2.3.2 - Chemical analyses

The Luvisol expressed an increased pH from 4.99 to 6.03 following wildfire ($P = 0.0449$), however this trend was not noticed in natural Organic soil. The natural Luvisol had lower pH than did organic soils ($P = 0.0008$); while in reclaimed soils FFM was greater than PM at 6.77 and 5.75, respectively ($P < 0.0001$). Charcoal and fertilizer amendments did not significantly affect soil pH (Table 2.1).

The many iterations (250) and low stress value in the final solution of the NMS ordination validate this procedure. Additionally, this was supported by large T statistics with corresponding significance at $\alpha = 0.05$ between multiple groupings (Figure 2.5; Table 2.2). Multiple response permutation procedures revealed significant differences in nutrient availability in ordination space due to soil type and fertilization effects; however no statistical difference was apparent due to wildfire or charcoal treatments. Most of the variation was explained on axis 1 (87 %), which is also the separation between reclaimed and natural sites. To a lesser extent, fertilized versus unfertilized and FFM versus PM also formed significantly different groups. Correlations with this axis include NH_4^+ - N ($r^2 = 0.667$), P ($r^2 = 0.739$), K ($r^2 = 0.897$) and S ($r^2 = 0.880$). Greater concentrations of NO_3^- in fertilized soils are correlated with axis 2 ($r^2 = 0.564$) which was associated with fertilized treatments.

Individual analysis of macro-nutrients revealed further differences attributable to treatment effects. Luvisolic soil showed a trend of increased total inorganic N (TIN) availability following wildfire, although this was not statistically significant. Natural soils had similar proportions of NO_3^- to NH_4^+ while TIN in reclaimed soils was predominantly NO_3^- (Figure 2.6). Natural soils tended to have greater TIN availability than unfertilized reclaimed soils, but not significantly; and fertilized soils were orders of magnitude greater than unfertilized ($P < 0.0001$; Figure 2.7). Phosphorous availability increased following wildfire in both upland and lowland natural sites ($P < 0.0001$) and was greater in natural soils than in reclaimed ($P < 0.0001$; Figure 2.8). Wildfire only showed a trend of increased potassium availability in upland forest soils ($P = 0.0751$) and was greater in natural soil than reclaimed ($P < 0.0001$; Figure 2.9). Sulfur availability was greatest in reclaimed soils ($P < 0.0001$), most of which in PM ($P < 0.0001$; Figure 2.10). Fertilization appeared to decrease S availability in PM, despite S being included in the amendment.

2.4 - Discussion

2.4.1 - Soil Biological Measurements

Microbial biomass-carbon was greatest in organic soils likely due to the laboratory incubation for basal respiration where increased temperature and optimal water content were maintained throughout the incubation (Figure 2.2). In another study where field fresh soil samples were measured for MB-C, natural benchmark soils were significantly greater than reclaimed treatments and FFM was greater than PM (McMillan et al. 2007). This is supported by a meta-analysis study of other forest disturbances (fire, harvesting, storm, insect outbreaks and pathogens) where MB-C was decreased by an average of 29.4 % (Holden and Treseder 2013).

Despite having little effect on available nutrients in reclaimed soils, charcoal did appear to increase basal respiration and consequently metabolic quotient in FFM, similar to the trend presented in wildfire disturbance. Wardle and Ghani (1995) suggest that metabolic quotient increasers with disturbance due to decreased microbial inefficiency. The trend found in my data corroborates this hypothesis in FFM and natural upland soils (Figure 2.4). Soil samples were thoroughly mixed during sampling, transport and preparation; as a result charcoal originally on the soil surface was incorporated into the soil sample, creating greater potential for measuring treatment effects during incubation. Charcoal likely presented a recalcitrant carbon substrate to microbial communities, which has been shown to increase metabolic quotient as a result of decomposition inefficiency and potential nutrient stresses (Wardle et al. 1995). Charcoal did not have a treatment effect in PM likely due to a greater microbial biomass than FFM, which could have overruled any apparent effect of charcoal on respiration or qCO_2.

2.4.2 - Nutrients

Soil disturbance breaks down soil structure and exposes soil particles to higher oxygen concentration, increasing soil C bioavailability resulting in drastically altered nutrient conditions (DeBusk et al. 2005). This study represents this in ordination space where most of the variation is explained on axis 1 (87.0%), which is largely the separation between reclaimed and natural analogues. Additionally, nutrient profiles were significantly different between organic and mineral soils in natural analogues (MRPP $P = 0.0291$), and reclaimed soils (MRPP $P = 0.0003$; Figure 2.5). Sulfur was highly correlated with axis 1 ($R^2 = 0.880$) while P, K and NH_4^+ with natural reference sites ($R^2 = 0.739, 0.897, 0.667$, respectively). Total N and NO_3^- were correlated with separation on axis 2, and mostly associated with the fertilized PM treatments ($R^2 = 0.564, 0.654$, respectively). The only amendment with an apparent effect is fertilization, which created distinct groups in reclaimed treatments (MRPP $P = 0.0001$). Data collected from anion and cation exchange resins provide a relevant account of nutrients available to plant roots since they remove many environmental covariates being an in-situ measurement experiencing identical environments. Criticisms of this method are mostly attributed to root competition as the rhizosphere encroaches on exchange membranes. Additionally, this measurement may be an understatement of the nutrient availability experienced by plants since mycorrhizal associations would further contribute to nutrient acquisition through the exchange of inorganic and potentially organic species of nutrients (Näsholm et al. 2009).

2.4.2.1 - Nitrogen

As a major natural disturbance, fire exacts physical and chemical changes to forest soils in the boreal forest, providing a range of nutrient regimes to which native species have adapted. Total inorganic N availability following fire disturbance on the Horizon Project was greater than the undisturbed natural Luvisol in the year following wildfire disturbance, despite vigorous plant regeneration (Figure 2.7). This is supported by evidence in the literature of increased N availability following fire in upland forested soils (Rokich et al. 2000, Choromanska and DeLuca 2001, Ball et al. 2010). Lowland organic soils demonstrated no N response to fire which may be due to denitrification or leaching losses since these soils were saturated (Neary et al. 1999).

When compared to natural analogues, un-amended reclaimed soils possessed slightly less plant-available N, yet differences are not significant due to high spatial variability and low sample sizes. The fertilization amendment displayed the greatest influence on TIN availability, especially on PM. Peat mix was could have had more N availability due to a lack of vegetative cover in the second season. The large variability on fertilized treatments was likely due to the nature of the application and size of sample plots. Fertilizer was aerially broadcast which lacks precision. Upon visual inspection of plots the day following application, no fertilizer pellets were visible on two of the FFM sites. When analyzed, these sites had similar nutrient values to unfertilized treatments.

The natural Luvisol contained similar proportions of NH_4^+ and NO_3^-. Inorganic fertilizer amendments typically used in the initial growing seasons following reclamation provide up to 2 orders of magnitude greater available N than unfertilized and fire-disturbed or natural reference sites. These results suggest that N fertilizer application was applied in excess of plant nutrient requirements. Virtually all available N was present as NO_3^-, indicating that soil microbial communities nitrified NH_4^+ to NO_3^- within 30 days without subsequent plant uptake. This represents a disconnect between mineralization, nitrification and plant uptake in disturbed soil (MacKenzie and Quideau 2010).

Some studies in boreal regions have demonstrated that adding a black carbon amendment to soils enhances microbial activity (Wardle et al. 2008) and indirectly increases N availability by sorbing phenolic compounds that inhibit N mineralization and nitrification in boreal soils (DeLuca et al. 2002). Alternately, adding black carbon to reclaimed soils can reduce measured N mineralization, likely due to its sorptive capacities (MacKenzie et al. 2014). In this study, adding charcoal only showed a slight trend in reducing TIN availability on reclaimed treatments without fertilizer amendment. The additional microbial activity measured by qCO_2 could be indicative of microbial N immobilization. I proposed three reasons to explain why charcoal amendment did not significantly affect N availability. Firstly, charcoal application rate may have underestimated actual loads in boreal soils since the value obtained from the literature was based on manual separation, and would have excluded particles too small to separate by hand. Secondly, broadcasting was not effective at establishing contact between with bulk soil, so soil water was likely unable to interact with charcoal. Vegetation was already present and precluded any possibility of incorporating charcoal into the bulk soil. Finally, one season may not be sufficient time to illicit measureable responses.

2.4.2.2 - Phosphorous

Similar to N, P availability increased following wildfire disturbance (Figure 2.8). Additional soil P was likely contributed from ash since P does not combust and tends to accumulate in the surface horizons following fire (Neff et al. 2005). Reclaimed sites had less available P than burnt natural forest stands, which were greater than all other treatments ($P < 0.0001$). Despite being included in the fertilizer mix, P availability on fertilized plots does not differ from unfertilized, indicating that excess N may have indirectly affected P availability by stimulating plant and microbial growth, coincidentally increasing immobilization. Higher concentrations of aluminum (Al), calcium (Ca), magnesium (Mg) and iron (Fe) in reclaimed soils ($P = 0.0063$, $P < 0.0001$, $P < 0.0001$, $P = 0.0002$, respectively) could lead to P complexation and thus immobilization across a range of pH values (Vetterlein et al. 1999, Wang et al. 2012). No difference was detected in P availability in between FFM and PM or with charcoal additions. Biochar has been found to increase P availability in some soils; however this is likely due to pyrolysis and additions from ash or mineralization from increased microbial biomass on old charcoal particles (Liang et al. 2010, Qian et al. 2013). The charcoal collected in this experiment was the result of open combustion and collected without the ash component and was only in place for a single growing season.

2.4.2.3 - Potassium

Potassium is important for a number of plant functions at the cellular level and greater including protein synthesis, enzyme activation and stomata opening and closing, and regulation of turgor pressure in cells (Leigh and Wyn Jones 1984, MacRobbie 1998). Wildfire has been shown to increase water-soluble and extractable K soon after burning, however in the following year, decreases to below pre-disturbance concentrations (Smith 1970). No significant differences in available K were observed with wildfire, amendment or soil type, however there was significantly less in reclaimed sites compared with reference sites (Figure 2.9). This is likely due to a concentration effect of K in upper horizons with time (Smith 1970). Potassium is highly mobile in soils however it accumulates at the soil surface as it is collected and vertically transported by vascular plants with a final fate in organic litter layers. Vegetation depletes K at depth and concentrates it near the soil surface from litter accumulation and decomposition. Potassium is slowly released through weathering of primary minerals and requires significant time to become available. As soils are salvaged, surficial horizons are admixed with deeper, nutrient depleted horizons thus reducing concentrations in reclaimed soils. This leaves potential for K losses if vegetation is not established sufficiently to immobilize K before it is leached below the rooting zone. As litter layers develop, increased K concentrations in surface horizons is hypothesized.

2.4.2.4 - Sulfur

Sulfur, Mg and Ca availability were high in reclaimed soils but low in natural soils ($P < 0.0001$). Plant uptake could be a determining factor since vegetation was most prominent on natural sites and least on unfertilized PM (which had the greatest S availability). During vegetation removal and direct placement of reclamation materials, a disconnect in soil nutrient cycling is created. With sufficient precipitation and a lack of immobilization pathways, nutrients could be leached from soil rooting zones. Soil pH was only significantly different between soil types and not between reclaimed and natural analogues and therefore not likely a determining factor controlling availability. Atmospheric deposition of S from vehicle traffic may play a role since RA-1 is immediately adjacent to, and downwind from the mine pit; however this relationship requires further investigation. There was a trend in decreased S availability with charcoal application; however this trend was not significant. Most of the charcoal amendment remained on the soil surface therefore charcoal would react with elements as they enter the soil system from above. This perhaps explains reduced sulfur availability since atmospheric deposition would first react with surficial charcoal deposits prior to percolation down into the zone of influence of the ion exchange membranes. Additionally, SO_4^- is highly mobile in soils and the greater water content in unfertilized PM (likely due to less evapotranspiration) could explain the observed results.

2.4.3 - Vegetation

When applied to RA-1, the barley nurse-crop benefitted from fertilization on both PM and FFM reclamation materials and was the dominant vegetative cover in 2011 (Figure 6.1, Figure 6.2). Thick barley cover in the first year on fertilized treatments may have out-competed native species in the soil propagule bank, which are slower to establish (Sloan and Jacobs 2013, Pinno and Errington 2015). This explains the lack of vegetation observed on the fertilized PM treatment in 2012, and the extensive cover of weedy species on the fertilized FFM treatment (Table 6.2). Comparatively, the unfertilized treatments had much less vegetative cover observed on walk-throughs and did not induce increases in seedling growth. Vegetation data collected and analyzed by the Canadian Forest Service for CNRL indicates that the factor predominantly separating vegetative communities in a non-metric multidimensional scaling (NMS) ordination is soil type on a primary gradient, with fertilization and coarse woody debris application differentiation on a secondary gradient (Figure 6.3) (Canadian Natural Resources Ltd. 2013). Vegetation communities in undisturbed mature stands shared the greatest similarity with stands disturbed by wildfire, followed by FFM and lastly PM. Fertilizer amendment contributed to community type distribution, yet coarse woody debris bridged the gap between fertilized and unfertilized plots. Understandably, FFM supported more species found in natural upland sites than did PM which is attributable to the inherent propagule bank found in FFM (Table 6.2).

2.5 - Conclusion

These results indicate that the soils used in reclamation are altered in their nutrient profiles from salvage to application, and that amendments used do not recreate nutrient conditions similar to the variation experienced in natural upland boreal forest soils. Fertilizer amendments targeting N show increases in plant growth, but these might be applied in excess on RA-1. Charcoal, or other black-carbon materials likely have greater potential to illicit effects in a/b ecosites where the benefits of water holding capacity, cation exchange capacity (CEC), and microbial habitat would be most apparent – benefits that rich, mesic ecosites with fine textured soils inherently possess. The attempt to reduce excess fertility with broadcast charcoal resulted in no difference in nutrient availability. A broadcast rate of 2,000 kg ha^{-1} may have been too miniscule to induce a measureable effect. Additionally, incorporating charcoal into soil may have provided greater reactive surface area contact with soil particles and reducing amendment losses from wind and water erosion. Some particles could have been translocated deeper into the soil profile through the many small cracks present in surface soils however this was not measured.

The contribution of forest floor litter layers to overall soil nutrition was not measured in this study. This is a difficult component to measure and compare with reclaimed sites which have yet to develop litter layers. These results should be interpreted within this context since plant nutrition in upland boreal soils is largely derived from the intense exploration of the mineral-organic interface by plant roots and mycorrhizal hyphae. In addition to this, future studies should address the impact of coarse woody debris applications on soil microbial communities and nutrient profiles. Application of CWD brings the added benefit of microsites to establishing vegetation, impacting soil moisture, temperature and nutrient status (Alberta Environment and Water 2012). An ongoing study by the Canadian Forest Service found CWD amendments bridge the gap between fertilized and unfertilized reclamation treatments (Figure 6.3). Assessing microbial community structure and function would elucidate further discrepancies in soil quality between topsoil types and amendments used on RA-1.

Tables and Figures

Table 2.1 – Basic soil characteristics from natural benchmarks and reclaimed soil types. Carbon, electrical conductivity (EC), sodium adsorption ratio (SAR) and bulk density (D_b) taken from Pinno and Errington (2015).

Soil type	Treatment	pH		Carbon (%) *	EC (mS cm^{-1}) *	SAR *	D_b (g cm^{-1}) *
Organic	Unburnt	6.74	(0.38)				
	Burnt	6.45	(0.30)				
Luvisolic	Unburnt	4.99	(0.56)				
	Burnt	6.03	(0.27)				
FFM	-	6.82	(0.16)	2.1	1.43	1.35	1.18
	Char.	6.89	(0.16)				
	Fert.	6.60	(0.21)	5.4	1.15	0.68	0.82
	Fert. + Char.	6.75	(0.08)				
PM	-	5.80	(0.62)	15.0	4.18	2.5	0.94
	Char.	5.61	(0.43)				
	Fert.	5.98	(0.23)	15.4	3.76	2.79	0.75
	Fert. + Char.	5.62	(0.45)				

Table 2.2 – Multiple response permutation procedure statistics of non-metric multidimensional scaling ordination of available nutrients adsorbed to PRS™ probes over a 6 week burial.

	Groups Compared			T	A	*P*
Soil Type	Natural Luvisol	-	Natural Organic	-4.52	0.15	0.0029
	Natural Luvisol	-	PM	-12.43	0.42	< 0.0001
	Natural Luvisol	-	FFM	-12.15	0.42	< 0.0001
	Natural Organic	-	PM	-9.62	0.38	< 0.0001
	Natural Organic	-	FFM	-9.64	0.35	< 0.0001
	FFM	-	PM	-6.38	0.14	0.0003
Treatment	Burn	-	Unburnt	-0.03	0.00	0.3793
	None	-	Charcoal	0.78	-0.02	0.7775
	Unfertilized	-	Fertilized	-7.08	0.15	0.0001

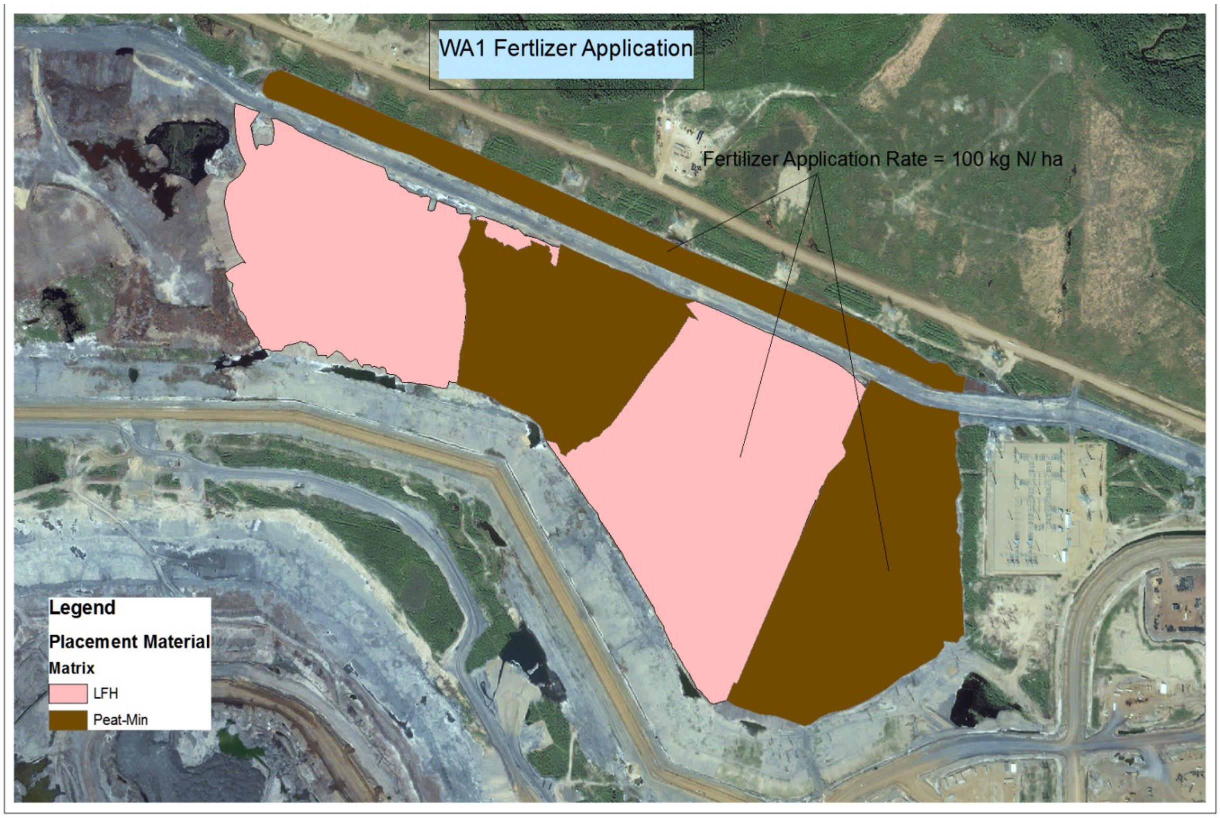

Figure 2.1 – Satellite image of Reclamation Area-1 (RA-1) soil types with unfertilized and fertilized treatments (22.9-9.1-9.1-9.1 N-P-K-S blend applied at a rate of 100 kg – N ha^{-1}) (Canadian Natural Resources Ltd. 2013).

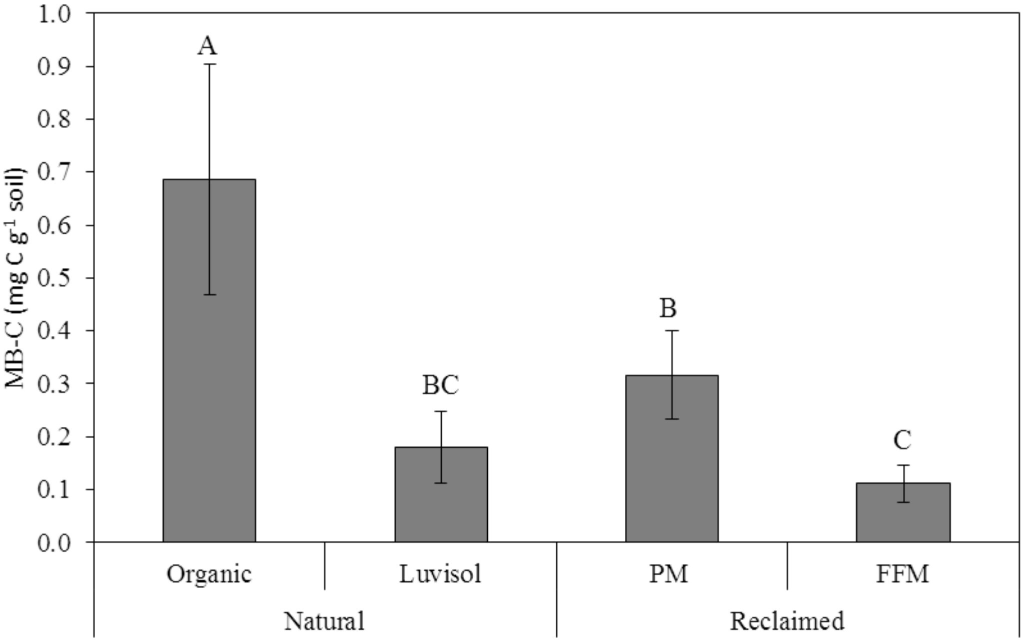

Figure 2.2 – Microbial biomass-carbon (MB-C) following lab incubation and basal respiration measurements for natural benchmark (n=6) and reclaimed soils (n=12) on RA-1. Means reported with error bars depicting standard error.

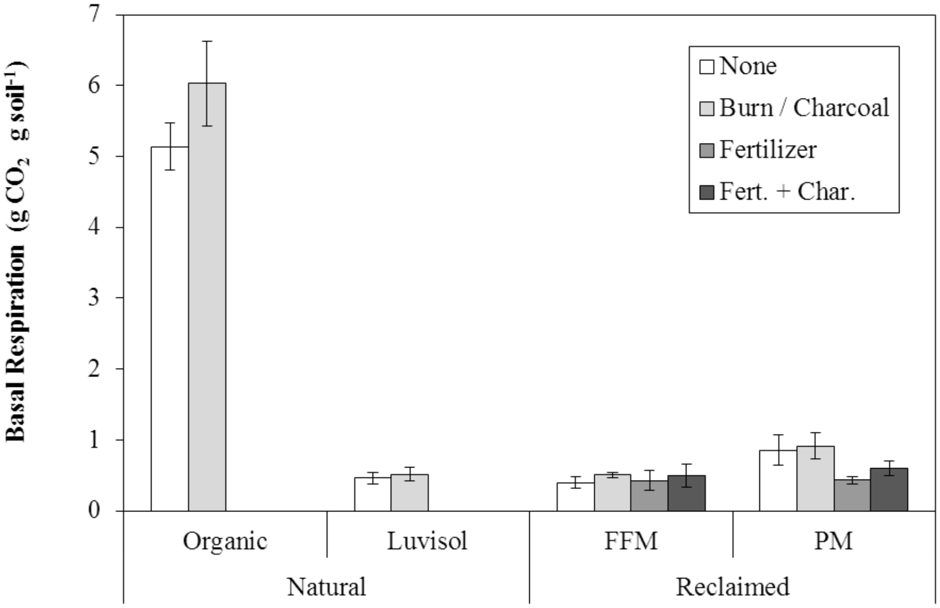

Figure 2.3 – Basal (heterotrophic) respiration from natural benchmark and reclaimed soil samples measured by alkali-trap method after over a 7 day incubation period at 22 °C. Means reported with error bars depicting standard error (n=3).

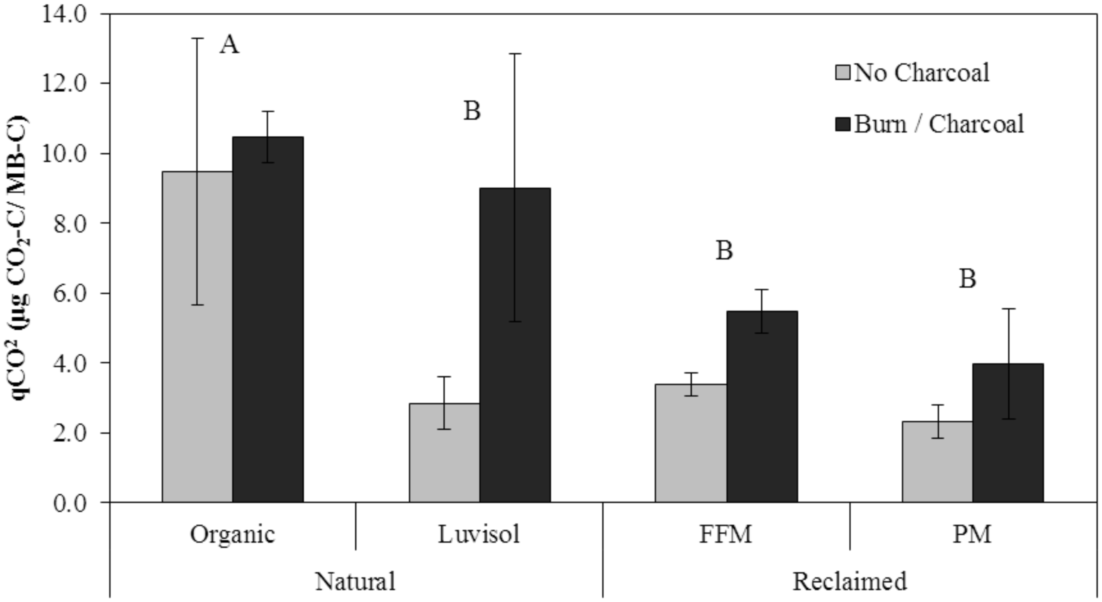

Figure 2.4 – **Metabolic quotient (qCO_2) indicates the amount of respired CO_2-C per unit of MB-C for natural benchmark (n=3) and reclaimed (n=6) soils. Means reported with error bars depicting standard error.**

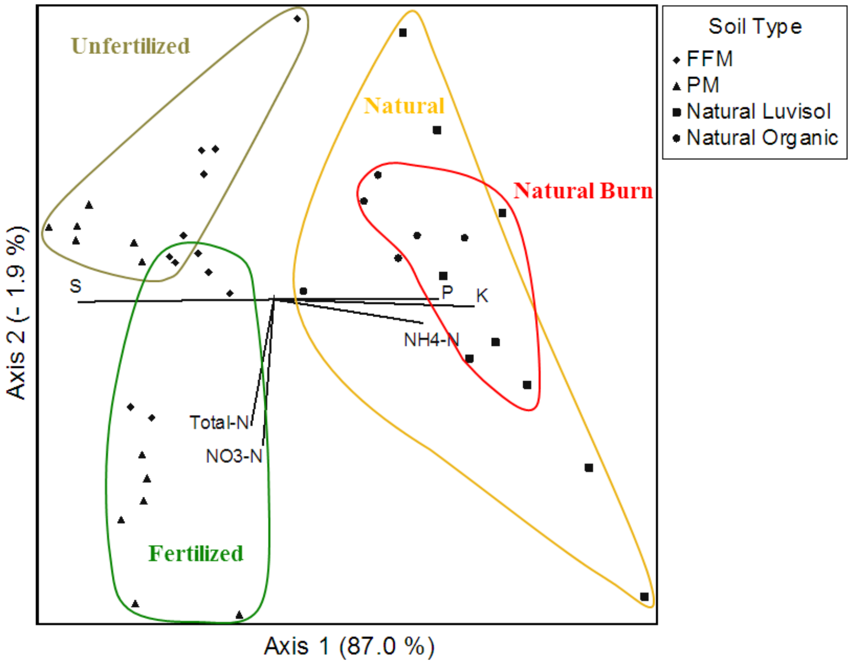

Figure 2.5 – **Non-metric multidimensional scaling ordination plot of available macro- and micronutrients adsorbed onto ion exchange membranes after a 6 week burial period, grouped by soil type and fertilizer treatment; vectors ($r^2 > 0.40$), final stress of 5.5 %.**

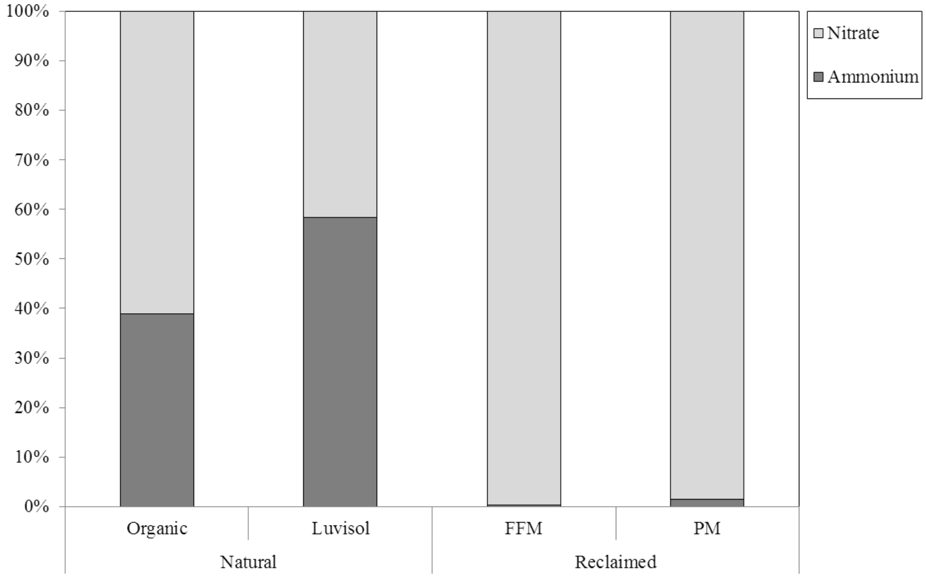

Figure 2.6 – Proportion of available nitrate (NO_3^-) and ammonium (NH_4^+) to total inorganic N adsorbed on ionic exchange membranes after a 6 week burial period in natural benchmark (n=6) and reclaimed (n=12) soils.

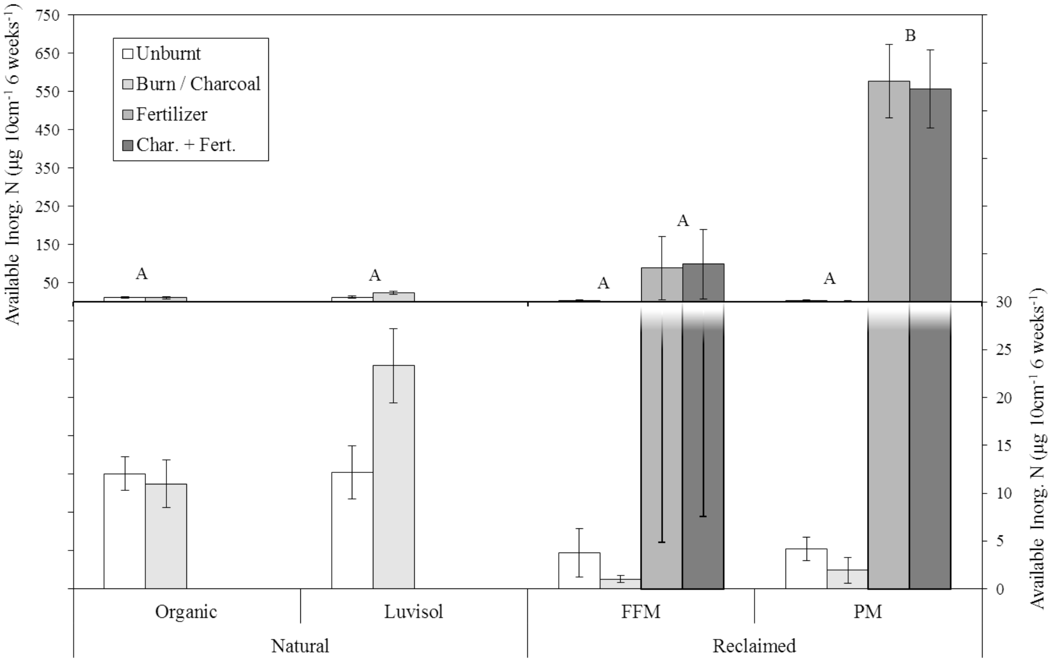

Figure 2.7 – Total inorganic nitrogen (TIN) adsorbed to ion exchange membranes after a 6 week burial period for natural benchmark and reclaimed soils; graph split into two scales to represent contributions from fertilized and unfertilized plots. Means reported with error bars depicting standard error (n=3).

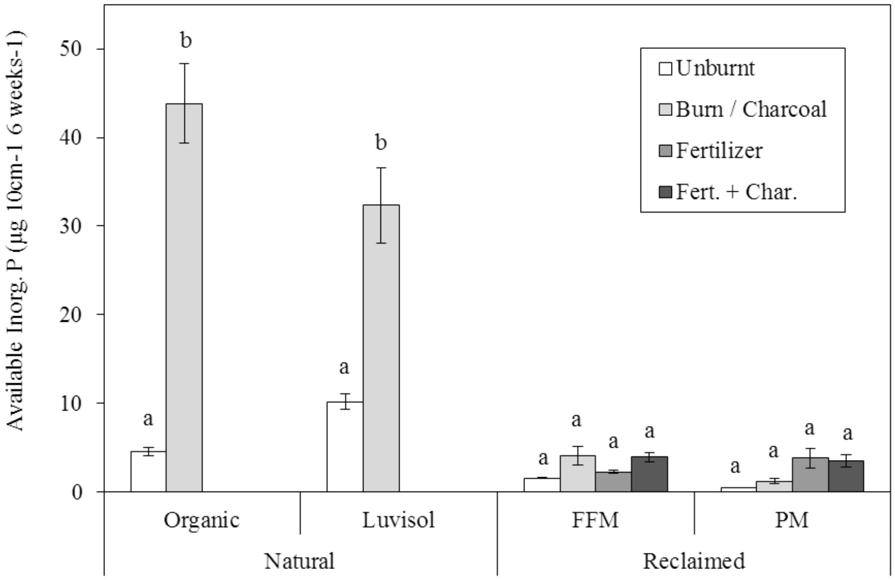

Figure 2.8 – Available P adsorbed to ionic exchange membranes after a 6 week burial period for natural benchmark and reclaimed soils; significance at *P* < 0.05 denoted by an asterix. Means reported with error bars depicting standard error (n=3).

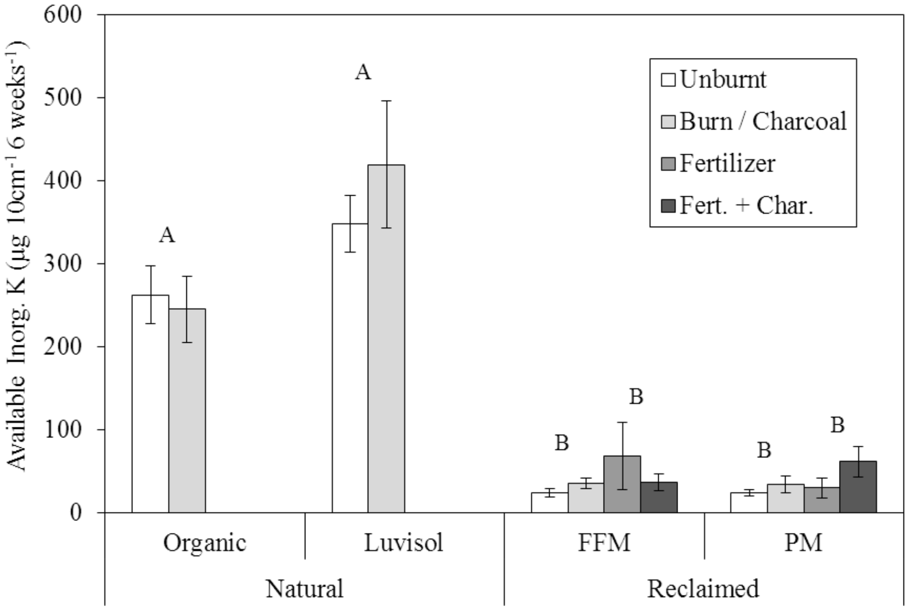

Figure 2.9 – Available K adsorbed to ionic exchange membranes after a 6 week burial period for natural benchmark and reclaimed soils. Means reported with error bars depicting standard error (n=3).

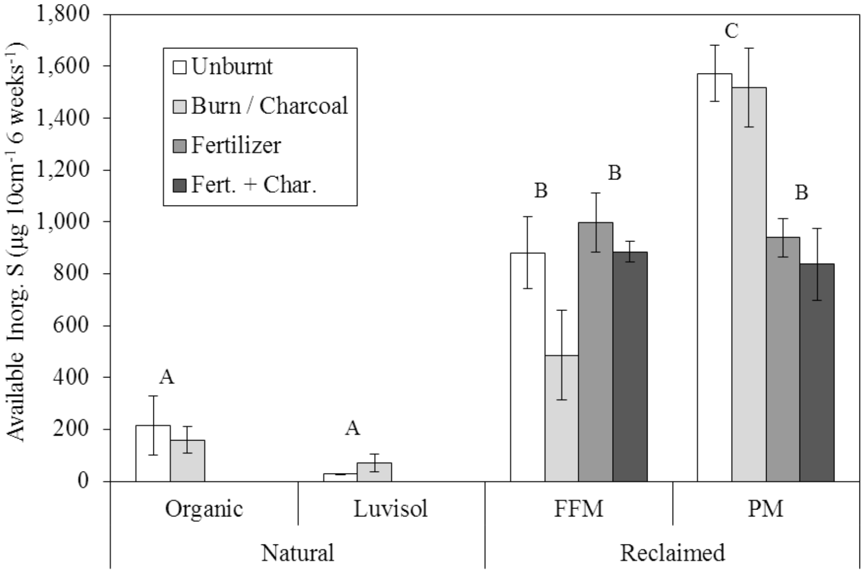

Figure 2.10 – Available S adsorbed to ionic exchange membranes after a 6 week burial period for natural benchmark and reclaimed soils. Means reported with error bars depicting standard error (n=3).

3.0 Implications of topsoil application depths to microbial communities and nutrient availability

3.1 - Introduction

The expanse of surface-minable oil sand underlies a variety of ecosystem types, supporting a range of productivity levels. Following mining disturbances, some of the more challenging areas to reclaim are a/b ecosites due to low nutrient and water retention capacities (Beckingham and Archibald 1996). Several mines are located in areas with coarse textured soils and overburden, and relatively shallow bituminous ore bodies. No such site has yet received government certification of reclamation. These areas could take longer to attain certification due to inherent nutrient and water limitations. It would therefore be useful to develop tools that accurately assess soil quality at early stages of reclamation in order to identify and address potential problems.

Comprehensive reclamation prescriptions can expedite ecosystem recovery by shifting the system from primary to secondary succession. Several recent studies have demonstrated that FFM outperforms PM in reclaiming upland ecosystems in the AOSR (Mackenzie and Naeth 2010, Hahn 2012, MacKenzie and Quideau 2012). This is intuitive since FFM originates from natural upland environments and retains many physical, chemical and biological characteristics when employed as a reclamation material. However, PM is the most abundant topsoil material available for reclamation. Until recently, regulatory requirements did not mandate FFM salvage, so PM was almost exclusively salvaged and stockpiled for future use in mine closure. Industry is faced with a legacy of this material which necessitates the assessment of PM in a variety of landscape positions and planned ecosites. Comparing soil biological qualities between PM and

FFM may indicate optimal uses for PM and identify shortfalls that could require further management.

In addition to the difficulties inherent to coarse textured soil supporting vegetation establishment, operational constraints must be considered when developing reclamation prescriptions. Ideally, topsoils salvaged from mine advancements are immediately transported and applied (direct placement) to reclamation areas in an effort to minimize losses to soil propagule viability, nutrient stores and speciation and microbial community structure and function (MacKenzie 2013). Material handling is costly and should be minimized wherever possible. This provides economic incentive for direct placement instead of stockpiling. Moreover, soils available for reclamation are in short supply, especially FFM. A goal of maximizing material handling efficiency and reclamation outcomes depends on the amount of materials applied to reclaimed sites. It is therefore crucial to understand benefits of coversoil placement depths to provide sufficient materials for forest growth. Current depth requirements are predominantly based on providing a minimum effective layer to isolate plant roots from potentially deleterious underlying media (typically LOS or highly saline materials) and to provide vegetation with an adequate supply of water and nutrients (Alberta Environment 2010). Most of the latest research conducted on placement depth of reclaimed soils in the AOSR has been based on success of plant recruitment from soil propagule banks (Mackenzie and Naeth 2010), tree rooting depths (Jung et al. 2014) and soil water movement (Naeth et al. 2011). Little consideration has been given to soil biology and its relevance to reclamation. The LCCS has been a tool used to assess sites for reclamation and contributes to topsoil application depth decisions (CEMA 2006), but this approach judges sites based on agriculturally-derived methods of soil physical and chemical characterization relating to optimal conditions for vegetative productivity and not necessarily re-establishing community diversity. Lately, soil biological measurements are receiving more attention in mine site reclamation assessments globally (Harris 2003, Mukhopadhyay et al. 2014), and microbial community responses to disturbance in the AOSR (Swallow et al. 2009, Dimitriu et al. 2010, MacKenzie and Quideau 2010, Sorenson et al. 2011). Benchmark studies in conjunction with chronosequence sampling may help create better models of reclamation trajectory and landscape assessments. Incorporating biological measurements into analyses of topsoil placement depths could provide a sensitive measure into material suitability immediately pursuant to site reclamation. To my knowledge, no study has yet been published on microbial community composition and function in coarse textured reclamation materials in the AOSR.

This research was located on the Aurora Soil Capping Study (ASCS) – an operational scale experiment designed to assess reclamation soil types and application depths in a multidisciplinary and collaborative study. My objective was to evaluate treatments on the ASCS in the context of soil microbiota to: 1) determine optimal soil type; and, 2) assess whether shallow or deep topsoil application depths create similar biogeochemistry to a benchmark soil. These questions were addressed by comparing two application depths of PM and FFM to a control with no topsoil, and a benchmark soil where a jackpine (*Pinus banksiana* Lamb.) stand had re-established following timber harvesting. Two subsoil types under deep topsoil applications were also compared to determine potential benefits to establishing vegetation. This study is intended to contribute to other studies on the site, which will culminate to provide a comprehensive review of reclaiming dry ecosites with directly placed soils.

Upon visual inspection of the ASCS, FFM appeared radically different due to extensive vegetative cover in comparison to PM. Additionally, the provenance of each material led us to hypothesize that soil nutrient profiles and microbial community structure, function, and respiration in FFM would be most similar to Harvest. I also expected to find greater total microbial biomass and diversity and nutrient profiles closer to the Harvest analogue with deep placements since subsoil materials in shallow placements lack organic matter and nutrients characteristic of surface soils. Operationally, I predicted that benefits of deep topsoil application would decrease with increasing depth. Deep applications may be considered excessive and not worth the cost of placement or loss of the resource. Due to disturbance, I also expected to encounter imbalances and disproportionate accumulations or depletions of important soil nutrients due to interrupted linkages in biological cycling (MacKenzie and Quideau 2010). With increasing depth, I expected differences in nutrient speciation due to changes in redox conditions, temperature and moisture availability. Lastly, I expected the blended B/C material to be more similar than the conventional subsoil (salvage from below 1 m) to natural conditions since it should have experienced pedogenic influences prior to soil salvage.

3.2 - Methods

3.2.1 - Study Area

Syncrude Canada's ASCS is located at the Aurora North Mine, 75 kilometers north of Fort McMurray, Alberta. Upland soils naturally present on this lease are predominantly coarse textured and mostly classified under the Brunisolic soil order (Haynes 1998, NorthWind Land Resources Inc. 2013). These soils support dry a/b ecosites consisting of mixed and pure stands of jackpine (*Pinus banksiana* Lamb.), trembling aspen (*Populus tremuloides* Michx.) and white spruce (*Picea glauca* Moench) (Cumulative Environmental Management Association 2006).

Upland soils are interspersed with saturated Organic and Gleysolic soils in lowlands, supporting black spruce (Picea mariana Mill.), birch (Betula spp.) and tamarak (Larix laricina Du Roi).

The ASCS was created to evaluate placement depths and topsoil types using locally available coarse textured materials present in this region. The study is situated at an elevation of ~ 350 MASL on a decommissioned LOS overburden dump. In 2013, on-site weather stations measured annual rainfall of 319.6 mm. Average annual temperature was 2.5 °C with daily highs and lows during the sampling period reaching 33.7 °C and 6.2 °C, respectively (O'Kane Consultants 2014). Twelve treatments were built to assess placement depths and material suitability for reclamation in order to improve on best management practices. Treatments in triplicate were randomized across the 36 ha study area with each experimental unit ~ 1 ha (Figure 3.1). Sample sizes were the minimum number required for statistical comparison (n = 3) due to economic limitations resulting from the size of the experiment. However, I expected that variability on reclaimed sites would largely have been homogenized from soil salvage and placement activities. Reclamation materials were applied using haul trucks and spread to desired thicknesses with bull dozers. Precision was achieved using GPS guided dozers back-blading materials to uniformly apply materials (Table 3.2).

Microtopographic variability was minimized to reduce possible confounding effects of introduced with microsite creation. Slopes on experimental units were < 2.5 % grade and were drained by networks of ephemeral swales capped with PM. These swales drain the site from north to south. Jackpine (*Pinus banksiana* Lamb.), trembling aspen (*Populous tremuloides* Michx.) and white spruce (*Picea glauca* Moench) 2 year old nursery stock were planted in the spring following soil placement.

Northwind Land Resources Inc. (NWLR) assembled a report of baseline soil information collected in the first year following soil placement. Although sampling protocol differed, data were not duplicated in this research. This report provided information on organic carbon % (OC), carbon to nitrogen ratio (C:N) and particle size distribution. Initial characterization of reclamation topsoil in 2012 identified mineral soils used for reclamation on the ASCS as having a sand texture (Table 3.1) (NorthWind Land Resources Inc. 2013). Peat mix is typically a 60:40 (v/v) mix of peat and surrounding mineral material, however no texture was documented in the NWLR report and visual inspections of PM soil samples revealed no mineral component. Organic carbon was greatest in PM, followed by FFM and finally SS. Forest Floor Mix C:N was greater than PM and SS. It is worth noting that dry weight nitrogen in SS was largely at or below detection limit and therefore may be overestimated.

3.2.2 – Field Work

Data were collected during the 2013 growing season. In-situ measurements were made wherever possible since physical manipulation of soil during sampling increases oxygen exposure and changes temperature and moisture conditions, therefore creating potential to distort conditions to which microbial and vegetative communities are exposed. Soil plots were established and instrumented June 26 - 29, 2013. Treatments 1, 2, 3, 7, 8, 10, 12 and a ~ 15 year harvested location were selected for analysis. These provide a comparison of subsoil (SS) salvaged from > 1 m depth control where no topsoil was applied (Control), Shallow and Deep placements of PM and FFM over SS, Deep PM and FFM underlain by B/C blend (B/C) originating from a salvaged composite of Brunisolic B and C horizons, and a recovering anthropogenically disturbed site (Harvest) (Figure 3.2). Harvest sites were selected based on close proximity to the Capping Study, ecosite similarity, ease of access, and aspect. Reclaimed sampling plots were randomly located by spacing 5 m south of the southeast corner of high density jackpine tree plots. Occasionally this was not possible due to instrumentation from other researchers, treatment boundaries and ATV trails. In such cases, a 5 m buffer was measured to minimize edge effects. Trees planted on reclaimed soil plots included a mixture of jackpine, trembling aspen and white spruce at 2,000 stems ha-1. Plot locations were considered acceptable if actual topsoil placement depths fell within ± 4 cm of the desired treatment depth, otherwise a new location was chosen (with the exception of cell 30 where the closest depth was 12 cm less than the desired depth) (Table 3.2). Each plot contained one pit dug to a maximum depth of 40 cm with the dimensions of a 20 liter pail. Excavated soil was segregated on tarps based on material types and depths, which prevented admixing of soil layers. Perforated polyethylene 20 liter pails were filled with the excavated material in the reverser order that it was removed, and the pails were sunk into the pits after instrumentation was installed (Figure 6.5). This system facilitated sample retrieval, minimized disturbance to soil gas and liquid fluxes and maintained pit integrity for future sampling. Digital photographs were taken from the north looking south to qualitatively document vegetation development (Figure 6.4). These were taken during setup and final sampling.

Charged anion and cation exchange membranes with 10 cm^2 surface area, known as plant root simulator (PRS™) probes (Western Ag Innovations Inc., Saskatoon, SK, Canada), captured plant-available nutrients at 5, 15 and 35 cm depths over a 57 day period from June – August (Figure 6.6). This method provides a more realistic approach to what plant roots experience as labile inorganic nutrients are measured in-situ with little disturbance and avoid synthetic extractants (Qian and Schoenau 2002). Sampling depths were chosen to ensure probes were inserted into the media immediately below substrate interfaces with a ± 5 cm buffer to allow for placement depth variability. Four subsamples were analyzed in composite from each depth. Probes were inserted horizontally with the ion exchange membranes oriented vertically to avoid pooling on membrane surfaces from vertical water flow. Probe placements were staggered such that probes at each depth did not align vertically with probes underneath thus reducing the likelihood of shallow-placed probes influencing moisture and nutrient dynamics of deeper probes. Upon retrieval, probes were bagged in composite, placed in a cooler with ice packs during transport, then refrigerated at 4 °C until shipment to Western Ag Innovations Inc. for analysis. Upon elution in 0.5 M HCl, analysis yielded nutrients adsorbed to membrane surfaces in µg 10 cm^{-2} 57 $days^{-1}$ for NO_3^- and NH_4^+ using a segmented flow Autoanalyzer III (Bran and Lubbe, Inc., Buffalo, NY); and for P, K, S, Ca, Mg, Mn, Al, Fe, Cu, Zn, B, Pb and Cd were measured using inductively-coupled plasma spectrometry (ICP), atomic adsorption spectrometry (AAS) and flame emission spectrometry (FES). Maximum adsorbance values are listed in Table 6.1.

One cell from each treatment was instrumented with an Em50 data logger, EC-5 Volumetric Water Content Sensors and RT-1 Soil Temperature Sensors (Decagon Devices, Inc., Pullman, WA, USA) to continuously log temperature and volumetric water content (VWC) at equivalent depths to PRS™ probes. Only VWC was measured at 35 cm due to limited available ports on data loggers. Measurements were taken in 15 minute intervals to capture diurnal variability. Temperature and VWC measurements were validated with hand-held probes from each pit on the final sampling date.

Soil respiration collars made from 12 cm lengths of 20 cm diameter PVC irrigation pipe were installed 1.5 m south of each pit. A minimum of 2 cm of the collars remained above ground to accommodate measuring equipment. Boundaries were marked around each plot using wooden stakes. These were offset from pits and collars by a minimum of 1 m due to concerns of the wood influencing localized microbial ecology. A LI-COR 8100 Infrared Gas Analyzer (IRGA) (LI-COR Biosciences, Lincoln, NE, USA) was used to measure CO_2 gas flux from each site during three sampling dates with a 2 week interval. This method provides insight to in-situ soil metabolic activity producing CO_2, which includes both autotrophic and heterotrophic respiration, and to a lesser extent chemical oxidation of carbonaceous compounds (Lundegårdh 1927, Bunt and Rovira 1954). Soil respiration was first measured one month after installation to reduce error introduced from disturbance during installation. Measurements were taken at different times during each sampling date to account for variability attributed to moisture and temperature differences experienced during diurnal fluctuations (Singh and Gupta 1977). Actual above-ground collar height was measured and input into software to adjust headspace calculations.

Soil sampling coincided with PRS probe removal from August 22 - 24, 2013. Samples were taken using a 6.35 × 30.48 cm split core sampler (AMS Inc., American Falls, ID, USA). Soil cores were partitioned based on 10 cm incremental depths to a maximum depth of 40 cm. Two cores were taken at each plot, hereafter referred to as core # 1 and core # 2. Actual topsoil placement depths were also measured at this time from pits. Soil samples for phospholipid fatty acid (PLFA) analysis were taken separately from cores to reduce the likelihood of contaminating deeper samples with material from above. A soil knife, washed with 70% ethanol in-between sampling, was used to collect these samples. Pit faces were scraped to bear a fresh, uncontaminated face for sampling at 5, 15 and 35 cm. All soil samples were placed in labeled Ziploc® freezer bags, transported in coolers with ice packs, and kept in an interim refrigerator prior to transport to the University of Alberta for storage in the 4 °C walk-in refrigerator in the

Natural Resource Analytical Laboratory (NRAL). Samples for PLFA analysis were immediately placed in a -80 °C super freezer upon arrival at the University of Alberta.

3.2.3 - Laboratory

Laboratory work was conducted in the Pyrogenic Ecosystem and Restoration Ecology Laboratory (PEREL) and the Soil Biogeochemical Laboratory at the University of Alberta, Canada. Weights of samples from both cores were averaged to determine bulk density. Samples from core #1 were manually passed through a 4 mm sieve to remove roots and bituminous aggregates. All core samples were kept at a temperature range of 1 – 4 °C until analysis. Field water content of samples was determined by drying 10 g of soil at 105°C for 24 hours (Kalra and Maynard 1991). Soil EC and pH were measured in 1:2 (mineral) and 1:4 (organic) ratios for air-dried soil:distilled water ratios, shaken for 30 minutes and centrifuged for 5 minutes.

3.2.3.1 - Water Holding Capacity

Subsamples from each substrate collected from core # 1 were used for determination of water holding capacity. Pressure plates and samples were saturated with distilled water for a minimum of 24 hours prior to the experiment. Some mineral samples required up to 72 hours to become saturated. Hydrophobic samples were excluded from analyses. Samples were then placed on pressure plates inserted into pressure chambers, sealed and pressurized for 24 hrs at 0.1 and 0.03 MPa, for FFM and PM respectively. Samples were removed, weighed, dried at 105 ^{0}C and weighed again to obtain maximum water holding capacity (Kalra and Maynard 1991).

3.2.3.2 - Basal Respiration

Basal respiration was measured to examine soil heterotrophic metabolic activity having similar temperature and VWC to daily highs observed from field sensors. The objective of using temperatures measured in the field was to recreate similar conditions and understand the contribution of each depth to total respiration. Average daily highs of 24.6, 20.4, 17.1 °C were measured from 5, 15, and 35 cm depths. Water content was adjusted to 60% water holding capacity since soils contained very little water due to collection at the end of the growing season. Un-sieved soils from core # 2 measured to 100g for mineral and 75g for peat were placed in 1 L mason jars. Jars were sealed and soil was pre-incubated for 7 days. Following pre-incubation, lids were removed for 30 min to remove accumulated CO_2. Evaporative water losses were returned prior to adding un-capped scintillation vials containing 20 mL of 1 M sodium hydroxide (NaOH) in samples from 5 and 15 cm depths, and 20 mL of 0.5 M NaOH in samples from 35 cm depths. Samples were then incubated at their respective temperatures for 9 days. Scintillation vials were then removed, immediately capped and refrigerated at 4°C until titration. Quantitative additions of 1 M hydrochloric acid (HCl^-) were added to a solution of 10 mL of NaOH sample with 15 mL of 2 M barium chloride ($BaCl_2$) and 3 drops of phenolphthalein indicator solution until colour change was apparent (Hopkins 2007).

3.2.3.3 - Microbial Biomass-Carbon

Microbial biomass-carbon analysis was performed using the $CHCl_3$ fumigation-extraction method (Vance et al. 1987). Immediately following basal respiration, 25 g mineral soil and 10 g peat soil were weighed into glass beakers. Two jars were used for each sample; one for $CHCl_3$ fumigation and one un-fumigated for immediate extraction. Water content was measured again for later use in calculations. Fumigated samples were placed with 40 mL ethanol-free $CHCl_3$ in an evacuated desiccator for 48 hrs. The decrease in partial pressure from the vacuum causes chloroform to evaporate and the $CHCl_3$-laden atmosphere fumigates the soil samples. After removing the beaker of $CHCl_3$ from the chamber, residual atmospheric and sample-bound $CHCl_3$

was removed by repeatedly de-pressurizing desiccator 5 times. Dissolved organic carbon and nitrogen were extracted with 1:2 (mineral soil) and 1:4 (organic soil) ratio of soil:extract solution, using 0.5 M potassium sulfate (K_2SO_4), and placed in a shaker for 1 hr. Solutions were then filtered via vacuum filtration through Whatmann #2 filter paper and frozen until analysis. Samples were analyzed by NRAL for TOC and N using a Shimadzu TOC-V/TN instrument (Mandel Scientific Company Inc., ON, Canada) (Voroney et al. 2006).

3.2.3.4 - Phospholipid Fatty-Acid Analysis

Samples collected for PLFA analysis were removed from -80 ° C superfreezer storage and immediately freeze-dried in sterile glassware. Soils were measured to 0.75 organic and 2 g mineral soil, due to an expected greater PLFA recovery from organic soils. Phospholipid fatty acids were extracted according to a method derived from Bligh and Dyer (1959) and modified by and White and Ringelberg (1998). Prior to extraction, a surrogate standard (19:0 biomarker) was added for calculating final PLFA recovery. Lipids are extracted from soil samples using chloroform-methanol-citrate buffer mixture (Bligh and Dyer 1959). The methanol-soluble fraction was removed prior to lipid fractionation. Neutral lipids and glycolipids were eluted with chloroform and acetone, respectively, through silicic acid SPE columns (Agilent Technologies, Wilmington, DE, USA), while polar lipids (phospholipids) were captured in a methanol eluate. Subsequently, phospholipids underwent mild alkaline methylation to produce fatty acid methyl esters (FAME) for quantification (Frostegård and Bååth 1996). An internal standard (Me 10:0) was added on the final step for calculating final PLFA concentrations. Soil FAMEs were assessed using a hydrogen carrier gas through a 25 m Ultra 2 (5 % - phenyl) – methylpolysiloxane column in an Agilent 6890 Series capillary gas chromatograph (Agilent

Technologies, Wilmington, DE, USA). Sherlock® Microbial ID System software identified and quantified FAME biomarker peaks from the gas chromatograph (MIDI, Inc., Newark, DE, USA).

3.2.3.5 - Community Level Physiological Profiles

Community level physiological profiles (CLPP) were assessed to determine the functional capability of soil biota (Campbell et al. 2003). Analysis was performed in accordance with the methods and equipment provided by MicroResp™ (Macaulay Scientific Consulting Ltd., Aberdeen, Scotland). This method provides greater discrimination compared with conventional substrate induced respiration (SIR) methods proposed by Degens and Harris (1997) (Lalor et al. 2007). Detection plates were prepared a minimum of 3 days prior to the experiment to allow the agar to thoroughly congeal. Indicator solution was prepared with 18.75 mg of cresol red, 16.77 g KCl, and 0.315 g $NaHCO_3$ dissolved in 1 L of de-ionized (DI) H_2O at 50°C. Solution was stored at 4°C until plating detection plates. Using a water bath heated to 60°C, agar was made by dissolving 3 g of purified agar in 100 mL DI H_2O. Agar was then autoclaved at 120°C and 20 kPa for 20 min. A 1:2 ratio of agar to indicator solution was prepared for plating. Once mixed thoroughly, indicator agar was dispensed into a 100 mL reagent reservoir in the same water bath. Pre-heated micropipette tips delivered 150 μL aliquots to detection plates. Tips were discarded after dispensing 6 rows due to an accumulation of agar within the tips leading to unequal dispensations.

A calibration curve (Eq. 1) was made for absorbance readings to CO_2 concentration by incubating plates in 500 mL mason jars with reclamation and agricultural soils over 1, 3, 6, 16 and 72 hours using a LI-8150 Multiplexer coupled with a LI-8100 IRGA (LI-COR Biosciences, Lincoln, NE, USA) (Figure 6.7). Detection strips (4 wells filled with detection agar solution) were taped inside jars and analyzed on a Synergy™ HT Multi-Mode Microplate Reader (BioTek

Instruments Inc., Winooski, VT, USA) at 570 nm wavelength. A total of 43 data points made up the calibration curve ($r^2 = 0.8051$).

Eq. 1: CO_2 Concentration = 443.32 × Absorbance $^{-3.377}$

Sieved samples from core # 1 were used since soil heterogeneity resulting from aggregates, roots and bituminous aggregates would increase sample variance due to the small amount of soil contained in deep well plates. All soils were brought to 50% WHC, covered and pre-incubated at 25°C for 3 days prior to running the experiment. Soil samples were then evenly spread into loading trays and an average weight of soil in each well was calculated to determine water content. Depending on soil water content, stock solutions of C substrates were diluted to achieve a final C concentration of 30 mg g^{-1} soil water.

Carbon substrates were selected based on typical substrates suggested by the MicroResp™ system which include compounds ranging in chemical structure and functional groups to provide a diversity of carbon or nitrogen sources for microorganisms. A selection bias is present due to limited solubility in water. Fifteen substrates and one control (DI H_2O) were used to assess samples for microbial community function (Table 3.4). Substrates were dispensed into deep-well plates in 3 replicates of 150 μL aliquots, according to alphabetical order. Stock solutions were made for each substrate and diluted according to the specific water content of each sample type to attain a substrate concentration of 30 mg g^{-1} soil H_2O. Stock solutions were refrigerated at 4 °C for the duration of the experiment.

Two soil samples were measured simultaneously on 96-well plates. Loading trays were filled with soil and weighed, and then soil was dispensed into deep wells uniformly to provide an identical T_0 when soil came into contact with substrate solutions. Deep well plates were outfitted with rubber gaskets and detection plates containing indicator agar were secured on top using clamps. Samples were incubated 25 °C for 6 hours. Mineralized CO_2 from soil microorganisms was absorbed by indicator agar forming carbonic acid (H_2CO_3)(Eq. 2). This decreases agar pH with the result of phenolphthalein indicator changing colour.

Eq. 2: $CO_2\ (aq) + H_2O \leftrightarrow H_2CO_3\ (aq)$

3.2.4 - Statistical Analyses

Data from PRS™ probe, PLFA and CLPP methodologies were analyzed using multivariate statistics in ordination space. Non-metric multidimensional scaling (NMS) was selected to generate a dissimilarity matrix plotted on a bi-plot using PC-ORD v. 6.0 (MjM Software Design, Gleneden Beach, Oregon, USA). Points closer to one another on bi-plots share greater similarity than those farther apart. Statistical differences based on distances between groups of topsoil type and depth were assessed using multiple response permutation procedures (MRPP, $\alpha = 0.05$). Reported statistics in this analysis include the P-value indicating statistical significance; T-value signifying the strength of the difference between groups with more negative values being more different; and the A-value representing the variation within groups where 1 indicates no variation and 0 indicates completely random associations (Zimmerman et al. 1985). Vectors indicate correlation with relevant environmental variables, but do not influence ordination results. These were included in ordination bi-plots if $r^2 \geq 0.40$. Dependent variables present in < 20 % of experimental units were excluded from analysis to reduce noise (PLFA and PRS™). Raw data from CLPP and PRS™ were transformed using a general relativization on experimental units and arcsine square root transformation on dependent variables. Ordinations were run on "Autopilot" with a "Slow and Thorough" analysis. All NMS ordinations and subsequent MRPPs were evaluated using the Sorensen (Bray-Curtis) dissimilarity index. No transformed was performed on PLFA data as this produced one-dimensional solutions. Therefore results were interpreted in the context of the total amount of PLFAs contributing to their location in ordination space rather than proportionate contributions of individual PLFAs to the overall response. The problem of measuring a local instead of the experimental minima, was addressed by running ordinations multiple times.

Average daily high/low temperature was calculated from continuous data collected using EM50 data loggers for use in laboratory analyses. Other statistical analyses were not performed on this dataset due to lack of replication. All other measured parameters were analyzed using mixed models in SAS v. 9.3 software (SAS Institute Inc., Cary, NC, USA) which generated least-squared means used for statistical comparison ($\alpha = 0.05$). Family-wise error (Type I) was controlled using Tukey's procedure. Soil respiration was analyzed with repeated measures ANOVA from samples collected on three dates, after $\log_{10}$ transformation. Since this measurement is largely temperature dependent (Singh and Gupta 1977), average daily high temperature was added as a covariate.

3.3 – Results

In general, I found clear differences in microbial communities and nutrient profiles between PM, FFM and Harvest. Forest floor mix was consistently similar to Harvest in most analyses performed. The parameters measured varied with depth, however this was predominantly controlled by soil type. Having many iterations (250) and a stress value in the final solution < 10 % validates the use of NMS ordinations to determine differences between groups. Additionally, T statistics with corresponding significance at $\alpha = 0.05$ elucidated the degree of separation in the distance matrices.

3.3.1 – Basic Soil Characteristics

The actual placement depths varied slightly from intended placement depths yet samples taken at 5, 15 and 35 cm remained inside the intended material at that depth (Table 3.2). Soil pH and EC were greater in PM than FFM, SS or Harvest ($P < 0.05$) and did not significantly vary with depth within material types ($P > 0.05$). Soil temperatures had greater diurnal variation in FFM than in PM, with both being insulated with increasing depth; most notably so in PM (Figure 3.3). Average daily high temperatures at 5 cm from June 28 to August 22, 2013 were 23.5 ° C ranging 8.2 – 29.3 ° C; and 27.2 ° C ranging 7.7 – 33.8 ° C, for PM and FFM respectively. Temperature at the Harvest site reported an average daily high of 19.1 ° C ranging 10.1 – 22.4 ° C, however this was only measured from June 28 to July 26, whereupon data collection was inadvertently terminated by wildlife interference. Field measurements of volumetric water content (VWC) were unreliable at 5 cm in sandy soils since water content often fell below detection limits. Difficulties with moisture sensors may have arisen due to improper contact with soil at 5 cm due to loose, unconsolidated material. Soil samples measured greater VWC in PM than FFM, SS or Harvest.

3.3.2 - Soil Respiration

Autotrophic and heterotrophic soil respiration, as measured by soil CO_2 efflux, showed that FFM had the greatest overall respiration rate (Figure 3.4; $P < 0.05$). Deep FFM placement showed no added respiration ($P = 0.8870$) while Deep PM had a trend of greater respiration than Shallow at ($P = 0.0562$). Blended B/C subsoil did not significantly alter respiration rates in PM or FFM (data not shown).

3.3.3 - Microbial Respiration and Biomass

Respiration from heterotrophic organisms in PM exceeded CO_2 evolved from all other measured soil types (Figure 3.5; $P < 0.0001$). Deep PM varied from Shallow at 5 cm depth which either indicates differences attributable to placement depth, or greater inherent variability of reclaimed soil types. Similarly, soil microbial biomass-carbon did not significantly differ with depth, rather with topsoil type (Figure 3.6; $P < 0.05$). While PM contained the greatest microbial biomass-carbon, all other treatments did not significantly vary from one another. Data from 35 cm are not shown due to contamination during analysis.

3.3.4 - Available Soil Nutrients

Macronutrient analysis found PM to have substantially larger proportions of available TIN and S while FFM and Harvest had greater availability of P and K (Figure 3.7). Harvest sites had greater proportion of NH_4-N, while NO_3-N was dominant in reclaimed soils. Ordination analysis revealed discrepancies in relative concentrations of plant micro- and macronutrients, confirming FFM's similarity to Harvest over PM (Figure 3.8; Table 3.5). Nutrient data revealed disparities between all topsoil materials at 5 cm ($P < 0.05$). Within soil types, nutrient profiles did not exhibit changes at different depths (Table 3.6). Yet PM did alter underlying SS away from Control indicating an influence from overlying materials on nutrient composition in SS (Figure 3.9). Additionally, the prevalence of base cations (Ca and Mg) likely contributes to a higher pH in PM.

It is important to remember to interpret this data in the context of a dry ecosite. Volumetric water content in Harvest and FFM was in some cases below sensor detection limit. This would limit nutrient adsorption to ionic resins, although vegetation would be experiencing the same limitations. During this same period, VWC of PM was consistently higher, therefore I expect nutrient adsorption was not inhibited by water conveyance.

3.3.5 - PLFA

Microbial community structure varied mostly by soil type (Table 3.7). Harvest, and less so FFM, correlated to fungi:bacteria (F:B) ratio and soil respiration (SR) (Figure 3.10). Structure appeared to change in harvested sites with increasing depths while PM or FFM remained similar from 5 to 15 cm (Figure 3.11; Table 3.8). All topsoils, except Deep FFM, showed differences from Harvest at 5 and 15 cm. At 15 cm in Shallow PM treatments, PLFA profiles showed no difference from Control, indicating the above topsoil had little influence on deeper microbial communities while FFM was altered from Control PLFA assemblages. No difference in community structure was observed between SS and B/C treatments (data not shown). Subsoil had consistent positive correlations with saturated:monounsaturated (S:M) PLFAs indicating microbial stress or a lack of substrate availability (Bossio and Scow 1998, McKinley et al. 2005).

3.3.6 - CLPP

Soil type elicited different responses in microbial community function (Table 3.9). Peat mix had the greatest cumulative respiration response to substrate addition (SSIR), while FFM was most similar to Harvest. Peat mix either met or exceeded most carbon mineralization rates witnessed in Harvest for carboxylic acid and amino acid substrates except for arginine which had a negative response (Figure 3.12). Forest floor mix and Harvest showed a slightly greater affinity for carbohydrate metabolism than PM.

Microbial community function was similar between Shallow FFM and Harvest at 0 – 10 cm depth and were well correlated in terms of decomposition potential for monosaccharides (arabinose, fructose, galactose, glucose), the amino acid arginine, and to a lesser extent the disaccharide trehalose (Figure 3.13). Contrastingly, the microbial community in PM was most effective at decomposing the amino acid cysteine and di- and tri-carboxylic acids (oxalic and citric) and had the greatest SSIR. Shallow FFM treatment showed greater similarity with Harvest than did Deep at the 0 – 10 cm sample interval.

No statistical difference is apparent at the 10 - 20 cm range between Shallow treatments of PM and FFM ($P = 0.9027$). Harvest is not statistically different from Shallow PM or FFM ($P > 0.05$) although some discrepancies are apparent (Figure 3.14). Shallow topsoil applications and Harvest demonstrated a strong affinity for decomposing mono- and di-saccharides (arabinose, fructose, galactose, glucose, N-acetylglucosamine and trehalose), amino acids (arginine, γ-aminobutyric acid and lysine) and water. Deep PM was again correlated with cysteine and citric acid while Deep FFM experienced proportionately greater respiration due to carboxylic acids (citric, malic, oxalic and ketoglutaric acids).

3.4 - Discussion

3.4.1 - Differences Between Soil Types

This study demonstrated the differences in microbial community structure and function, and nutrient availability on the ASCS. My evidence corroborates other findings in the literature, concluding that FFM recreates soil conditions similar to natural benchmarks in oil sands reclamation (Mackenzie and Naeth 2010, Hahn 2012, MacKenzie and Quideau 2012). I expected drastically different results when comparing PM to FFM and Harvest due to the profound differences in origin, and therefore composition.

Soil respiration is an important measure of cumulative autotrophic and heterotrophic metabolic activity and has recently been incorporated into models for success in mine reclamation (Mukhopadhyay et al. 2014) and used to study post-mining landscapes (Helingerová et al. 2010, Bujalský et al. 2014). Soil respiration rates have been found to be positively correlated with soil temperature (Singh and Gupta 1977), fine root biomass (Shibistova et al. 2002) and soil organic matter quality (Leifeld and von Lützow 2014). My study indicated that soil respiration rates were greatest in FFM, followed by Harvest and finally PM (Figure 3.4). Throughout the second season of growth on the ASCS, PM lacked vegetative cover other than what was planted. This left an exposed black organic soil surface which did not exhibit elevated temperatures as expected (Figure 3.3) due to peat's thermal insulative capacity and greater water content (Rydin and Jeglum 2006). In contrast, FFM had greater daily high temperatures, and also hosted greater vegetative cover from regeneration of endogenous propagules (Figure 6.4). The higher temperatures, greater prevalence of vegetation (with associated fine root biomass) and residual OM transported with salvage all likely contributed to the greater CO_2 efflux observed in FFM. Shibistova et al. (2002) suggested that seasonal variation in soil respiration in sandy boreal soils is largely due to temperature fluctuations, with the exception of extremely dry conditions, where moisture becomes a controlling factor. The Harvest benchmark was dry, predominantly supporting lichen and ericaceous shrubs with some forbs and graminoid species, and had thin LFH horizons (~2 cm). Water content was below detectable limits with handheld equipment, and intermittent for data loggers (data not shown). Lower average soil temperature and VWC explains the lower CO_2 efflux rates in Harvest. I attributed the majority of Harvest respiration to autotrophic contributions from roots and carbon allocations to microbial symbionts (Högberg et al. 2001).

Smaller heterotrophic respiration rates witnessed in FFM over PM was expected due to reduced carbon content (OC 1.3 % versus 15.4 %, respectively; Figure 3.5). Heterotrophic, rather than autotrophic, respiration likely contributed a greater proportion to CO_2 efflux in PM due to the abundance of SOM supporting greater MB-C than all other treatments (Figure 3.6), while the minimal vegetative cover could not have contributed much CO_2 from autotrophic respiration. Conversely, FFM and Harvest possessed less OM and greater vegetative cover, therefore increasing contributions from root respiration.

Soil microbial community function governs important ecosystem processes including decomposition and nutrient cycling (Grayston and Prescott 2005) and is therefore an important component of ecosystem reclamation (Dimitriu et al. 2010). Microorganisms metabolize organic compounds thus releasing nutrients into soil solution for plant uptake. Community level physiological profiling assesses the catabolic diversity of the heterotrophic microbiota present in a soil sample by measuring CO_2 respired following the addition of a variety of carbon substrates commonly found in soil organic matter. This method has been used for monitoring trajectories of reclaimed mine sites in several ecosystems globally (Yin et al. 2000, Jasper 2007, Lewis et al. 2010, Banning et al. 2012). In my study, PM exceeded FFM and Harvest respiration responses to carboxylic acids and for most amino acids and amines, but was similar or less active with addition of carbohydrates (Figure 3.13). Arginine had an inhibitory effect on respiration response in PM. These differences could be explained by the quality of organic matter and the microbial community present in the soils prior to salvage. Rowland et al. (2009) found decreased surface soil litter decomposition rates on reclaimed sites compared to natural analogues. Turcotte et al. (2009) assessed SOM quality in the region and found that reclaimed PM sites were closely related to O-alkyl groups (a legacy of peat plant residue since similar

NMR spectra are found in boreal Organic soils). Alternately, SOM in undisturbed stands from upland sites (a1 – d3 ecosites) was dominated by alkyl groups, phenolics and aromatics. This suggests this SOM is likely more diverse, and contains more highly humified and complex compounds requiring a suite of enzymes from a functionally diverse microbial community for decomposition (Degens et al. 2000). Another contributing factor to differences in SOM quality is charcoal residue present in undisturbed stands which would provide more recalcitrant forms of C (Preston and Schmidt 2006).

Banning et al. (2012) also suggested optimized substrate selections could provide sufficient discriminatory power, as they discovered 31 substrates explained 90 % of the variation found in an 86 substrate analysis. I was only able to analyze the CO_2 response to 15 substrates which lacked complex compounds that are described as characteristic of mature natural stands. However, the substrate selection that I used was still able to discriminate between soil types. One concern for PM, which originates in cool organic soils that are at least partially anaerobic, is that it may not perform adequately in an aerobic upland position. Peat mix had the greatest total CO_2 response to substrate addition, which is likely due to greater MB-C. Proportionate to the SSIR response, PM was most adept at mineralizing carboxylic acids; Harvest (and to a lesser extent FFM) carbohydrates, and Control was more closely associated with amino acids (Figure 3.13). Lehman et al. (1995) similarly concluded that carbohydrates were preferred by surface soil and aquifer microbial communities and amino acids in subsurface soils. This is indicative of a remnant microbial legacy from SS materials, demonstrating little pedogenic development.

Phospholipid fatty acid analysis may be useful in determining the progression towards a functioning natural system since microbial community structure and function is sensitive to biotic and abiotic environmental stresses like SOM content and inorganic N concentration

(Mummey et al. 2002). Stand types have been shown to be a dominant controlling factor in microbial community structure for natural and reclaimed settings (Grayston and Prescott 2005, Hannam et al. 2006, Swallow et al. 2009, Dimitriu et al. 2010, Sorenson et al. 2011). Community composition is largely due to the type and quality of organic material present, which is principally derived from litter-fall on surface horizons in boreal forests. However, this is not the case at newly reclaimed sites since vegetation is not established enough to provide a litter layer. My results corroborate findings that soil prescription has a strong influence on microbial communities (Rowland et al. 2009, Sorenson et al. 2011). Microbial community structure significantly varied between soil types at 5 cm depth (Figure 3.10). It is reasonable to assume microbial communities in different reclamation treatments will progress towards similarity with undisturbed soils as age increases, keeping vegetation constant. However, soil pH, F:B ratio, C:N ratio and stand type (with > 30 % canopy cover) are more important than time since reclamation for explaining variation in microbial community structure and function (Högberg et al. 2007, Dimitriu et al. 2010, Sorenson et al. 2011). These parameters could have caused the dissimilarities witnessed at 5 cm between all topsoil types (MRPP $P < 0.05$). Harvest and FFM bore the closest resemblance in ordination space with PLFA data, and were associated with F:B; while elevated pH was correlated with PM while FFM was most acidic and had the highest C:N ratio. This was expected since FFM and Harvest likely possess similar OM quality for microbial growth due to similar vegetative composition and inputs prior to disturbance.

Additionally, slope position and associated seasonal soil water content have been shown to affect microbial community structure, as measured by PLFA analysis, under certain stands (Swallow et al. 2009). Actinomycetes dominate moist sites while fungi are most prolific in well-drained upper slopes. Increased VWC in PM likely contributes to these differences since PM contained similar quantities of actinomycete and fungal PLFAs to Harvest, but less in proportion to total PLFA biomass. Large bacterial populations supported by a ubiquitous carbon source in PM are responsible for discrepancies in these ratios. The PLFA biomarker 16:1 w5c was found in greater quantities in PM than FFM. This has been used as a biomarker for arbuscular mycorrhizal fungi (AMF) and was found in PM despite supporting fewer plant symbionts. However Frostegaard et al. (2011) caution against its exclusive use for identifying AMF since it is also found in some bacterial cells (Nichols et al. 1986). A decreased in AMF is expected following salvage due to a disintegration of hyphal networks following physical disturbance (Jasper et al. 1991). Typical of disturbed soils, the F:B ratio was depressed in all reclaimed treatments compared to Harvest and has not had a chance to recover in the 2 years since disturbance. Zak and Parkinson (1983) demonstrated that after 2 years of amending tailings sand with different organic amendments, PM had the highest level of wheat grass root colonization by AMF of all other amendments. Should these fungal networks develop, they could alleviate nutrient discrepancies in PM.

Macronutrients are distinguishably different from PM to FFM and Harvest. Total inorganic N and S availability is significantly greater in PM while FFM and Harvest share similar macronutrient profiles (Figure 3.8, Figure 3.9). This supports other evidence suggesting that soil P may be more limiting than nitrogen in reclaimed PM sites (MacKenzie and Quideau 2012, Pinno et al. 2012). Dissimilarities may limit potential growth of some species, however aspen seedling growth is more responsive to additions of N and inherent soil K than other macronutrients, as described by Pinno et al. (2012) in a greenhouse experiment. In this study, despite low concentrations of soil P, fertilization with a P and K combination created only a small growth response compared to N. While P has been shown to be sourced from B horizons in some ecosites in Alberta (Lanoue 2003), my results do not suggest increased P availability when including a the B/C subsoil salvaged from this location (data not shown).

Total inorganic N speciation differed in proportions from Harvest to PM and FFM sites. A greater proportion of NO_3^- is found in PM soils while FFM and SS have slightly greater amounts of NH_4^+. This is supported by Hemstock et al. (2010) who found nitrate dominated N availability in reclaimed PM soils. In a laboratory experiment, MacKenzie and Quideau (2012) also found that PM quickly converted additions of NH_4^+ to NO_3^-, while additions of NH_4^+ to FFM was rapidly absorbed or transformed. Having greater TIN availability, one concern with PM (especially on reclaimed sites where N fertilizer is applied) is that this management method could favour highly productive invasive species instead of natural species acclimated to natural, low-fertility soils (Marrs 1993, Norman et al. 2006). Nitrogen dynamics are expected to change as forest litter layers develop and will mostly depend on initial litter quality inherent to the species providing it (Norris et al. 2013b). High NO_3^- availability indicates a disconnect in plant-soil interactions characteristic of recently disturbed sites.

Base cations Ca, and less so Mg, presented greater availability in PM, contributing to higher pH and likely EC. Peat mix on the ASCS was likely salvaged from moderate to extremely rich fens which tend to possess pH ranges of ~ 5.5 – 8.0 (Vitt et al. 1995). Greater prevalence of base cations in fen soils is a result of water chemistry where cations were transported from underlying sedimentary geological strata, which may be highly saline (Purdy et al. 2005). Higher pH and greater concentrations of Ca present in PM could explain low P availability due to complexation.

3.4.2 - Effect of Placement Depth

Most of the important edaphic ecological processes in boreal mixedwood stands occur in organic and A horizons of forest soils. Jackson et al. (1996) found that 80 – 90 % of root biomass in boreal systems was located within the top 30 cm of soil. Although most of the variation in my data is explained by reclaimed soil type, meaningful differences do exist with depth.

In a study of Organic soils in the Canadian boreal forest, Rayment & Jarvis (2000) found greater soil respiration with increased organic layer depth. This is similar to my results where greater application depths of PM showed a trend of increased soil respiration (Figure 3.4). Considering the greater heterotrophic activity and MB-C and lower plant cover, it is reasonable to assume that soil respiration is predominantly a measure of heterotrophic activity on PM materials. It is likely that deeper placements will continue to be metabolically active into the fall even after surface soil has frozen since PM acts as an insulator (Rayment and Jarvis 2000). Conversely, from field observations in the spring prior to sampling, PM remained frozen longer than its mineral counterparts which could cause delays in plant growth. In FFM, depth did not influence soil respiration efflux rates. This was likely due to less inherent OM and comparatively abundant vegetation contributing to autotrophic respiration; which likely marginalized heterotrophic contributions that may arise with increased application depth.

Organic matter mineralization is affected in peatlands subjected to warming, drainage and changes in vegetative cover (Ward et al. 2014). This brings into question the peat as a topsoil amendment in upland reclamation. Little is known about long-term fate of reclaimed PM soils since the only site to receive reclamation certification was reclaimed in the early 1980's. To date, however, there is no evidence suggesting that PM horizons are shrinking due to decomposition, despite placement in well drained topographic positions with fertilizer amendments (Marty Yarmuch, personal communication, November 11, 2014).

It is important to remember that these reclaimed sites have not yet developed a litter layer (LFH horizon) responsible for decomposition, nutrient cycling and water retention (Prescott et al. 2000). This is where the greatest microbial diversity exists, with decreasing community richness with increasing depth. Sorensen et al. (2011) found that microbial communities responded to vegetation type instead of reclamation treatment when 30 % canopy cover is achieved and LFH horizons develop. My study did not assess the LFH layer in the Harvest site (~3 cm depth) since specified depths were being compared on reclaimed sites possessing no litter layer. Microbial communities in reclaimed soils will eventually deplete labile C from donor soil and will need to rely on endogenous contribution (Hannam et al. 2005). Development of a functioning litter layer is critical to achieving successful reclamation, so my study underestimates microbial presence on Harvest treatments due to exclusion of LFH layer analysis. The composition and depth of this layer largely depends on over- and under-story species composition, temperature, water availability. Since reclaimed sites were planted with three different tree species, PLFA community composition will develop differently under different vegetation especially if trembling aspen becomes dominant (Hannam et al. 2006). If PLFA is to be used as an indicator of reclamation success, natural soils used for reclamation benchmarks should be ecosite-specific.

Microbial community structure varied between soil types, but not depth within FFM or PM (Figure 3.11). Since soils were recently placed, little pedogenic development has occurred and soil materials possess homogeneous abiotic and biotic factors within soil types – with the exception of temperature and VWC which do not appear to influence community composition.

Figure 3.9 provides evidence of labile nutrients moving from PM into underlying SS, so we can assume that some DOC would also be translocated. Any DOC percolating down from PM-SS interfaces is too little to shift community structure from SS in Control ($P > 0.05$). This is also reflected in with high saturated:monounsaturated (S:M) PLFA ratio in SS, indicating microbial stress. Since this material has a Sand textural class and was salvaged from below the zone of pedogenic development, it contains little SOM or nutrients and has minimal capacity to retain water. As a result, I expect contrasting PLFA signatures to persist across soil interfaces.

This is different from FFM where overlying topsoil altered SS PLFA communities away from the SS in Control. I hypothesize that this is attributable to textural similarities between the two soil types. Water will percolate through soil interfaces of similar textures more readily than the contrasting textures (and resulting matric potentials) of PM over sandy SS (Naeth et al. 2011), and translocate labile nutrients, DOC and potentially inoculate lower layers with similar microbial communities.

Surficial fluctuations documented by Swallow et al. (2009) showed seasonal shifts in soil moisture and temperature can significantly alter microbial community structure in boreal litter layers. Since abiotic factors are more constant at greater depths, I hypothesize that microbial communities at depth are less likely to shift seasonally compared to what they would at the surface.

Harvest CLPPs differed slightly between the uppermost sample interval (0 – 10 cm) and lower intervals (10 – 20 cm and 30 – 40 cm), but not between the lower two intervals, indicating that the relative importance of microbial communities diminishes with depth from the surface (Figure 3.15). This is similar to findings on sandy Mediterranean soils where CLPPs varied spatially between geographically separate groups, but not by depth within groups (Martirosyan et al. 2013). The trend provided by Deep placements of PM and FFM indicates similar separation on axis 1 between the upper two intervals, while the underlying SS remained similar to Harvest. Community level physiological profiles were sensitive to depth within FFM and PM soil, likely due to changes in abiotic environmental conditions (Holden and Fierer 2005). Moving deeper in the profile (10 – 20 cm), CLPPs reflect changes in soil type as opposed to changes with depth Figure 3.14. My results indicate that soil type is the determining factor of CLPPs in early reclaimed soils. Since SS has similar abiotic properties to Harvest soils, there is an apparent shift in Shallow treatments of both PM and FFM towards SS Control and Harvest. Since CLPP under Shallow PM and FFM topsoil applications are similar to Harvest, differences witnessed in PLFA results could indicate functional redundancy of some microbial communities. Shallow applications, especially FFM, may provide sufficient microbial function required for metabolizing organic compounds due to the similarity to Harvest at each sample depth interval. Measurements of CLPP will become more relevant with time as the forest litter layer develops and contributes species-specific SOM. As such, caution must be taken when interpreting data retrieved from newly reclaimed sites since the functional ability of soils to decompose and release nutrients from plant litter will dictate reclamation success in the context of soil microbial ecology.

I expected nutrient availability to change with depth due to differing biological activities, temperatures, water contents, material types and oxygen concentrations. Biological activity in the top 20 cm of soils exerts the greatest control on availability of biologically relevant nutrients while other factors such as leaching, weathering and atmospheric deposition also influence vertical nutrient distributions (Trudgill 1988). Plant macronutrients (N, P and K) critical for physiological processes, are typically concentrated and tightly cycled in the uppermost soil horizons and decrease with increasing depth (Jobbágy and Jackson 2001). This is largely due to an upward translocation of nutrients through plant tissues which are eventually deposited on the soil surface, forming a litter layer. The results for Harvest generally follow this rule while reclaimed sites are still in the process of nutrient accumulation and stratification, especially in the case of K availability.

Peat Mix contains different proportions of available nutrients than does FFM or Harvest (Figure 3.7). With deeper applications of PM, disproportionate amounts of nutrients are increased per unit area of soil. Conversely, FFM lacks significant variability with increased application depth. Harvest varies slightly as depth increases yet little difference exists between measurements at 15 and 35 cm likely due to the similarity of conditions at these depths resulting from weak pedogenic development. At each depth in reclaimed soil profiles, FFM provides available plant-macronutrients in similar proportions to Harvest, with two exceptions. Firstly, Deep application of FFM may provide more available P per unit area, yet not as much as Harvest due to differing concentrations found at 35 cm. Shallow FFM seemed to increase available P at 15 cm but not up to concentrations found at Harvest sites. Increased P storage was an important factor justifying separately salvaged B/C horizon for subsoil use in reclamation treatments, however my study found that available P was not increased with this treatment (data not shown). Secondly, Harvest supplies significantly greater K at 5 cm than all other treatments which is consistent with the hypothesis that scarce nutrients will have shallower vertical distribution (Jobbágy and Jackson 2001). This is due to a disconnected litter supply on reclaimed sites since most K in soil solution is supplied from biological cycling rather than dissolution from minerals. Available K in Harvest soils is likely intercepted and biologically bound before leaching to deeper horizons. With biological cycling over time, we can expect to find increased concentrations of K in reclaimed soil surface layers as plant roots extract K from deeper horizons and release it on the soil surface through litter decomposition. My results indicate that P and K availability are similar to Harvest at 15 cm in FFM, however PM appears to be deficient in comparison. Since PRS™ probes only measure available nutrients in soil solution, PM may contain more nutrients than what were measured since they are bound in organic matter, which will be released over time through decomposition and re-translocated to the soil surface through plant litter (Damman 1978).

Highly mobile, available inorganic N (mostly NO_3^-) and S in PM, migrate into underlying SS materials altering them from control at 15 and 35 cm (Figure 3.7). Sulfur availability did not change with depth in Harvest soils as would be expected (Jobbágy and Jackson 2001). Peat Mix retained most of the S, however some accumulation was noticed in SS below Shallow and Deep treatments. In Shallow PM, NO_3^- concentrations were elevated in underlying SS compared to Control (Figure 3.9). This could explain PLFA and CLPP differences in SS underlying PM and FFM applications since DOC would be transported along with nutrients. These data provide some evidence of water movement across soil interfaces, however the low amounts of precipitation likely restrict ion leaching to periodic rainfall episodes and especially during spring runoff. We can assume that nutrient distributions will stabilize towards natural analogues as roots and mycelial networks develop, and with expression of litter horizons.

3.5 - Conclusion

Disparities between FFM and PM are readily apparent upon visual inspection of reclaimed sites. Forest Floor Mix reflected qualities bestowed from its provenance (Table 3.1, Table 3.2). Therefore, soils salvaged from dry terrestrial/upland positions are pre-conditioned to support like vegetation in reclaimed settings which is supported by their similarity in ordination space. However, the legacy of early reclamation practices and the ubiquity of PM within oil sand leases necessitate its use as a reclamation material. Application depth of scarce topsoil resources should be assessed for every intended reclaimed ecosite. My results suggest Shallow applications of directly placed, coarse textured FFM and PM over suitable SS material may provide best use of resources in establishing microbial function and nutrient availability when reclaiming to dry ecosites. Due to increasing recognition of the importance of soil biology in land reclamation, it is becoming increasingly important to develop standardized analyses that can be applied by industry and government to assess reclaimed sites.

Tables and Figures

Table 3.1 – Soil chemical and physical characteristics from selected plots on the Aurora Soil Capping Study measured by NorthWind Land Resources Inc. in 2012 from samples taken from 0 - 20 cm (reporting means with standard error).

Reclamation Soil Type	OC (% dry weight)		C:N Ratio		Particle Size Distribution						Texture
					S		Si		C		
PM	15.39	(5.99)	23.52	(3.72)	-		-		-		-
FFM	1.27	(0.34)	30.56	(5.32)	91.8	(2.8)	3.9	(2.3)	4.3	(0.9)	Sand
SS	0.38	(0.33)	16.33	(15.04)	96.8	(2.5)	2.1	(3.0)	2.7	(1.0)	Sand

Table 3.2 – Basic soil properties measured from samples collected from the Aurora Soil Capping Study and a benchmark site in August 2013 from 3 depth ranges (reporting means with standard error in parentheses).

Soil Type	Treatment	Topsoil Depth (cm)	Sample Depth (cm)	pH	EC (dS m^{-1})	Db (Mg m^{-3})	VWC (%)
PM	Shallow	9.7 (0.33)	0 - 10	7.66 (0.04)	0.37 (0.065)	0.89 (0.19)	16.95 (5.01)
			10 - 20	6.73 (0.17)	0.05 (0.014)	1.76 (0.07)	6.30 (0.36)
			30 - 40	6.40 (0.40)	0.06 (0.01)	1.66 (0.08)	5.88 (0.53)
	Deep	27.2 (1.09)	0 - 10	7.43 (0.06)	0.74 (0.104)	0.51 (0.05)	45.69 (2.95)
			10 - 20	7.52 (0.02)	0.66 (0.069)	0.48 (0.02)	55.03 (4.20)
			30 - 40	6.45 (0.27)	0.11 (0.025)	1.69 (0.15)	5.99 (1.41)
	B/C	25.7 (4.63)	0 - 10	7.46 (0.09)	0.39 (0.143)	0.37 (0.08)	34.24 (3.19)
			10 - 20	7.41 (0.15)	0.63 (0.059)	0.45 (0.15)	52.48 (12.05)
			30 - 40	7.56 (0.02)	0.18 (0.069)	1.76 (0.09)	- -
FFM	Shallow	11.0 (1.00)	0 - 10	5.58 (0.19)	0.06 (0.012)	1.28 (0.12)	3.61 (1.16)
			10 - 20	7.08 (0.10)	0.03 (0.005)	1.72 (0.02)	5.76 (1.48)
			30 - 40	7.31 (0.17)	0.04 (0.008)	1.67 (0.02)	5.72 (1.39)
	Deep	17.7 (2.67)	0 - 10	5.60 (0.11)	0.04 (0.006)	1.24 (0.14)	2.25 (0.40)
			10 - 20	6.68 (0.66)	0.05 (0.013)	1.59 (0.06)	3.89 (1.40)
			30 - 40	7.14 (0.76)	0.05 (0.028)	1.75 (0.03)	5.99 (3.17)
	B/C	14.3 (0.67)	0 - 10	5.68 (0.11)	0.05 (0.008)	1.35 (0.08)	2.67 (0.24)
			10 - 20	7.05 (0.11)	0.04 (0.002)	1.75 (0.04)	3.82 (0.95)
			30 - 40	7.32 (0.24)	0.04 (0.008)	1.72 (0.13)	7.22 (1.40)
SS		- -	0 - 10	7.55 (0.41)	0.04 (0.013)	1.73 (0.02)	4.55 (0.91)
			10 - 20	7.12 (0.50)	0.03 (0.012)	1.90 (0.03)	6.42 (0.84)
			30 - 40	6.43 (0.77)	0.04 (0.012)	1.79 (0.06)	5.89 (1.18)
Harvest		- -	0 - 10	5.14 (0.09)	0.03 (0.001)	1.29 (0.01)	2.60 (0.32)
			10 - 20	5.14 (0.13)	0.02 (0.002)	1.47 (0.06)	3.09 (0.45)
			30 - 40	5.81 (0.09)	0.02 (0.001)	1.81 (0.02)	3.33 (0.25)

Table 3.3 – Plant macronutrients adsorbed to PRS™ probes on the Aurora Soil Capping Study and a harvested benchmark site, reporting means (µg 10 cm $^{-2}$ 57 days $^{-1}$) with standard error in brackets and significance denoted by letters ($\alpha > 0.1$; n=3).

Depth	Treatment		Inorganic Nitrogen			Phosphorus			Potassium			Sulfur		
5 cm	Shallow	PM	121.59	(24.6)	a	0.27	(0.1)	a	6.99	(1.4)	a	713.86	(104.4)	a
		FFM	11.33	(1.9)	b	3.38	(0.5)	a	76.25	(6.3)	b	30.08	(7.9)	b
	Deep	PM	82.31	(17.4)	a	0.62	(0.2)	a	10.19	(1.6)	a	1393.75	(179.4)	a
		FFM	17.71	(12.0)	b	1.85	(0.9)	a	69.95	(5.9)	b	26.71	(18.7)	b
		Control	9.94	(2.3)	b	0.62	(0.3)	a	21.15	(3.0)	c	35.65	(10.2)	b
		Harvest	4.69	(1.6)	b	3.49	(1.9)	a	192.15	(41.5)	d	8.71	(2.2)	b
			F value = 12.0; P = 0.0002			F value = 3.4; P = 0.0435			F value = 72.2; P < 0.0001			F value = 19.5; P < 0.0001		
15 cm	Shallow	PM	34.55	(4.5)	a	0.32	(0.2)	a	25.90	(3.2)	a	205.94	(22.4)	a
		FFM	10.05	(4.4)	b	1.31	(0.4)	bc	92.41	(14.8)	b	33.09	(7.8)	b
	Deep	PM	108.55	(10.8)	c	0.23	(0.1)	ac	9.44	(0.9)	a	1494.72	(20.1)	c
		FFM	9.13	(3.0)	b	2.35	(0.6)	b	78.27	(15.5)	b	25.27	(7.8)	b
		Control	9.17	(0.6)	b	0.51	(0.3)	ab	21.30	(7.9)	a	19.14	(3.3)	b
		Harvest	2.75	(0.6)	d	3.48	(0.7)	b	72.67	(16.0)	b	5.28	(0.7)	d
			F value = 24.0; P < 0.0001			F value = 8.2; P = 0.0026			F value = 12.3; P = 0.0002			F value = 113.9; P < 0.0001		
35 cm	Shallow	PM	26.98	(1.8)	-	0.40	(0.2)	a	29.45	(9.6)	-	133.28	(33.0)	a
		FFM	12.35	(3.9)	-	0.52	(0.1)	ab	62.92	(42.3)	-	16.63	(0.7)	b
	Deep	PM	13.23	(3.8)	-	0.41	(0.3)	ab	25.61	(3.4)	-	581.06	(34.7)	c
		FFM	13.46	(4.7)	-	0.24	(0.1)	ab	19.40	(2.6)	-	19.61	(7.8)	b
		Control	15.18	(1.2)	-	0.23	(0.1)	a	16.62	(3.9)	-	11.53	(1.7)	bd
		Harvest	9.16	(6.3)	-	2.10	(0.8)	b	32.60	(10.5)	-	5.88	(1.0)	d
			F value = 1.7; P = 0.2007			F value = 3.3; P = 0.0513			F value = 0.9; P = 0.5168			F value = 55.4; P < 0.0001		

Table 3.4 – Substrates used to measure community level physiological profiles in the MicroResp™ method (30 mg g^{-1} soil H_2O).

Carbohydrates	Amines & Amino acids	Carboxylic acids
L-(+)-arabinose	N-acetyl glucosamine	citric acid
D-(-)-fructose	L-alanine	α-ketoglutaric acid
D-(+)-galactose	γ-amino butyric acid	L-malic acid
D-(+)-glucose	L-arginine	oxalic acid
D-(+)-trehalose	L-cysteine-HCl	
	L-lysine-HCl	

Table 3.5 – Statistics from multiple response permutation procedures analysis of non-metric multidimensional scaling ordination for available nutrients adsorbed to PRS™ probes in different soil types at 5 cm depth.

Groups Compared			**T**	**A**	***P***
FFM	-	PM	-11.06	0.53	0.0000
FFM	-	Harvest	-5.14	0.26	0.0010
FFM	-	Control	-3.82	0.21	0.0055
PM	-	Harvest	-7.20	0.60	0.0002
PM	-	Control	-6.58	0.47	0.0002

Table 3.6 – Statistics from multiple response permutation procedures analysis of non-metric multidimensional scaling ordination for available nutrients adsorbed to PRS™ probes in different soil types at three depths for Shallow and Deep topsoil applications.

	Shallow						**Deep**					
Depth	**Groups Compared**			**T**	**A**	***P***	**Groups Compared**			**T**	**A**	***P***
5 cm	FFM	-	PM	-2.85	0.54	0.0224	FFM	-	PM	-2.86	0.52	0.0224
	FFM	-	Harvest	-2.72	0.33	0.0232	FFM	-	Harvest	-2.08	0.21	0.0376
	FFM	-	Control	-1.19	0.16	0.1207	FFM	-	Control	-2.16	0.21	0.0340
	PM	-	Harvest	-2.99	0.70	0.0216	PM	-	Harvest	-2.99	0.69	0.0216
	PM	-	Control	-2.94	0.57	0.0219	PM	-	Control	-2.97	0.62	0.0217
15 cm	FFM	-	PM	-2.61	0.32	0.0240	FFM	-	PM	-2.94	0.61	0.0218
	FFM	-	Harvest	-2.84	0.36	0.0224	FFM	-	Harvest	-2.35	0.23	0.0272
	FFM	-	Control	0.24	-0.03	0.5021	FFM	-	Control	-0.20	0.03	0.3426
	PM	-	Harvest	-2.97	0.60	0.0217	PM	-	Harvest	-2.99	0.74	0.0215
	PM	-	Control	-2.37	0.26	0.0262	PM	-	Control	-2.94	0.62	0.0218
35 cm	FFM	-	PM	-1.51	0.17	0.0826	FFM	-	PM	-2.94	0.50	0.0218
	FFM	-	Harvest	-1.75	0.21	0.0607	FFM	-	Harvest	-2.16	0.28	0.0313
	FFM	-	Control	0.77	-0.07	0.7691	FFM	-	Control	0.94	-0.06	0.8247
	PM	-	Harvest	-2.70	0.34	0.0234	PM	-	Harvest	-2.90	0.49	0.0221
	PM	-	Control	-1.51	0.14	0.0816	PM	-	Control	-2.92	0.42	0.0220

Table 3.7 – Statistics from multiple response permutation procedures analysis of non-metric multidimensional scaling ordination for PLFAs in different soil types at 5 cm depth.

Groups Compared	T	A	*P*
FFM - PM	-10.89	0.48	0.0000
FFM - Harvest	-0.32	0.01	0.3015
FFM - Control	-7.27	0.73	0.0002
PM - Harvest	-6.27	0.28	0.0003
PM - Control	-6.92	0.57	0.0002

Table 3.8 – Statistics from multiple response permutation procedures analysis of non-metric multidimensional scaling ordination for PLFAs in different soil types at three depths for Shallow and Deep topsoil applications.

	Shallow				**Deep**			
	Groups Compared	**T**	**A**	***P***	**Groups Compared**	**T**	**A**	***P***
5 cm	FFM - PM	-2.85	0.48	0.0225	FFM - PM	-2.93	0.58	0.0219
	FFM - Harvest	0.55	-0.05	0.6374	FFM - Harvest	0.01	0.00	0.3936
	FFM - Control	-2.98	0.79	0.0216	FFM - Control	-2.99	0.81	0.0216
	PM - Harvest	-2.90	0.36	0.0221	PM - Harvest	-2.93	0.40	0.0219
	PM - Control	-2.89	0.64	0.0222	PM - Control	-2.98	0.75	0.0216
15 cm	FFM - PM	-2.63	0.38	0.0242	FFM - PM	-0.46	0.06	0.2522
	FFM - Harvest	-2.59	0.26	0.0238	FFM - Harvest	-0.74	0.08	0.2108
	FFM - Control	-2.88	0.52	0.0223	FFM - Control	-2.72	0.60	0.0235
	PM - Harvest	-2.87	0.59	0.0223	PM - Harvest	-2.14	0.16	0.0319
	PM - Control	-0.92	0.09	0.1614	PM - Control	-2.96	0.67	0.0218
35 cm	FFM - PM	0.17	-0.01	1.0000	FFM - PM	-0.25	0.03	0.3161
	FFM - Harvest	-0.87	0.05	0.1861	FFM - Harvest	-1.24	0.06	0.1067
	FFM - Control	-0.22	0.02	0.3772	FFM - Control	0.31	-0.04	0.5672
	PM - Harvest	-0.95	0.07	NaN	PM - Harvest	-0.67	0.12	0.2237
	PM - Control	0.77	-0.16	NaN	PM - Control	0.83	-0.12	0.7896

Table 3.9 – Statistics from multiple response permutation procedures analysis of non-metric multidimensional scaling ordination for CLPPs in different soil types at 0 - 10 cm depth.

Groups Compared	T	A	*P*
FFM - PM	-10.39	0.31	0.0000
FFM - Harvest	-0.95	0.04	0.1542
FFM - Control	-0.74	0.02	0.2005
PM - Harvest	-6.50	0.34	0.0002
PM - Control	-1.60	0.06	0.0739

Table 3.10 – Statistics from multiple response permutation procedures analysis of non-metric multidimensional scaling ordination for CLPPs in different soil types at three depths for Shallow and Deep topsoil applications.

	Shallow				**Deep**			
	Groups Compared	**T**	**A**	***P***	**Groups Compared**	**T**	**A**	***P***
0 - 10 cm	FFM - PM	-2.89	0.34	0.0222	FFM - PM	-2.89	0.32	0.0220
	FFM - Harvest	0.51	-0.04	0.6687	FFM - Harvest	-2.78	0.25	0.0230
	FFM - Control	0.09	-0.01	0.4552	FFM - Control	-1.31	0.08	0.1000
	PM - Harvest	-2.92	0.42	0.0220	PM - Harvest	-2.94	0.42	0.0219
	PM - Control	-0.55	0.06	0.2641	PM - Control	-0.58	0.06	0.2609
10 - 20 cm	FFM - PM	1.20	-0.09	0.9027	FFM - PM	-1.49	0.12	0.0802
	FFM - Harvest	-1.65	0.07	0.0589	FFM - Harvest	-2.92	0.36	0.0220
	FFM - Control	0.64	-0.12	0.7270	FFM - Control	-0.20	0.03	0.3938
	PM - Harvest	-0.94	0.07	0.1729	PM - Harvest	-2.85	0.52	0.0225
	PM - Control	0.27	-0.01	0.5883	PM - Control	-1.33	0.17	0.0996
30 - 40 cm	FFM - PM	0.57	-0.06	0.6768	FFM - PM	0.87	-0.05	0.8046
	FFM - Harvest	-1.77	0.24	0.0538	FFM - Harvest	-1.51	0.10	0.0767
	FFM - Control	0.71	-0.12	0.7405	FFM - Control	0.86	-0.18	0.8017
	PM - Harvest	-0.94	0.12	0.1478	PM - Harvest	-0.64	0.03	0.2479
	PM - Control	1.01	-0.10	0.9099	PM - Control	0.92	-0.06	0.8197

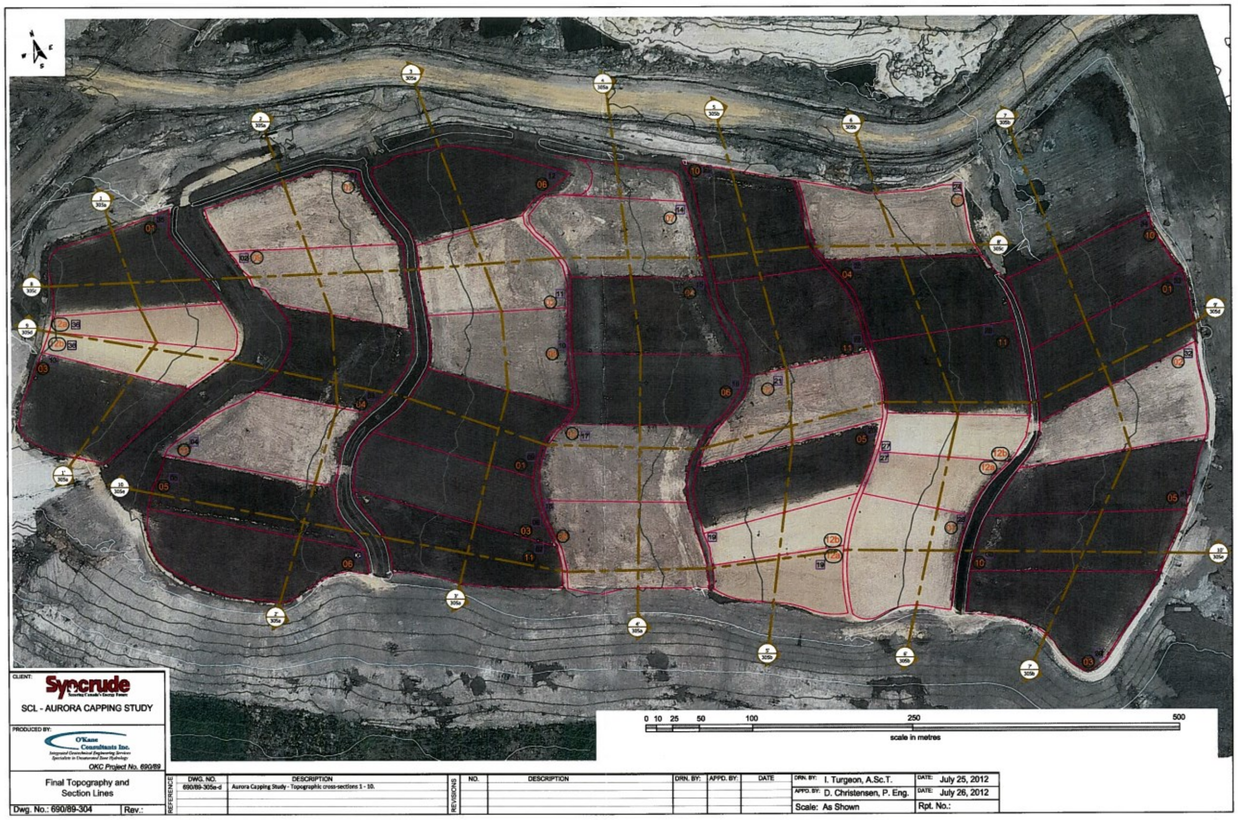

Figure 3.1 – Aerial view of Syncrude Canada's Aurora Soil Capping Study in July 2012 (Syncrude 2012).

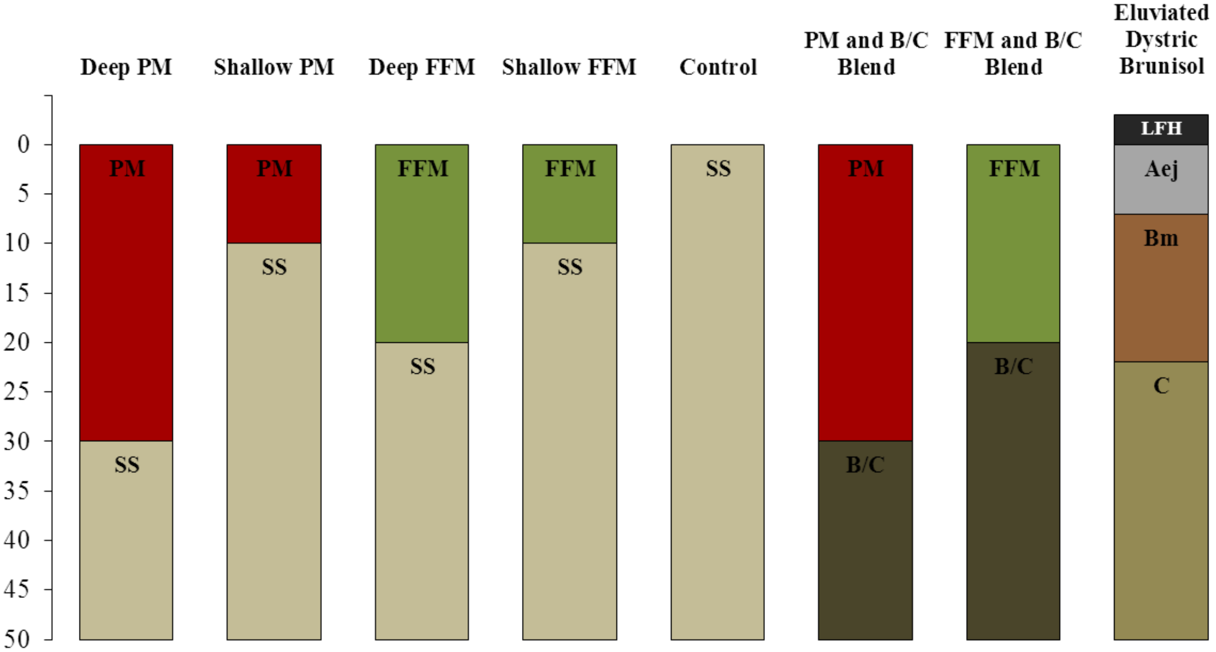

Figure 3.2 – Soil treatments selected for analysis from the Aurora Soil Capping Study compared to a benchmark harvested site (n = 3).

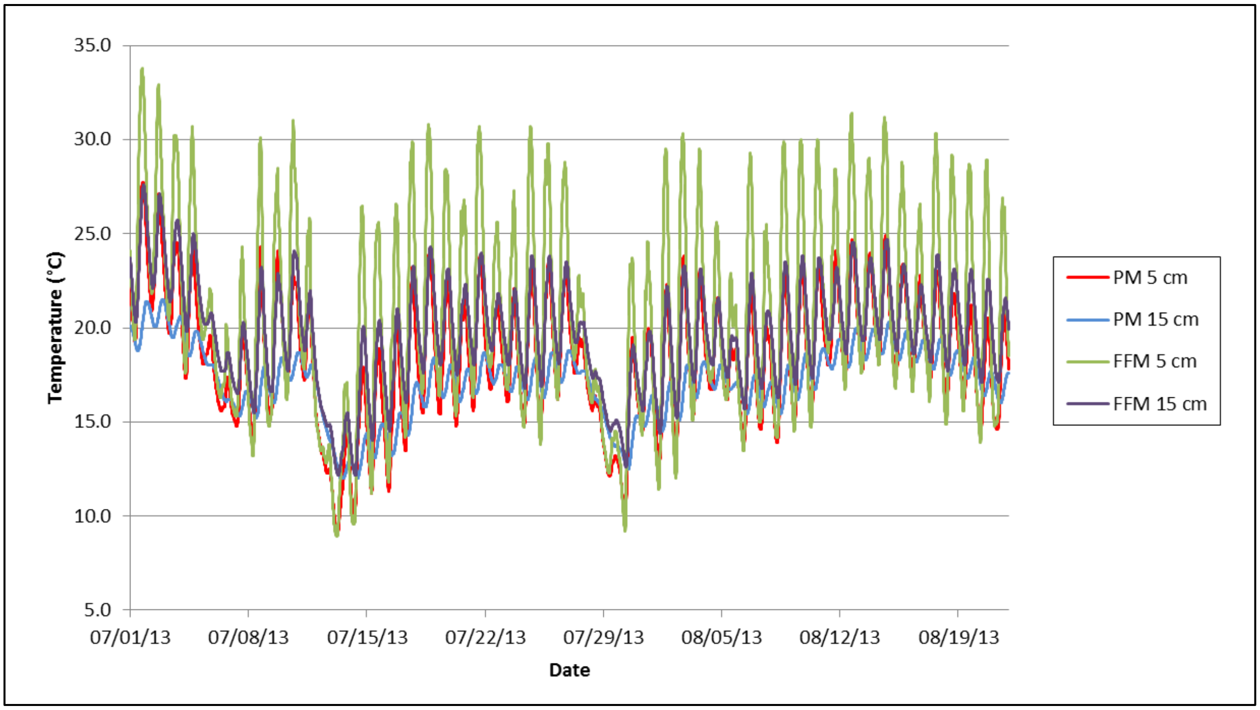

Figure 3.3 – In-situ soil temperature in Deep PM and FFM at 5 and 15 cm on the Aurora Soil Capping Study, logged at 15 minute intervals (n=1).

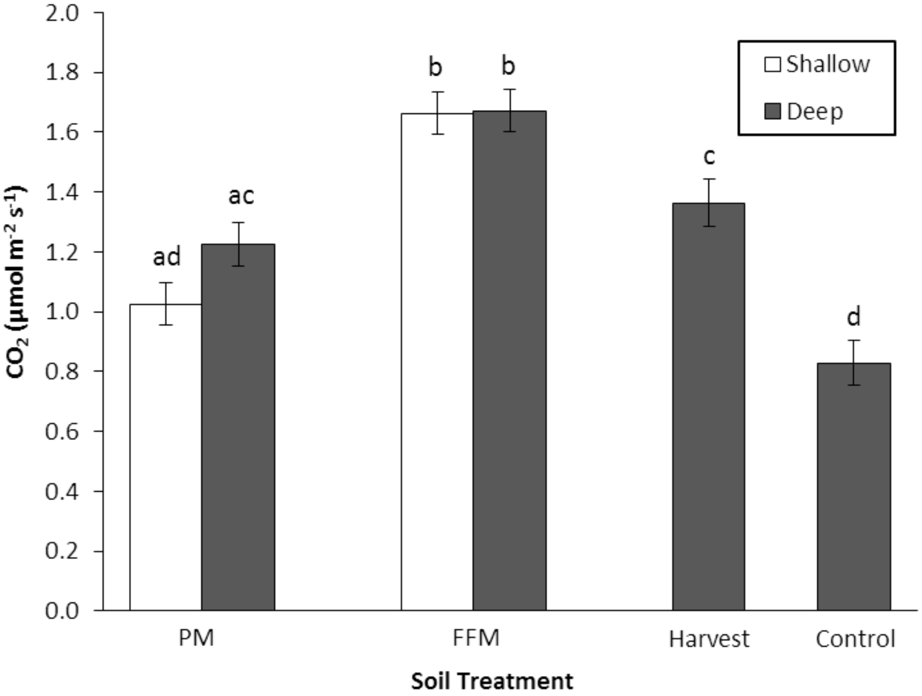

Figure 3.4 – In-situ soil CO_2 efflux analyzed using repeated measures ANOVA from data collected on July 24, August 6 and August 23, 2013. Means reported with error bars depicting standard error (n=3).

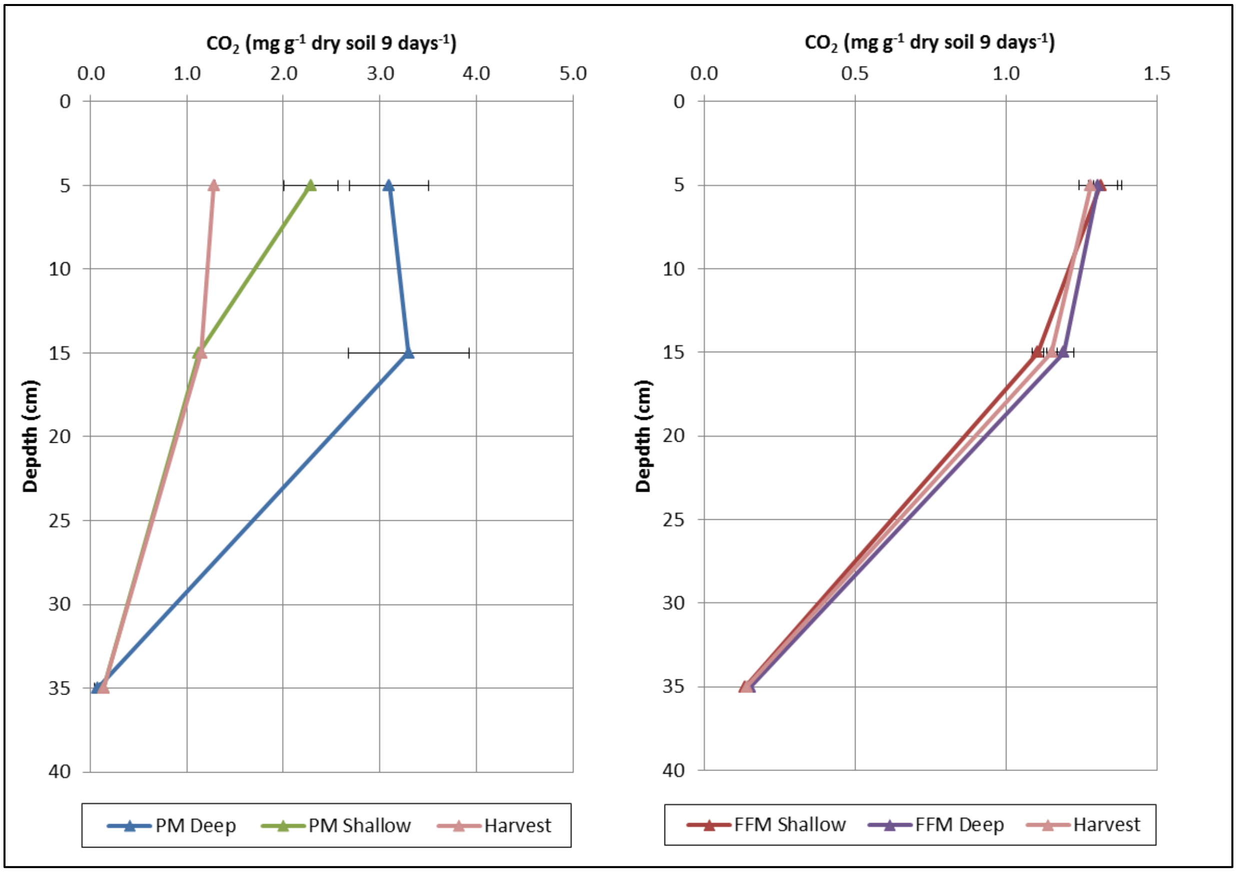

Figure 3.5 – Basal (heterotrophic) respiration measured in the laboratory using the alkali-trap method over 9 days; samples from each depth interval incubated at average daily high temperatures. Means are reported error bars depicting standard error (n=3).

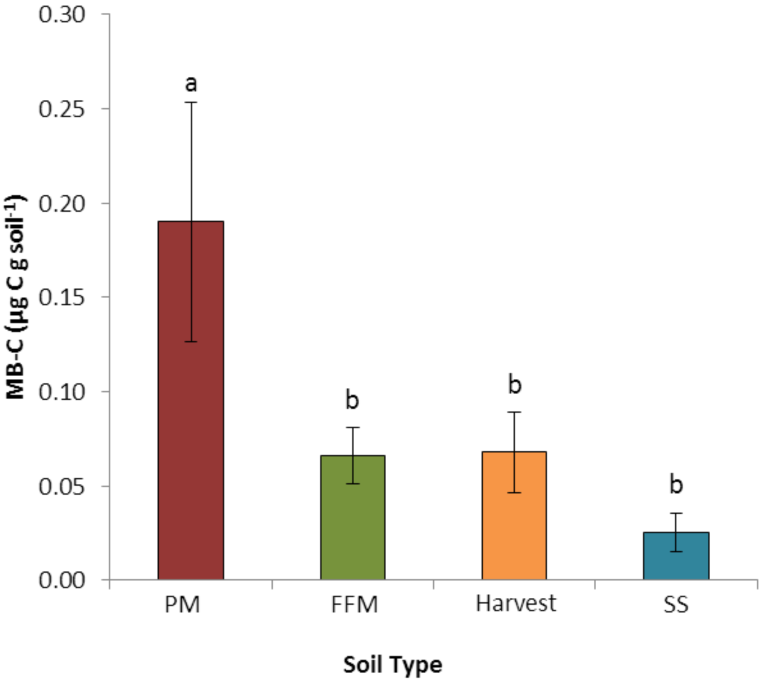

Figure 3.6 – Microbial biomass-carbon measured using chloroform fumigation-extraction following a 9 day incubation at average daily high temperature and water content for PM and FFM (n=6), and Harvest and SS (n=3). Means are reported with significant differences denoted by letters (α = 0.05).

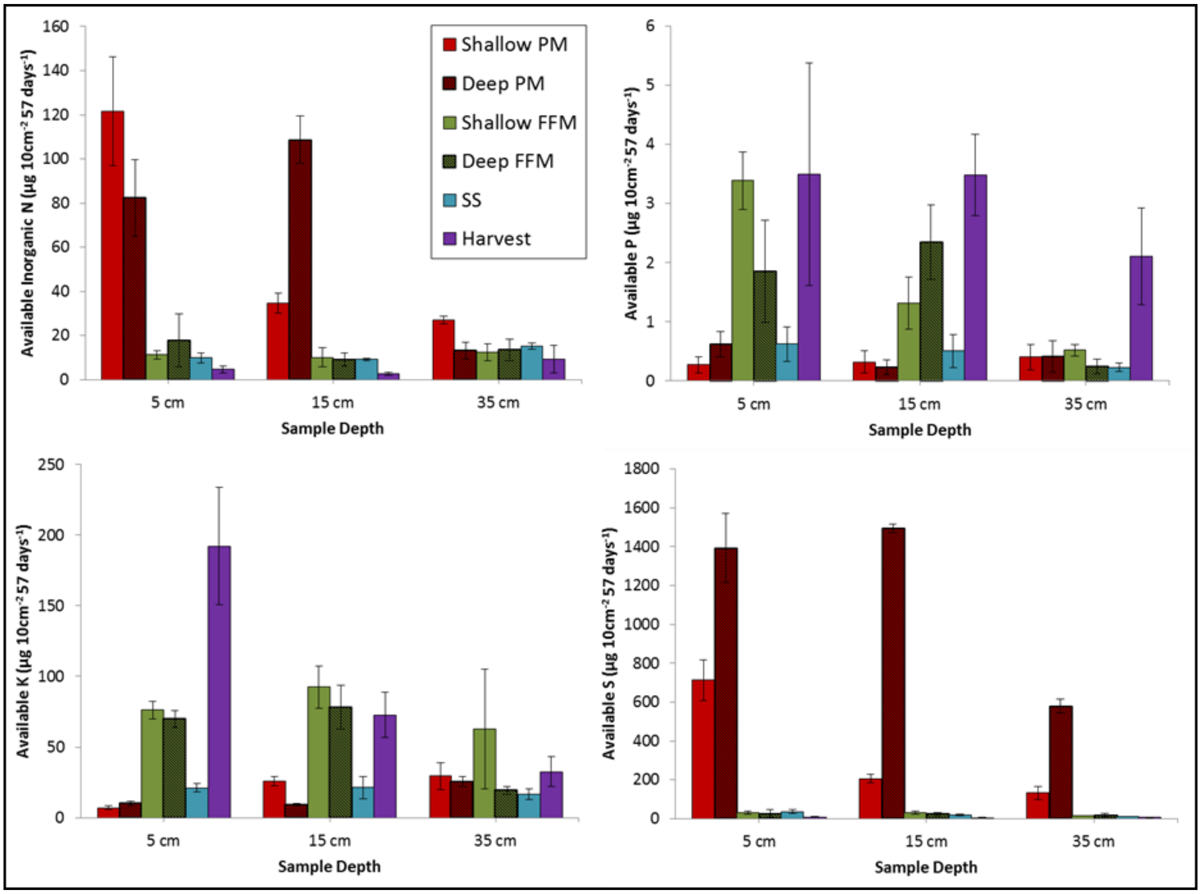

Figure 3.7 – Available nitrogen, phosphorus, potassium and sulfur adsorbed to ionic exchange membranes at each sampled depth over a 57 day burial. Four subsamples measured in composite, means reported with error bars depicting standard error (n=3), statistics included in Table 3.3; note different scale for each nutrient.

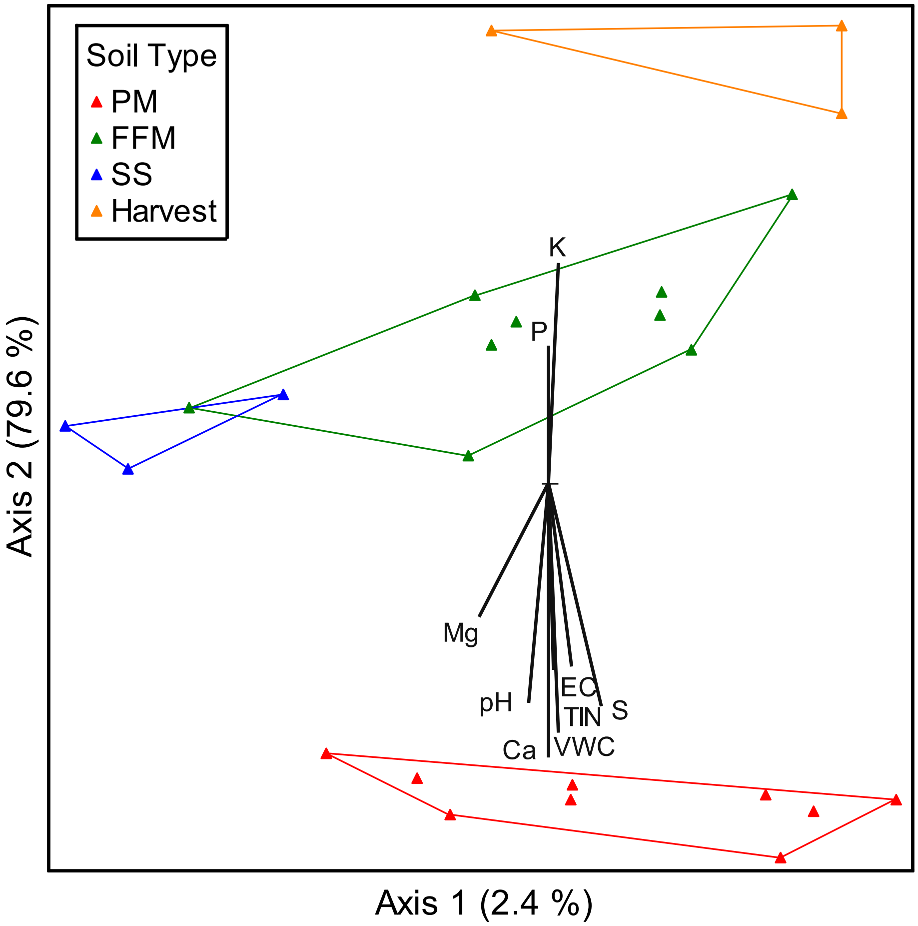

Figure 3.8 – **Non-metric multidimensional scaling ordination of available nutrients measured by adsorption to PRS™ probes at 5 cm from a 57 day burial (stress = 3.2%; minimum vector $r^2 = 0.40$). Vectors include plant-available nutrients (TIN, P, K, S, Ca, Mg), soil acidity (pH), electrical conductivity (EC) and volumetric water content (VWC).**

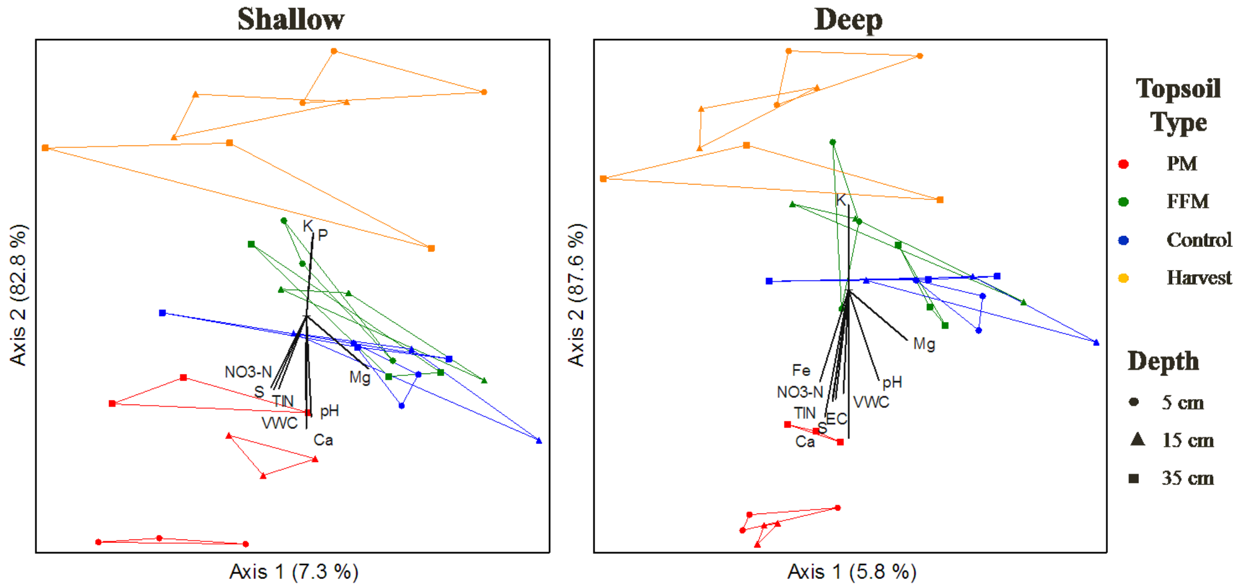

Figure 3.9 – Non-metric multidimensional scaling ordination of available nutrients measured by adsorption to PRS™ probes over a 57 day burial from PM, FFM and Harvest plots at 5, 15 and 35 cm depths grouped in Shallow (stress = 7.2 %) and Deep (stress = 6.3 %) topsoil application depths. Vectors (minimum $r^2 = 0.40$) include plant-available nutrients (TIN, NO_3^-N, P, K, S, Ca, Mg, Fe), soil acidity (pH), electrical conductivity (EC) and volumetric water content (VWC).

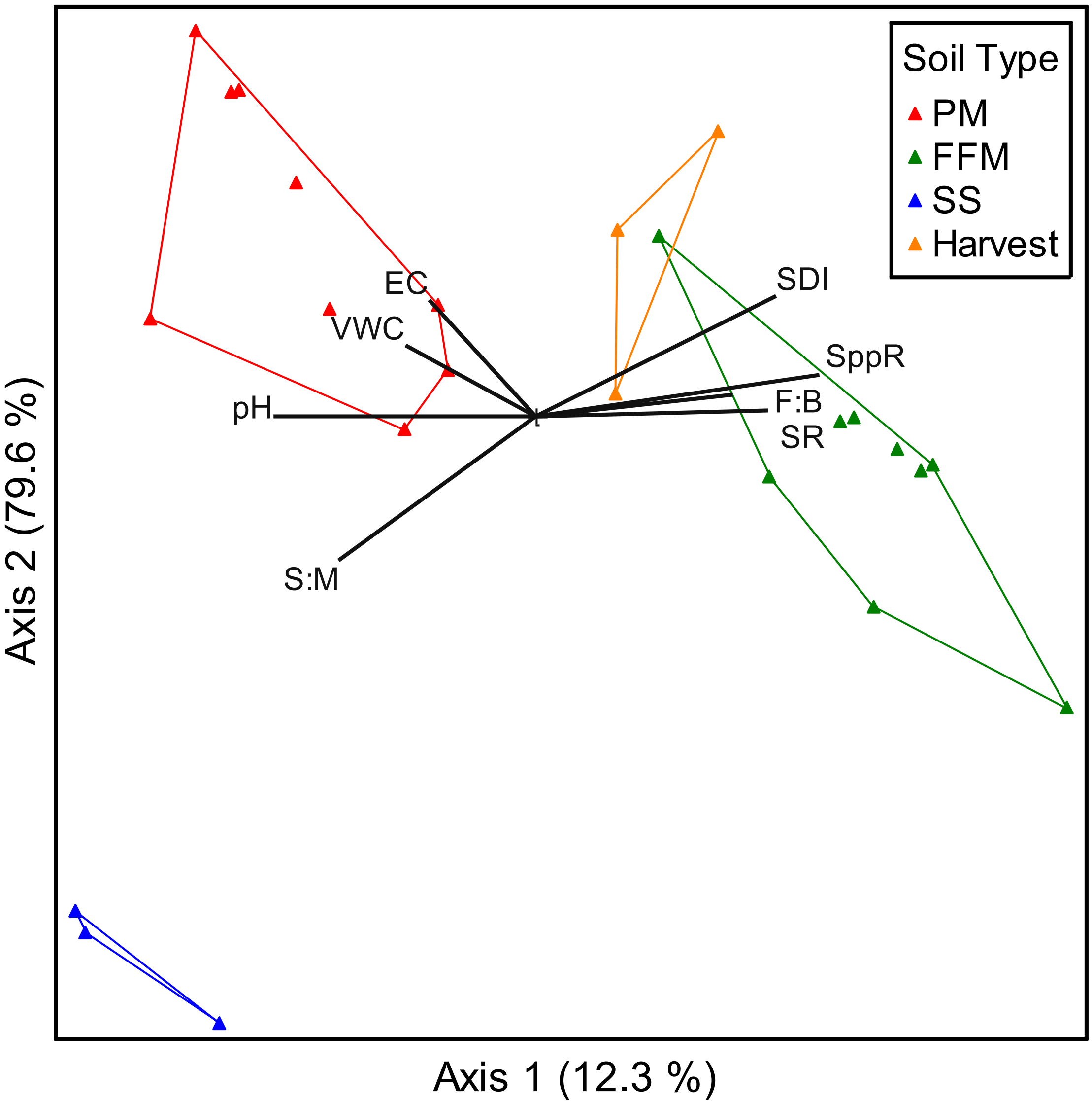

Figure 3.10 – **Non-metric multidimensional scaling ordination of phospholipid fatty acids (PLFA) from reclaimed and Harvest sites of samples taken at 5 cm (stress = 3.4 %; minimum vector r^2 = 0.40). Vectors include electrical conductivity (EC), volumetric water content (VWC), soil acidity (pH), saturated to monounsaturated fatty acid ratio (S:M), Shannon diversity index of PLFAs (SDI), species richness of PLFAs (SppR), fungi to bacteria ratio (F:B) and soil respiration (SR).**

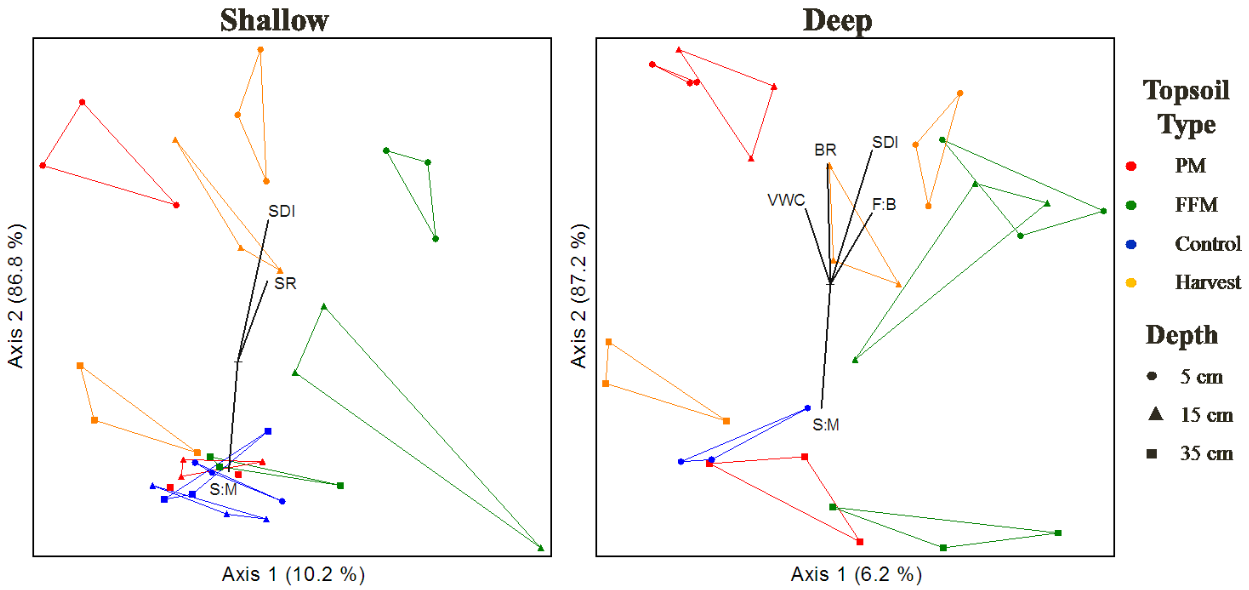

Figure 3.11 – Non-metric multidimensional scaling ordination of phospholipid fatty acids (PLFA) from PM, FFM and Harvest plots at 5, 15 and 35 cm depths grouped in Shallow (stress = 5.0 %) and Deep (stress = 3.0 %) topsoil application depths. Vectors (minimum r^2 = 0.40) include Shannon diversity index for PLFAs (SDI), soil respiration (SR), saturated to monounsaturated fatty acids (S:M), volumetric water content (VWC), basal respiration (BR), and fungi to bacteria ratio (F:B).

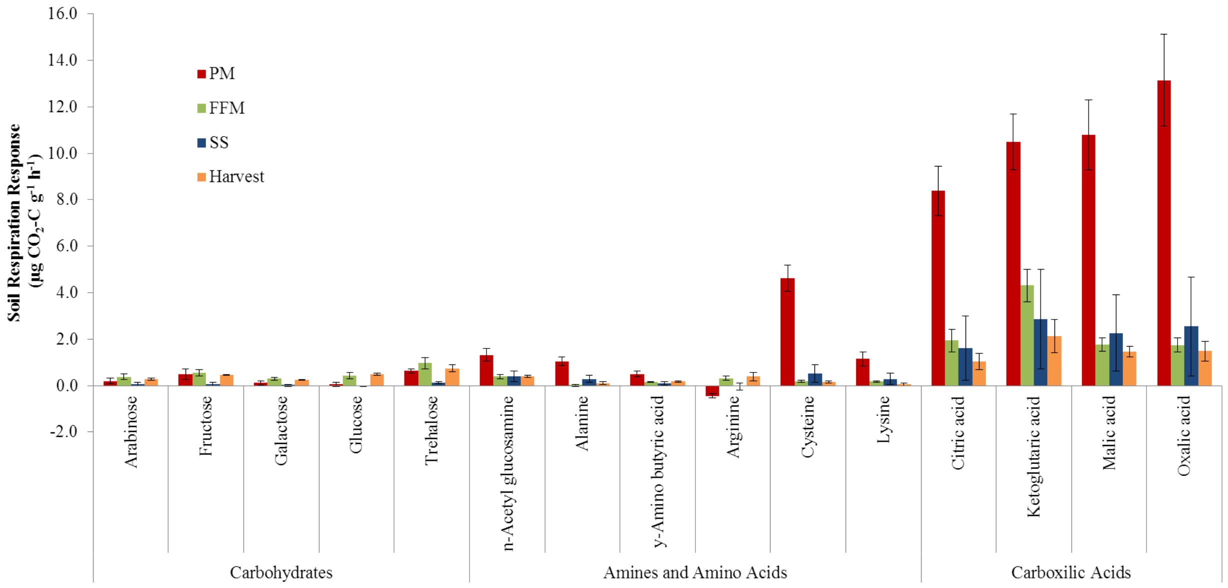

Figure 3.12 – Soil respiration response to the addition of substrates in community level physiological profile analysis for PM and FFM (n=9), and Harvest and Control (n=3) from soils sampled from the 0 – 10 cm interval. Means reported with error bars depicting standard error.

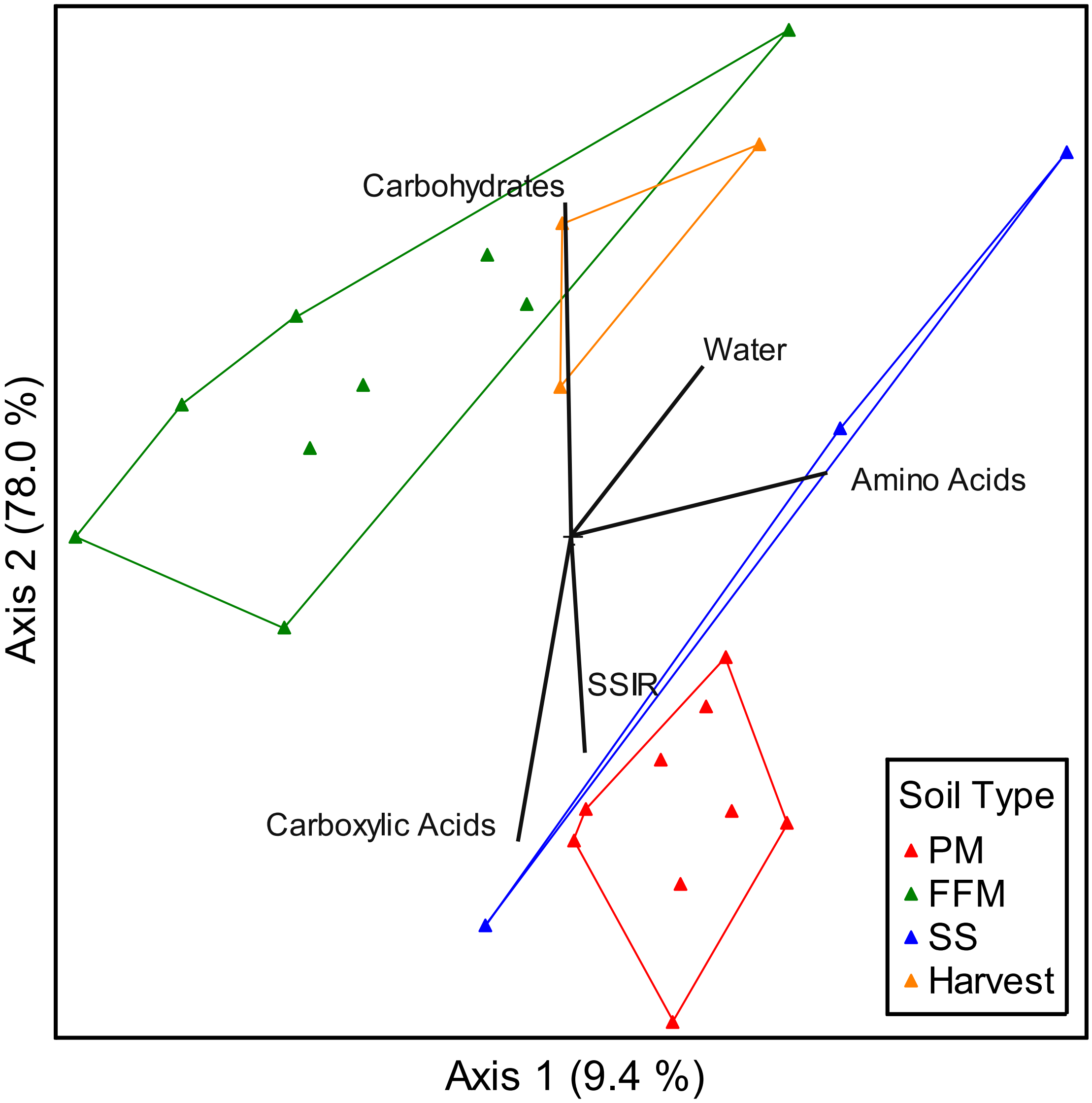

Figure 3.13 – Non-metric multidimensional scaling ordination of community level physiological profile analysis from the 0 – 10 cm sampling interval for Shallow and Deep topsoil application depths compared to Control and Harvest soils (stress = 7.1 %; minimum vector r^2 = 0.40). Sum of substrate induced respiration (SSIR) is the cumulative respiration response of added substrates.

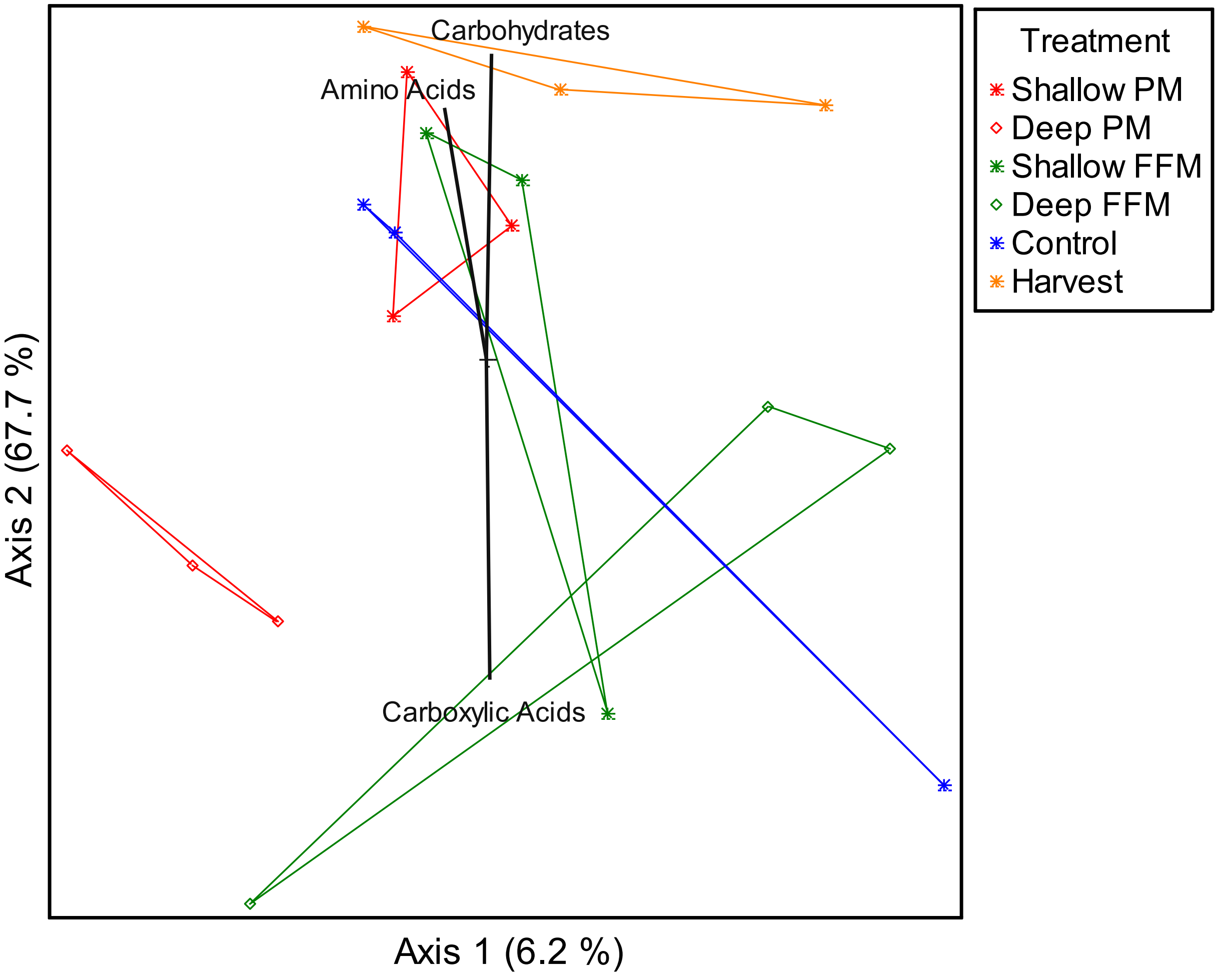

Figure 3.14 – Non-metric multidimensional scaling ordination of community level physiological profile analysis from the 10 – 20 cm sampling interval for Shallow and Deep topsoil application depths compared to Control and Harvest soils (stress = 4.3 %; minimum vector $r^2 = 0.40$).

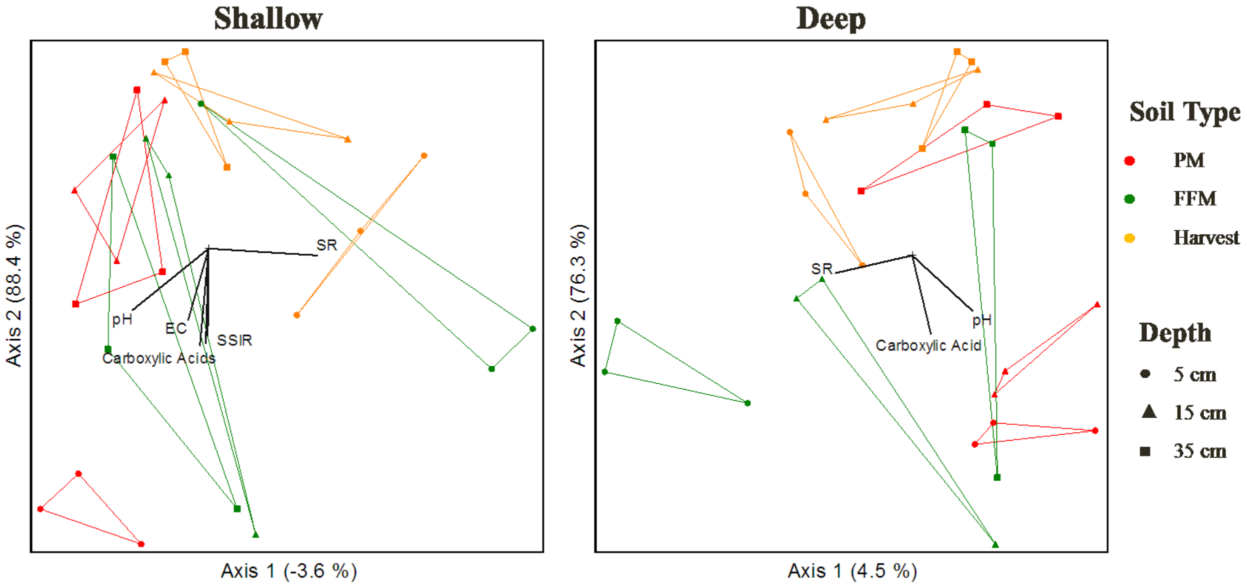

Figure 3.15 – Non-metric multidimensional scaling ordination of community level physiological profile analysis from whole profiles of Deep PM and FFM samples (stress = 11.1 %; minimum vector r^2 = 0.40; vectors 75 % of maximum length).

4.0 Technology Transfer and Study Implications

4.1 - Introduction

In an effort to communicate research findings with industrial and regulatory partners, this chapter summarizes salient points derived from these studies to contribute to the direction of future research and best management practices for reclamation in the AOSR. The principal goal of reclamation is to return functionally similar ecosystems relative to what was present prior to disturbance. Our effectiveness as reclamation practitioners will depend on the type and amount of materials used for reclamation and on management following soil placement. Our efforts aim to expedite reclamation by creating disturbed sites similar to secondary succession, instead of leaving them to begin from primary succession. Ideally, with increasing time these sites will begin to resemble natural forests. However it is likely that much of the disturbed landscape will result in novel ecosystems, which differ from natural boreal forest but will hopefully possess similar functional capacities to the pre-existing landscapes.

4.2 - Type of Materials

Organic soils salvaged from lowlands with saturated conditions are high in residual organic matter from moss accumulation. This is the result of slow decomposition rates in cold and anaerobic conditions. Conditions forming Organic soils are uncharacteristic of well-drained upland positions; however, historically PM has been industry’s topsoil of choice since it is found in greater quantities than FFM within mineral leases. It is not disputed that PM can support trees and other species of interest. It provides several benefits for establishing vegetation, hence its use in the horticultural industry. These include soil nutrients mineralized from decomposition, high porosity, high water holding capacity, greater MB-C and low bulk density. It is logical to assume that placing PM as topsoil in an aerobic upland environment may lead to decomposition and loss of topsoil over time; however this has yet to be measured on reclaimed sites in the AOSR. Questions remain as to whether PM will return major ecosystem processes, and be resilient to natural disturbances. This is of particular interest due to regular fire intervals in boreal regions. Peat fires in bogs or fens with low water tables, can burn for very long periods of time, even through winter months. Although PM is typically mixed with mineral components and has a high water holding capacity, there still exist potential for topsoil loss which could remove plant propagules and annihilate soil microbial communities due to incineration and heat pulses (Neff et al. 2005).

Standard soil physical and chemical tests identify compositional and performance differences between topsoil types. When compared with FFM, PM is less optimal for use in upland reclamation in many ways. Peat mix was frozen later into the spring than was FFM which will likely result in seasonal postponement of biological activity (O'kane Consultants 2014). Mackenzie and Naeth (2010) found FFM outperformed PM in establishing native species cover. Propagules salvaged with the FFM provided greater species diversity on reclaimed sites when soil was directly placed. Despite belonging to diffing ecosites, both of my studies supported findings in the literature that FFM shares the greatest similarity with natural upland benchmarks with respect to nutrient availability and soil biology. Coupled with the inherent propagules in directly placed FFM, these qualities promote establishment of vegetative communities with greater native species diversity. Similarly, FFM introduces a similar microbial community structure and function to reclaimed sites as demonstrated in CLPP and PLFA analyses. These results support the concept of returning FFM and PM to similar topographic and drainage postions from which they were salvaged.

However, the sites measured in this study were young and plants had not had a chance to explore deeper horizons, so it is unclear how much this will change in the future. Additionally, mycorhizzal associations are expected to proliferate as woody species become established. The progression of microbial communities should be assessed in monitoring programs to determine if communities found shortly following soil placement and re-forestation efforts can provide early indicators of reclamation success. Furthermore, microbial community structure is expected to change when a forest floor litter layer develops, as per Sorenson et al. (2011). This will largely be determined by the quality of litter that established woody species will contribute.

4.3 - Amendments

In specific circumstances, amendments may need to be used to condition soils for plant establishment. These are likely extremely nutrient poor or degraded sites. However many plant communities have adapted to nutrient-poor conditions and can regenerate without additional inputs.

4.3.1 - Fertilizer

Increased nitrogen availability following fire disturbance contributes to vegetation reestablishment in natural upland forests, but the conditions created on reclaimed sites with inorganic fertilizer additions amplifies this considerably above the spectrum to which native plants have adapted. In these scenarios, N is disproportionately available to plants thereby affecting plant community composition (Canadian Natural Resources Ltd. 2013) (Figure 6.3). In my first study, fertilization at 100 kg ha $^{-1}$ dramatically increased N availability above the observed values in natural benchmarks. Although fertilization does have positive effects on plant growth, it may be counter-productive in establishing native ecosystems with tightly cycled nutrients. Nitrogen fertilization has been found to express negative effects on fungal community diversity and may promote establishment of aggressive plant species that out-compete the desired native species (Frey et al. 2004). Instead of scheduled fertilizer application, reclaimed sites should be individually evaluated by ecosite type using ecologically relevant measurements of available nutrients, to determine whether increased fertility is appropriate. In many cases, native pioneer species can colonize degraded soils and initiate soil processes without needing amendments (Polster 1989). In this way, successional reclamation uses pioneer species (like *Salix spp*. and *Alnus spp*.) for future establishment of climax species by conditioning soils. If fertilization is deemed suitable, my study suggest that soil P availability may be more limiting in PM soils than N, and therefore fertilizer prescriptions should account for this.

Typically, forest vegetation responds to N fertilization. Pinno et al. (2012) found that trembling aspen responded greatest to N-P-K fertilizer over just P-K or N alone and that PM had similar growth potential without fertilization. Therefore, in cases where suitable FFM is not available for upland reclamation, PM provides a secondary option that could alleviate the need for fertilization on severely degraded soils. This could reduce reclamation costs and eliminate potentially adverse effects associated with fertilizer use. Fertilizing forest soils with inorganic N has been shown to alter microbial community composition, lower F:B ratios, reduced ectomycorrhizal diversity (Frey et al. 2004). This could be due to fungi being poor competitors under high N availability (Fog 1988), or from indirect effects controlled by vegetative communities (Allison et al. 2007).

Additionally, after initial available N pulse following wildfires, boreal systems are typically N limited until they reach what is called retrogression; which happens after ecosystem productivity begins to decline. This inflection point occurs when P becomes more limiting than N (Lagerström et al. 2009, Menge et al. 2012). In my study, it seemed PM may be lacking P and is therefore already the limiting nutrient (Rowland et al. 2009). Instead of prioritizing nutrient requirements based on N availability, it may be more prudent to provide sufficient P to align with natural progression of nutrient limitations.

4.3.2 - Charcoal

Black carbon amendments are receiving greater attention as a conditioner to improve degraded soils. This has predominantly been studied in agricultural soils, however there are potential benefits to its use in reclamation. Naturally present black carbon in the form of charcoal is a by-product of wildfire. Replacing a similar material on reclaimed sites could re-create greater similarity in edaphic nutrient conditions as this will usually raise pH, sorb nutrients and organic matter due to its high surface area, provide refuge from predation for specific microbial populations, enhanced water holding capacity and have the potential to slowly release nutrients in years to come (Preston and Schmidt 2006). Contrasting the differences between, and perhaps creating conditions similar to post-fire disturbances may aid in expediting or creating more comprehensive land reclamation. Zackrisson et al.(1996) demonstrated the importance of charcoal in boreal ecosystems, whereby growth of certain species may be depressed due to an abundance of phenolic compounds in the soil. When amended with charcoal, the allelopathic compounds in soil were sorbed resulting in reduced reactivity which promoted the establishment of understory species like mosses and ferns. This could be beneficial in enhancing understory regeneration from reclaimed forest floor material, creating greater biodiversity. Contrary to my hypothesis, I found that broadcasting crushed charcoal at 2,000 kg ha $^{-1}$ did not significantly influence nutrient availability. Soil microbial qCO_2 was altered with charcoal addition, however this is likely a result of soil being thoroughly mixed with the amendment during sampling and lab analysis. Despite this, black carbon amendments may have a place in specific circumstances in oil sands reclamation. Dry, coarse-grained soils that are nutrient poor and have poor water holding capacity, like those in Chapter 3.0, could benefit from the aforementioned properties bestowed by black carbon additions. Additionally, effects will likely be more pronounced if black carbon is incorporated into soils, and applied at higher rates.

Burning organic matter in an anaerobic environment (pyrolysis) is a method which not only produces a form of black carbon, but may also be used for energy production by creating syn-gas or bio-oil. The practice of burning un-merchantable timber following harvest wastes a potentially valuable resource since most of the carbon is immediately released into the atmosphere. One suggestion would be to use this material as a feedstock for pyrolysis, which would decrease CO_2 emissions, provide another source of energy and create a soil amendment as a by-product.

4.4 - Topsoil Depth

Minimum capping depths to create separation between adverse materials should be maintained to reduce potential contaminant (salts) migration to surficial horizons. Topsoil application depths should not be restricted to a set framework, but rather be based on topographic position. Additionally, application depth is critical to evaluate because it determines the amount of salvaged soil resources that are allocated to final landforms. If the application depth is too deep, there will be insufficient coverage of topsoil on the final landscape. Conversely, if depths are too shallow, desired reclamation goals may not be met as vegetation struggles to establish.

Brunisolic soils are considered juvenile due to short time of development and weaker influence of pedogenic factors. However, the observed shallow depth of pedogenic development in the harvested treatment is still capable of supporting a recovering jackpine stand – a vegetative community accustomed to dry, nutrient poor soils. Biological contributions rapidly diminish with increasing depth and abiotic factors likely become more important contributors to overall plant productivity, especially water retention, quality and movement. This is reflected in my research, which suggests that deep applications of FFM do not provide statistically different nutrient profiles from shallow applications and microbial community function shares greater similarity to the harvested benchmark. Deep FFM did create greater similarity in microbial community structure than shallow, but microbial diversity introduced from the shallow application may translocate deeper into the soil profile. Moreover, if we are aiming to return a functioning ecosystem, microbial community structure may not be as important due to functional redundancy. From visual inspections it appears that shallow applications support less vegetation in the second year of growth (Figure 6.4). This makes sense since fewer plant propagules would have been transported per unit are of salvaged material. However, when presented with the alternative of placing PM on these sites, shallow FFM has much more vegetative cover. Since shallow applications of FFM do not differ substantially from deep, it may be prudent management to favour shallow applications in order to cover a greater surface area of the final reclaimed landscape. Since regulatory requirements for salvage of FFM have only been instituted within the last 10 years, PM will be the dominant soil type used in reclamation, making up roughly 80 % of Syncrude Canada's closure plan. Closure plans define most of the reclaimed landscape as upland topography, while lowlands will be predominantly end-pit lakes – effectively removing most of the original peatland component present in these ecosystems (Rooney et al. 2012).

Similarly to FFM, shallow PM shared a greater similarity with the harvest analogue than deep applications, most notably so in microbial community function at 10 – 20 cm depth. Since this material does not provide plant propagules native to upland ecosystems like FFM, deeper applications likely do not result in greater vegetative cover in the initial growing seasons following reclamation. Thin layers of PM could act as a surrogate upland organic horizon (LFH) on reclaimed sites, providing additional nutrition to inherently nutrient poor subsoil. Insulation will also be less and may defrost sooner than in deep applications. One benefit of PM in upland positions is that it could provide greater resilience to drought conditions than sandy soils with low water holding capacity.

Depths of natural soils widely vary depending on length and intensity of contributing soil forming factors. On the ASCS, most of these factors are held constant so that uniform depths offer comparability. When extrapolating this information to a broader context, it may be necessary to change topsoil application depths according to topographic position, precipitation inputs and intended vegetative communities. What is true for dry ecosites may not be true of sites with greater productivity and water availability. These sites will likely possess natural soils with greater pedogenic development. Furthermore, application depths will likely need to vary with stockpile residence time. Beneficial attributes of topsoil materials will dissipate with storage time and therefore similar studies to the ASCS need to be completed using stockpiled materials.

4.5 - Measures of Reclamation Success

Certification is achieved through rigorous monitoring and testing programs to allow for reasonable assurance that ecosystem processes have been established and a stable and self-sustaining system requiring no further management inputs is created. Only one site has received certification and release of liability to date, with reclamation beginning in 1983 and certification in 2008. Since this process takes decades, it is valuable to develop measures to assess restoration trajectories at early stages. Assessment for reclamation certification of the sites in these studies will likely require decades of monitoring. Measures of reclamation success are currently unclear since “equivalent land capability” of an ecosystem is poorly defined in provincial regulations. Regulators and extraction companies would benefit from standardized land capability assessments which base decisions of reclamation success on appropriate benchmarks and comprehensive ecological measurements.

Until recently, the LCCS was a tool used to rate soil productivity based on nutrient and moisture regimes. This rating system provided a good framework for straight-forward land assessments, but did not provide adequate sensitivity. This is likely related to the relatively simple measures of soil nutritional qualities that were employed as proxy measurements of biological carrying capacity. Soil chemical analyses included TOC, TIN, EC, SAR and pH. While there is merit in these measurements, TOC is not a good indicator for reclamation success since it has similar values between reclaimed and natural sites (Turcotte et al. 2009, Norris et al. 2013a). This is likely due to the use of PM as topsoil which is predominantly of organic origin and may be different when comparing FFM to natural since it is predominantly mineral. Additionally, the quality of organic matter has profound implications to soil nutrient dynamics and the microbial communities governing nutrient availability. Similarly, TN includes organic-N and mineral-N in solution and ionically bound and therefore is not all immediately available to organisms. Soil sampling for the LCCS is done at three depth intervals: 0 – 20 cm, 20 – 50 cm and 50 – 100 cm. While it is valuable to segregate sampling based on depth, this sampling protocol often admixes different materials and horizons which dilute the activity in the upper solum.

Unlike agronomic crops, little information exists about ideal soil conditions for establishing native boreal vegetative biodiversity. Instead, holistic approaches should be undertaken which include components of soil biology. Integrating soil biological measurements could provide greater discriminatory power that was lacking in the LCCS. Soil ecology, which drives major ecosystem processes, presents sensitive measurements of ecosystem rehabilitation (Rowland et al. 2009). This would involve selecting appropriate natural benchmarks and determination of acceptable ranges for reclaimed soil. Contemporary analytical techniques have made biological measurements easier and more cost effective due to an increase in soil biological assessments in recent years.

The importance of incorporating soil biological components into reclamation trajectory and certification is becoming more apparent in mine reclamation literature for in-situ measurements and laboratory analyses. Phospholipid fatty acid and CLPP analyses show potential as relatively quick and cost effective measurements of microbial community structure and function, however these also have limitations. Both can be extremely variable, not only by treatment type, but also by seasonality. Greater specificity generated by metagenomics analyses may play a role too but are currently expensive, time consuming and difficult to interpret. Soil respiration and enzyme activity have been incorporated into a Mine Soil Quality Index (MSQI) in India (Mukhopadhyay et al. 2014). Several studies of bauxite mine reclamation in Australia and Jamaica have used CLPP for assessing soil microbial function (Lewis et al. 2010, Banning et al. 2012). Banning et al. (2012) assessed CLPPs in bauxite mined jarrah (*Eucalyptus marginata*) forest soils and found they did not fully recover after 26 years, despite a return of soil heterotrophic activity within the first 13 years following reclamation. A comprehensive assessment of microbial properties and activity in reclaimed open-cast coal mines in Czech Republic, using microbial respiration, biomass and cellulose decomposition was provided by Helingerová et al. (2010). These types of assessments provide greater discriminatory power than that of the LCCS due to the sensitivity of microbial communities to their surrounding environments. Multivariate statistical approaches can help with interpretation of complex data sets and are relatively simple to use. Incorporating these measurements into a rating framework similar to the LCCS could improve upon current assessment techniques.

4.6 - Conclusion

Reclamation research has progressed greatly since its inception and helps to mitigate deleterious environmental impacts imposed by resource extraction activities. Although industry is slow to adopt research findings that may not be economically beneficial, the cooperation between industry, government and academia has improved and has led to overall improvement in practice. This is especially important for major localized disturbances like oil sands mines. It is my hope that this research contributes to the improvement of land management in the AOSR.

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6.0 Appendix

Table 6.1 – Maximum absorbance capacity for PRS™ probes (Western Ag Innovations 2015).

Units: µg 10 cm^{-2}

Cl^-	5288	NO_3^--N	2088	K^+	9273	Cu^{2+}	9731	Fe^{3+}	8552
Na^+	5455	NH_4^+-N	3320	Ca^{2+}	4753	Mn^{2+}	8412	Al^{3+}	4131
SO_4^--S	4782	$H_2PO_4^-$-P	4620	Mg^{2+}	2883	Zn^{2+}	10012	$B(OH)_4^{3+}$-B	1600

Table 6.2 – Distribution of vascular plant species found in natural plots within the soil types of RA-1 (Canadian Natural Resources Ltd. 2013).

	Species found in natural plots	Species only found in natural plots	Species in RA1 and natural plots	Species in PMM and natural plots	Species in LFH and natural plots
Blocks	*G or H*	*G or H*	*(G or H) and (A, B, C, D, E or F)*	*(G or H) and (B, E or F)*	*(G or H) and (A, C or D)*
Forbs	17	6	11	4	11
Graminoids	3	0	3	3	2
Low shrubs	3	3	0	0	0
Shrubs	6	4	2	2	2
Trees	2	1	1	1	1
Total	31	14	17	10	16

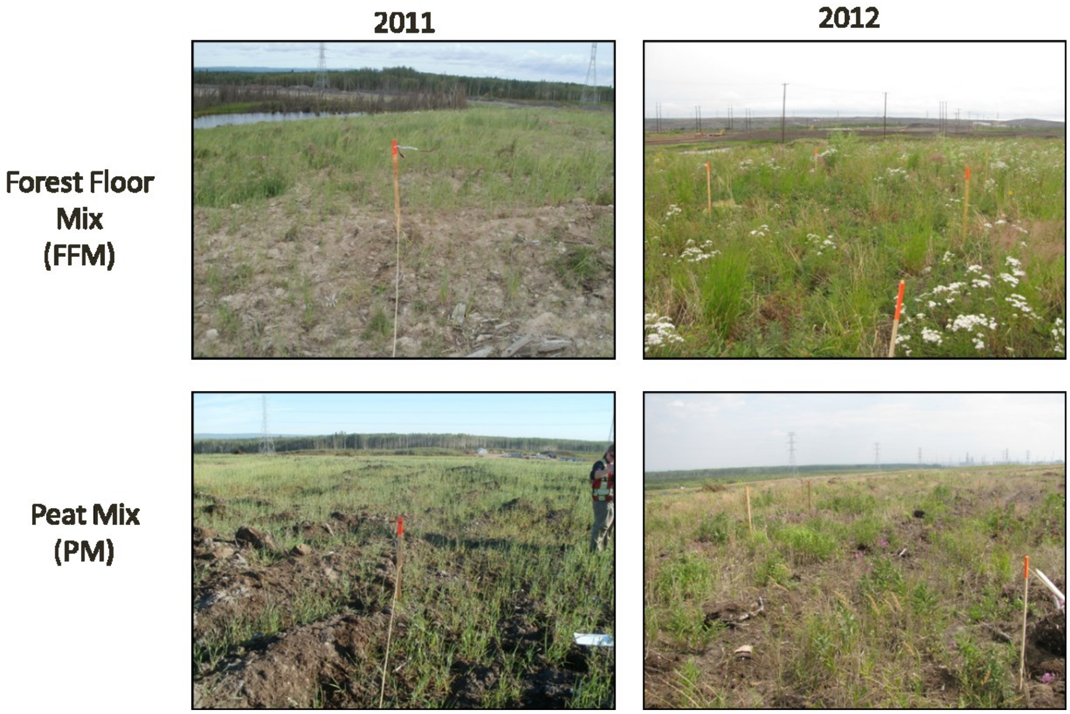

Figure 6.1 – Pictures taken in August 2011 and 2012 of unfertilized FFM and PM at Canadian Natural Resources Ltd.'s Reclamation Area-1.

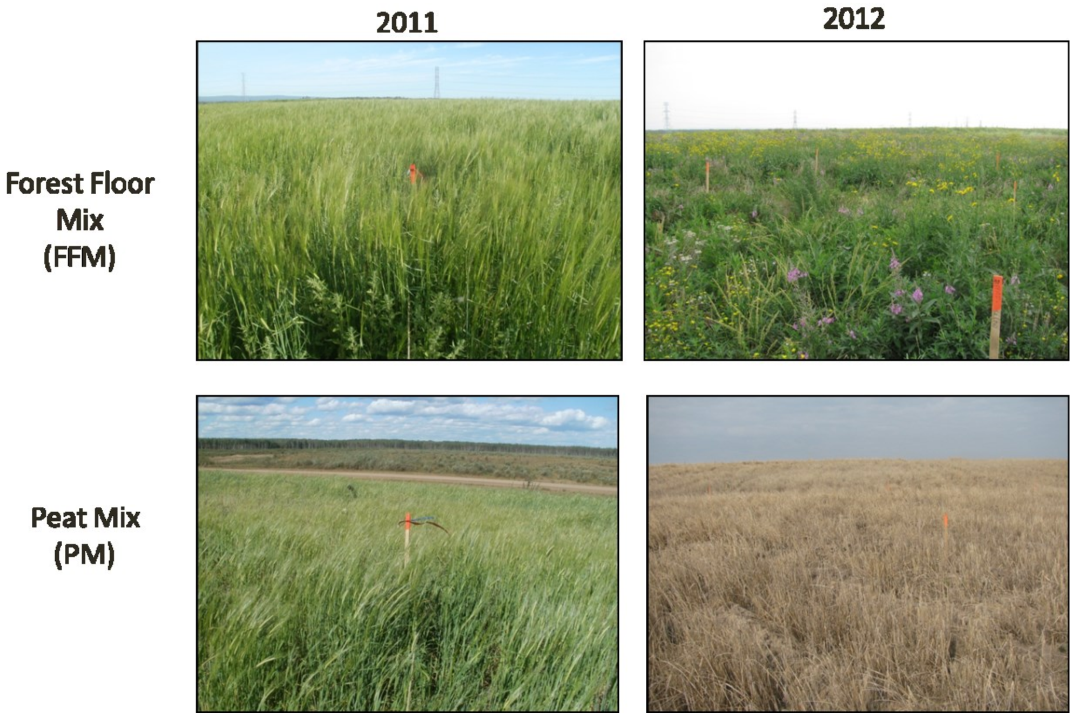

Figure 6.2 – Pictures taken in August 2011 and 2012 of fertilized FFM and PM at Canadian Natural Resources Ltd.'s Reclamation Area-1.

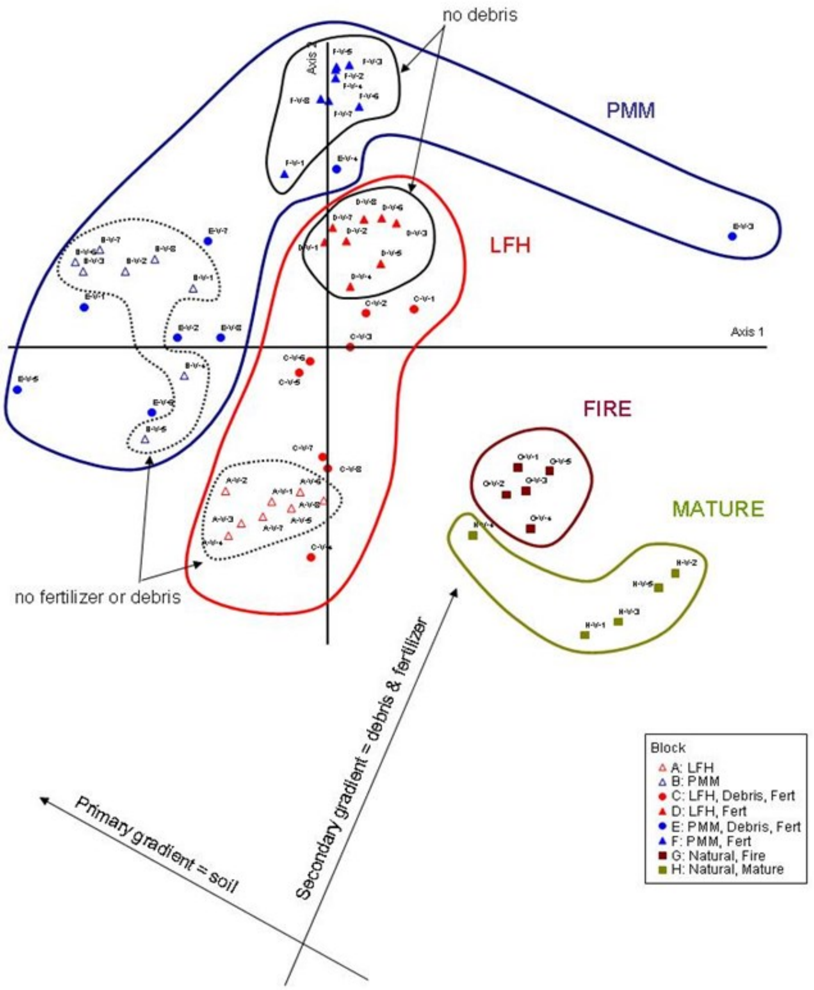

Figure 6.3 – Ordination bi-plot of vegetation communities on reclaimed sites and natural analogues at Canadian Natural Resources Ltd.'s Reclamation Area-1 (Canadian Natural Resources Ltd. 2013). The term LFH is synonymous with FFM in this case.

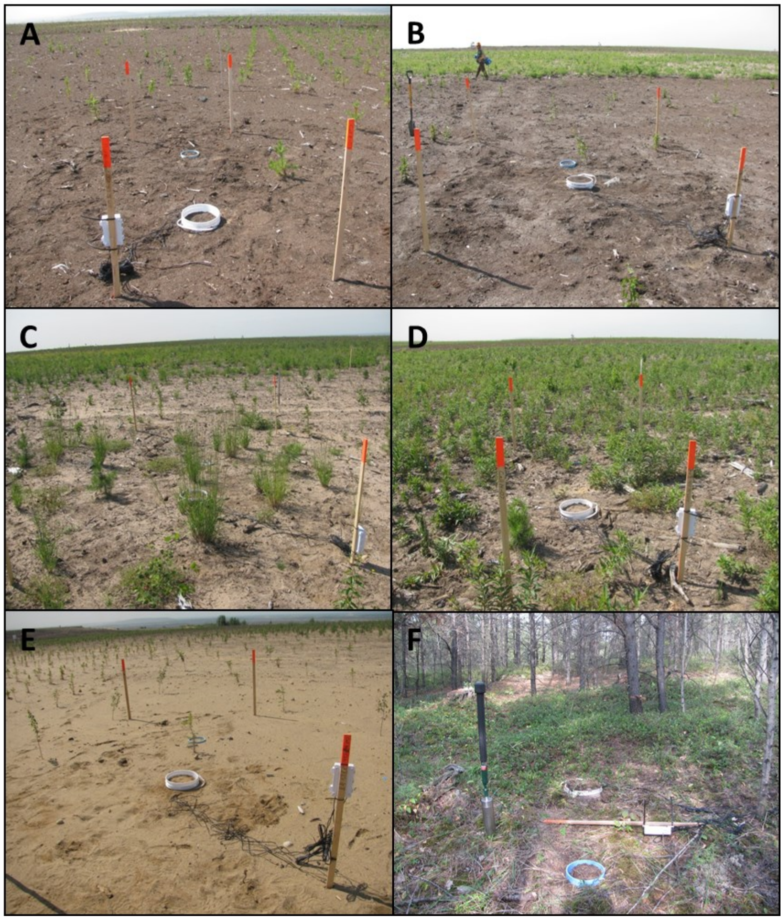

Figure 6.4 – Pictures of plots on the Aurora Soil Capping Study immediately after setup for Shallow PM (A), Deep PM (B), Shallow FFM (C), Deep FFM (D), Control (E); and during take-down for Harvest (F).

Figure 6.5 – Perforated 20 liter polyethylene pails for backfilling sampling pits that were instrumented with ionic exchange membranes, temperature and moisture sensors in the Aurora Soil Capping Study.

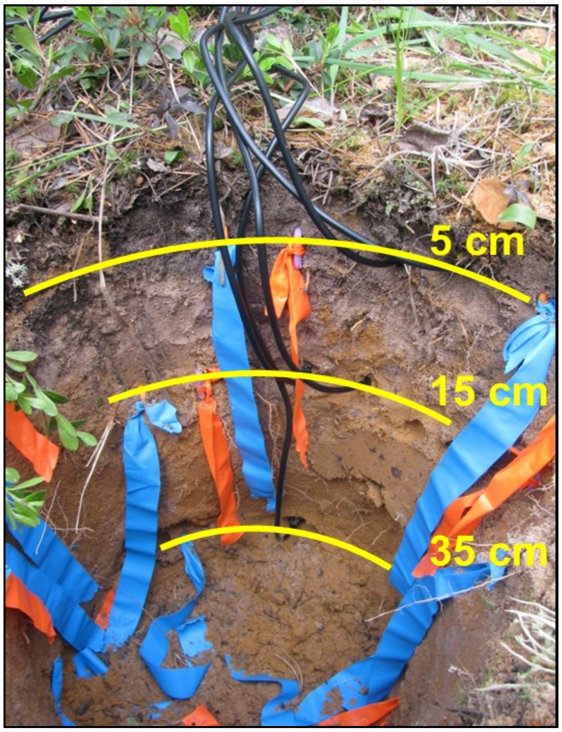

Figure 6.6 – Pit setup for PRS™ probes and temperature and moisture sensors in the Aurora Soil Capping Study.

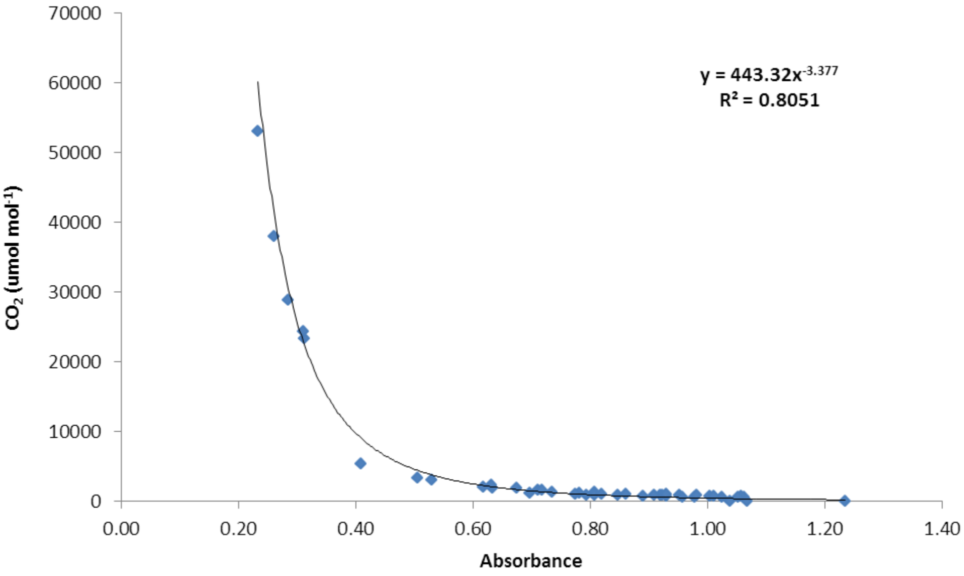

Figure 6.7 – Relation of absorbance values measured at a wavelength of 570 nm on a microplate reader from detection wells incubated with several soil types for 5 time periods, used to measure community level physiological profiles.