

Effects of Stockpiling on Soil Physical Properties and Soil Carbon

by

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## ABSTRACT

The boreal forest is a major ecosystem in Alberta, subject to a number of anthropogenic disturbances such as oil and gas extraction. Prior to disturbance, top soils are salvaged for use in future reclamation projects; however, a large portion of this soil ends up in stockpiles, undergoing changes to soil physical properties, aggregate and carbon distributions, as well as carbon dynamics. This study sought to assess changes in these parameters, relative to natural sites, to determine implications on the suitability of stockpiled topsoil for use in reclamation.

Sampling sites were established at an open pit mine and an *in situ* development in the boreal forest. Eight stockpiles and six natural sites were selected, with three soil pits being dug per site and three depths sampled per pit. Soil physical quality (SPQ) was assessed using basic soil properties, capacity-based measurements, and energy parameters as all of these variables relate to S, defined as the slope at the inflection point of a water retention curve. As such, S provides insight on the pore-size distribution of a soil, with larger values denoting higher SPQ.

Clay content and bulk density were higher in natural soils, whereas total soil carbon, S, capacity-based measurements, and energy parameters were higher in stockpiled soils. Significant differences between natural and stockpiled soils were present only in the two subsurface depths. Differences existed within natural soil profiles between the surface and two subsurface depths, while stockpiled soils remained similar across all depths. Higher levels of soil carbon, lower bulk densities, and lower clay contents in stockpiled soils were associated with the highest S values for sampled soils, supporting previous observations published in scientific literature. S was significantly correlated with most capacity-based measurements, supporting additional theories that link these parameters together. Energy parameters were also significantly correlated with S,

though lower values for these parameters are associated with higher SPQ. Together, natural soils had lower SPQ than stockpiles using basic soil properties, capacity-based measurements, and S, while outcomes using air and water retention energies were inconclusive.

Changes to aggregate size class and carbon distributions, carbon dynamics, and soil organic matter lability criteria were assessed at both the whole soil and individual aggregate scale. No significant differences in relative proportions of aggregate size classes were found between natural and stockpiled soils for any of the sampling depths, although natural soils did have higher proportions of both aggregate sizes. Higher basal respiration rates and  $\delta^{13}\text{C}$  values were found in natural soils, whereas stockpiled soils had greater total and light fraction carbon quantities, along with higher C:N ratios. Significant differences between natural and stockpiled soils were largely confined to the two subsurface sampling depths. Differences within natural soils were between surface and subsurface depths, whereas stockpiles remained uniform across all depths. Basal respiration rates significantly correlated with total carbon, light fraction carbon proportions, whole soil C:N ratios, and  $\delta^{13}\text{C}$  values. Similar trends were observed for each aggregate size.

The results of this study differed greatly from the majority of findings present in the current literature base and demonstrate the importance of looking at the physical, chemical, and biological components of soil quality together. In the case of this study, taking a more holistic approach allowed for the identification of lower biological quality in stockpiled materials, despite these same materials having higher soil physical and chemical quality regardless of sampling depth. Identifying these limitations, in turn, would then allow for a more thorough assessment on the suitability of stockpiled topsoil for use in future reclamation activities and the potential for reclamation success post-placement.

## **PREFACE**

This thesis is an original work by Kyle Stratechuk. No part of this thesis has been previously published.

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## LIST OF ABBREVIATIONS

<b>A<sub>a</sub></b>	Absolute air retention energy
<b>AW<sub>r</sub></b>	Relative air and retention energy ratio
<b>AOSR</b>	Alberta oil sands region
<b>AC</b>	Air capacity; calculated from the water retention curve
<b>FC</b>	Field capacity; calculated from the water retention curve
<b>CLPP</b>	Community level physiological profiling
<b>CNRL</b>	Canadian Natural Resources Limited; industry partner
<b>EC</b>	Electrical conductivity
<b>LFC</b>	Light fraction carbon
<b>LFH</b>	Litter, fibric, and humic layer; surface organic layer in forested environments
<b>NMDS</b>	Non-metric multidimensional scaling; multivariate statistical approach
<b>NRAL</b>	Natural Resources Analytical Lab; analytical lab at the University of Alberta
<b>PAWC</b>	Plant available water content; calculated indirectly from the water retention curve using field capacity and permanent wilting point values
<b>PWP</b>	Permanent wilting point
<b>RFC</b>	Relative field capacity
<b>SAGD</b>	Steam assisted gravity drainage; <i>in situ</i> method of oil extraction
<b>S</b>	Soil physical quality index
<b>SOM</b>	Soil organic matter
<b>SPQ</b>	Soil physical quality
<b>SWRC</b>	Soil water retention curve
<b>WHC</b>	Water holding capacity
<b>WR<sub>a</sub></b>	Absolute water retention energy

## I: GENERAL INTRODUCTION

### 1.1. BACKGROUND

The boreal forest is a major ecosystem in Alberta, covering 381,046 km<sup>2</sup>, or 58%, of the province (Downing and Pettapiece, 2006). In addition to its large geographic expanse, the boreal forest provides a significant number of ecosystem services including, but not limited to: provisioning of timber, fuelwood, and water, regulating climate, flooding, and disease, supporting soil formation, primary production, and nutrient cycling, while also creating cultural importance in the forms of education, recreation, and aesthetics (Brandt et al., 2013). The ability of the boreal forest to provide these ecosystem services varies in accordance with the type, size, frequency, and severity of disturbance it is subjected to. The boreal is a disturbance-driven ecosystem, adapted to a variety of natural disturbances such as climate alterations, fire, pests, or pathogens (Brandt et al., 2013). However, anthropogenic disturbances in the boreal are growing, especially in relation to the oil and gas industry.

Oil sands development in Alberta's boreal forest is one of the province's largest industries due, in part, to the estimated 1.6 trillion barrels of oil present in three large oil sands deposits (Rowland et al., 2009; Hein, 2006). Of these three formations, it is the Athabasca oil sands region (AOSR) that is the largest and currently most developed, characterized by both open pit mining and *in situ* extraction operations (Alberta Department of Energy, 2007). About 20% of Alberta's oil sands reserves are estimated to be recoverable utilizing current open pit mining technologies, with the rest being recoverable using *in situ* methods (Alberta Energy Regulator, 2017; Hein, 2006).



Therefore, open pit mining has been, and will continue to be, a major anthropogenic disturbance in the boreal forest, impacting all ecosystem components that historically existed there. This is largely tied to mining operational practices, where natural soils and vegetation are removed over a large area to mine oil sands ore (Rowland et al., 2009). Removal occurs in multiple steps, one key step being the provincially mandated salvaging of topsoil. Typically targeted at the organic LFH and mineral topsoil layers, soil salvage attempts to conserve these nutrient rich soils, with favourable physical properties and an inherent viable seed bank, for later use in reclamation (Government of Alberta, 2013).

*In situ* methods, such as steam-assisted gravity drainage (SAGD) operations, represent a large and growing footprint in the boreal forest. In contrast to open pit mining, SAGD operations do not require open excavations to access oil sands ore; instead, paired horizontal wells are drilled deep into the formation. Steam is injected to decrease the viscosity of the oil, allowing it to drain into collection wells from the surrounding geological material and be transported to the surface for processing (Jordaan et al., 2009; Ezeuko et al., 2012). Because of the lack of large, open-pit excavations, SAGD disturbances have a smaller aerial footprint, but the construction of exploratory, extraction, and monitoring well pads, as well as other associated infrastructure, results in a more fragmented forest environment (Jordaan et al., 2009). Operators are expected to comply with provincial legislation and, like open pit mining, salvage LFH and topsoil during facility construction for use in future reclamation (Government of Alberta, 2013).

Once salvaged, there are two methods of storage that can be used in reclamation practice for this soil material: direct placement and stockpiling. Direct placement is the preferred practice as it entails using the soil immediately following salvage. In this way, soil structure, organic matter, seed viability, nutrients, and soil biota are better conserved, facilitating quicker revegetation and

re-initiation of soil processes post-reclamation (MacKenzie, 2011). Stockpiling, however, is often an inevitable storage method, especially at project onset when permanent reclamation areas are not yet available or are too small to accommodate large volumes of salvaged material (Naeth et al., 2013). As such, stockpiling requires transporting excess salvaged soil to pre-determined locations and storing it until needed. Stockpiles can be separated based on the type of soil substrate (ie. forest floor/topsoil, peat mineral mix, subsoil, etc.) and, within mineral soil materials, coarse and fine textures (MacKenzie, 2011).

Regardless of which storage and placement method is used, alterations to soil properties will occur as a result of the initial salvaging process, a disturbance of relatively large magnitude. Numerous studies have documented the effects of other large-scale disturbances, such as repeated conventional tillage in agricultural settings, on a number of soil properties. To name a few, these studies have found that with increasing tillage intensity, soil aggregation declines and organic matter turnover is increased (Six et al., 1998). Soil aggregation and organic matter turnover are linked to each other because disruption/destruction of soil macroaggregates increases the proportion of microaggregates and free silt and clay particles (Hunter and Currie, 1956; Wick et al., 2007). In turn, the organic material originally contained in the macroaggregates, mainly light fraction carbon that is more labile, is made available to microorganisms for decomposition (Tisdall and Oades, 1982; Cambardella and Elliott, 1993; Six et al., 1998, 2000). This increased loss of soil organic matter makes these soils increasingly susceptible to further structural damage, leaving a soil with poorer physical quality and an aggregate size distribution dominated by carbon depleted microaggregates and unassociated free silt and clay (Hunter and Currie, 1956; Abdul-Kareem and McRae, 1984; Six et al., 2000).

## 1.2. OBJECTIVES

While the effects of soil tillage on soil physical quality, aggregation, as well as soil carbon are better understood, the impacts of soil salvage and subsequent stockpiling on these same parameters have not been as extensively studied. Of the studies conducted on soil stockpiles, the majority of these have occurred outside of Canada, where local climates and soils can vary greatly from those seen in the AOSR. As well, many of these studies have a small number of sampled stockpiles, or sampling depths are confined to the stockpile surface. Therefore, the overall objectives of this study are to:

1. Assess the impacts of soil stockpiling, over a variety of depths, on a number of basic soil properties and overall soil physical quality in comparison to natural reference sites in the Alberta oil sands region.
2. Assess the impacts of soil stockpiling, over a variety of depths, on aggregation, soil carbon distributions, and carbon dynamics in comparison to natural reference sites in the Alberta oil sands region.

These research objectives will be addressed using two data chapters; the first will explore soil physical quality in natural and stockpiled soils, while the second will focus on soil aggregation and soil carbon. Both chapters will outline the field and laboratory methods used to collect samples and generate data, the statistical process for analyzing this data, as well as a discussion on the implications of the results for each chapter. Both chapters will be concluded with a summary of the results and some implications. The two chapters will then be synthesized with a discussion of some project limitations and potential future research that could augment the results of these studies.

## **II: INFLUENCE OF STOCKPILING ON BASIC SOIL PARAMETERS AND SOIL PHYSICAL QUALITY**

### **2.1. INTRODUCTION**

Land reclamation in Alberta is governed by the concept of equivalent land capability, meaning that reclaimed sites need to be capable of supporting similar land uses as to what existed there prior to disturbance (Government of Alberta, 1993). Using appropriate soil material is crucial for successful reclamation and stockpiled soil is an important resource to accomplish this.

Stockpiled soil is comprised of salvaged topsoil, collected prior to the onset of industrial operations, which was not able to be directly placed (MacKenzie, 2011). Major alterations can occur within stockpiled soil while it remains in storage, affecting soil quality.

Soil quality can be divided into three components: physical, chemical, and biological, all of which influence the ability of a soil to provide a diverse array of ecosystem functions and services (Bacher et al., 2019). However, while all three components are important to use when assessing overall soil quality, soil physical quality (SPQ), most influenced by soil structure, demonstrates cascading effects on soil chemical and biological components (Dexter, 2004). Improvements in soil structure, which directly influences water infiltration, drainage, aeration, organic matter, and general plant rootability, would also improve overall SPQ and the suitability of stockpiled soil for use in reclamation (Dexter, 2004; Rabot et al., 2018).

#### **2.1.1. Soil Physical Quality Theory**

A number of different metrics can be used when trying to assess SPQ. Capacity-based parameters such as field capacity (FC), plant available water content (PAWC), air capacity (AC), and macroporosity have been extensively used in agricultural settings, being seen as proxies for

overall SPQ because all of these properties are expressions of soil pore volume, soil function, and, therefore, soil structure (Reynolds et al., 2009).

An alternative to these capacity-based indicators is a pore-size distribution-based index, such as the S index. The S index is derived from a soil water retention curve (SWRC), representing the inflection point (the potential at which the second derivative of the SWRC is equal to zero) of the curve (Dexter, 2004). As such, the S index considers two major regions of a SWRC; the draining of structural pores (from saturation to the inflection point) and the draining of textural pores (after the inflection point). Generally, textural pores are not affected by soil management; however, structural pore distributions can be altered following disturbance and subsequent management practices such as soil salvage and stockpiling (Richard et al., 2001). As soil structure affects many important soil physical properties, alterations to structural pores can have a direct impact on many soil processes, which would be captured by changes to the S index (Dexter, 2004; Rabot et al., 2018).

The S index incorporates the effects of three basic soil parameters as the primary influencers of SPQ: soil texture (i.e., clay content), compaction (i.e., bulk density), and organic matter content (i.e., total carbon) (Dexter, 2004). Overall, increases in clay content and bulk density, and decreases in total soil carbon, would result in declines in S and SPQ. Due to the cascading effects of soil texture, bulk density, and total soil carbon on aggregation and soil structure, for medium- to fine-textured soils, S values are often also significantly correlated with capacity-based measurements such as FC, PAWC, and AC (Reynolds et al., 2009).

One benefit of using the S-index is that it should allow for SPQ comparisons between different soils (Bacher et al., 2019). This could be important for land reclamation, especially when trying

to assess differences between heavily disturbed and natural forest soils. However, there are limitations in using S as the sole measure of assessing SPQ, primarily because it is not sensitive enough to capture the full variability of a SWRC (Bacher et al., 2019). Therefore, more dynamic variables are proposed by Armindo and Wendworth (2016) and Bacher et al. (2019) which include absolute air energy ( $A_a$ ) and absolute water retention energy ( $WR_a$ ). Both  $A_a$  and  $WR_a$  better capture the full variability of a SWRC and so should allow for a better spatial and temporal assessment of soil structure for both undisturbed and heavily disturbed soils (Armindo and Wendroth, 2016; Bacher et al., 2019). Since these indicators, together, represent the entire area underlying a SWRC, they can be used in conjunction with capacity-based indicators such as those proposed by Reynolds et al. (2009).

### **2.1.2. Current State of Understanding of Soil Physical Quality in Stockpiles**

Study results for the above-mentioned physical parameters vary greatly in stockpiled soils, depending on the variable being considered. With regards to bulk density, Abdul-Kareem and McRae (1984) found that, regardless of soil texture, bulk densities in 1.5 to 7-year old stockpiles were higher than those in natural soil counterparts. However, bulk density measurements were confined to the surface of each stockpile due to sampling difficulties at depth. Similar bulk density increases were observed for younger stockpiles (< 2 years old) and were attributed to the compacting effects of heavy equipment (Gee and Bauer, 1976). Some studies observed changes in bulk densities with salvaged soils; however, these changes were noted only after stockpiled soils were replaced on the landscape (Wick et al., 2009).

It has been noted in Hunter and Currie (1954) that upon stockpiling, total soil carbon decreases, although the time frame of this decline differs depending on the study. Visser et al. (1984) found

that total carbon significantly declined immediately following stockpiling due to the dilution of topsoil with underlying subsoil material during salvage and storage (Visser et al., 1984; Wick et al., 2009). Other studies have reported similar declines in total carbon, though these were attributed to microbial decomposition of soil organic matter (Hunter and Currie, 1956; Abdulkareem and McRae, 1984; Wick et al., 2009).

Increases in bulk density and loss of organic carbon in stockpiled soils can create a detrimental feedback loop with respect to soil structure; ultimately, aggregate stability will decline over time, shifting the pore size distribution of the soil towards a micropore-dominated substrate. This, in turn, will have direct implications on water infiltration, retention, and availability (Boyle et al., 1989; Dexter, 2004). Plant-available water content in stockpiles are lower than in natural soil counterparts, but little work has been done to quantify any additional hydraulic properties in stockpiled material (Schwenke et al., 2000b).

A number of studies assessed the impact of large-scale disturbance on other soil hydraulic properties. Banning et al. (2008) found reduced water holding capacity in disturbed, post-mining soils when compared to undisturbed soils under forest vegetation. Soils disturbed during pipeline construction in Alberta's boreal forest also had reduced water retention and infiltration rates (Soon et al., 2000). In both cases, reductions in hydraulic capability could be attributed to the preferential formation of micropores (Lynch and Bragg, 1985).

The above literature shows that some work has been done on characterizing the basic soil properties that influence the S index. However, little work has been done in linking these variables to the S index for stockpiled and natural soils in the boreal forest. Likewise, while some work has been done looking at water infiltration and retention in stockpiled and disturbed boreal

soils, the derivation of capacity-based indicators, with the exception of PAWC, and application of air and water retention energies, is largely lacking in the literature.

## **2.2. RESEARCH OBJECTIVES**

The objective of this chapter is to better quantify the impacts of stockpiling on basic soil properties and general SPQ. The focus will be on mineral top soils from the Alberta boreal forest, particularly those associated with both open pit mining and SAGD operations. Soils will be assessed using alterations in basic soil properties and water retention parameters, between natural and stockpiled soils, over a variety of depths.

### **2.2.1. Research Questions**

Throughout this chapter, the following research questions will be explored:

1. To what extent do basic soil properties, such as clay content, bulk density, and total soil carbon, differ between natural and stockpiled soils over a range of depths?
2. Do changes in these basic soil properties and related capacity-based measures, such as field capacity (FC), plant available water content (PAWC), air capacity (AC), and macroporosity, result in similar changes to SPQ indicated by the S index?
3. How do alternative SPQ indicators, such as air and water retention energies, compare to the S index values for the sampled soils?

### **2.2.2. Hypotheses**

Clay contents are expected to be higher in natural soils than in stockpiles, especially at depth, due to the presence of illuviated clays in natural subsoil horizons and a subsequent lack of clays in salvaged topsoil due to previous eluviation processes. Bulk densities and total soil carbon are



expected to be higher in stockpiled soils than in natural sites, regardless of depth, due to the compaction effects of soil salvage, transport, and storage and the integration of LFH during these disturbances.

Overall, natural soils are expected to have higher FC, AC, PAWC, and macroporosity values than stockpiles, largely due to the destructive influence of soil salvaging on soil structure and aggregation, shifting pore-size distributions in stockpiles towards a micro-pore dominated profile. Natural soils, therefore, are expected to have a larger S index, and lower air and retention energies, than stockpiled sites.

It is expected that no differences will be observed for clay contents, bulk densities, total soil carbon, FC, AC, PAWC, macroporosity, the S index, air energy, and retention energy between any of the stockpile depths, due to the homogenization of soil material during salvage, transport, and placement. In contrast, natural soils are expected to see an increase in clay contents and bulk density with increasing depth due pedogenic eluviation and illuviation of clays. Total carbon is expected to decline with depth, due to both the continual decomposition of deep soil organic matter and poor levels of LFH integration in subsoil layers. Increasing clay contents and bulk densities, with declining levels of total carbon, will likely be accompanied by decreases in AC, PAWCs, macroporosity, and S, along with increases in FC, air energies, and retention energies in natural soils, due the cascading influence of soil texture and organic material on aggregation, soil structure, water retention and SPQ.

## **2.3. METHODS**

### **2.3.1. Field Site Description**

This study took place at CNRL Horizon and Wolf Lake oil sands operations.

### 2.3.1.1. CNRL Horizon

CNRL Horizon is an open-pit mine located approximately 75 km northwest of Fort McMurray, Alberta. It is located in the Central Mixedwood Natural Subregion, with an upland overstory consisting primarily of trembling aspen (*Populus tremuloides* Michx.) and white spruce (*Picea glauca* (Moench) Voss), and a lowland overstory mainly represented by black spruce (*Picea mariana* Mill.) (Downing and Pettapiece, 2006). Upland soils in the region are primarily Gray Luvisols on fine textured parent materials, or Eutric and Dystric Brunisols on coarse textured parent materials. Lowland soils are typically Organic (Downing and Pettapiece, 2006).

Selected stockpile and natural sites at CNRL Horizon can be found in Table 2.1 and Figure 2.1. All stockpiles, created at Horizon through the use of excavators and heavy haulers, were classified as topsoil and had a crest greater than 50 m in length. Vegetation composition varied from predominantly herbaceous invasive species to a mix of herbaceous invasive and woody/herbaceous native species. All natural reference sites had an upland canopy of trembling aspen and white spruce. The mining footprint at Horizon required natural sites to be located approximately 5 km from stockpiles to reduce anthropogenic effects on soils and vegetation.

**Table 2.1: Stockpile and natural reference sites for the CNRL Horizon study area. All stockpiles were labelled using the operator’s identification number (ie. RMSx). -J or -S codes were amended to denote younger and older ages of stockpiles, respectively, that had the same identifier. Mature sites were labelled by plot number (ie. Mature x).**

Stockpile	Sampling Area	Site Type	Age (years)	Location (latitude, longitude)
RMS4-J	CNRL Horizon	Stockpiled	0.5	57.314, -111.866
RMS5-J	CNRL Horizon	Stockpiled	1.5	57.375, -111.944
RMS4-S	CNRL Horizon	Stockpiled	5	57.314, -111.865
RMS5-S	CNRL Horizon	Stockpiled	7	57.378, -111.956
Mature 4	CNRL Horizon	Natural	Mature	57.387, -111.870
Mature 13	CNRL Horizon	Natural	Mature	57.388, -111.869



**Figure 2.1: Stockpile and natural site locations at CNRL Horizon.**

### **2.3.1.2. CNRL Wolf Lake**

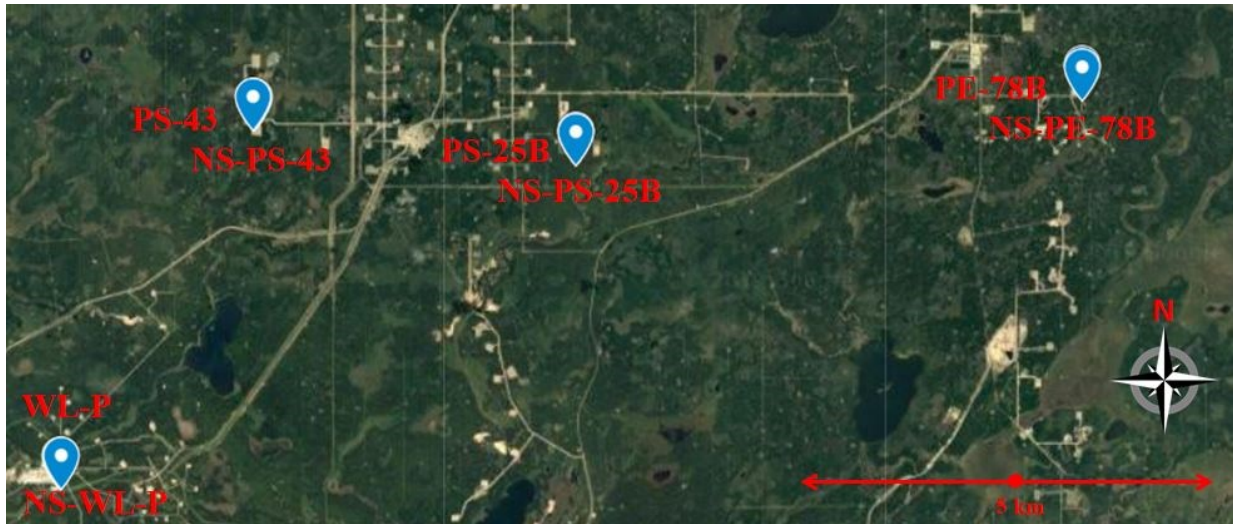
CNRL Wolf Lake is a SAGD development about 55 km north of Bonnyville, Alberta, located in the Central Mixedwood Natural Subregion with natural vegetation and soils similar to those described for Horizon (Downing and Pettapiece, 2006).

The same criteria as Horizon were used at Wolf Lake when selecting stockpiles and natural sites. Stockpile creation differed at Wolf Lake, relying primarily on dozers to scrap off topsoil layers at well pad locations. Vegetation communities on stockpiles also differed at Wolf Lake, with the majority of stockpiles either being mix of herbaceous invasive and woody/herbaceous native species or predominately grass covered. The more fragmented nature of SAGD disturbances at Wolf Lake allowed one stockpile and natural site to be situated adjacent to each other, creating a

paired sampling design (Table 2.2; Figure 2.2). Natural sites were situated away from the forest edge to account for disturbance impacts on soils and vegetation.

**Table 2.2: Stockpile and natural reference sites for the CNRL Wolf Lake study area. All stockpiles were labelled using the operator’s identification number (ie. PS/PE/WL-X). Mature sites were labelled with an NS- amended to the stockpile identifier.**

Stockpile	Sampling Area	Site Type	Age (years)	Location (latitude, longitude)
PS-43	CNRL Wolf Lake	Stockpiled	2	54.775, -110.638
PS-25B	CNRL Wolf Lake	Stockpiled	6	54.768, -110.514
PE-78B	CNRL Wolf Lake	Stockpiled	11	54.783, -110.314
WL-P	CNRL Wolf Lake	Stockpiled	28	54.694, -110.721
NS-PS-43	CNRL Wolf Lake	Natural	Mature	54.775, -110.643
NS-PS-25B	CNRL Wolf Lake	Natural	Mature	54.767, -110.515
NS-PE-78B	CNRL Wolf Lake	Natural	Mature	54.781, -110.313
NS-WL-P	CNRL Wolf Lake	Natural	Mature	54.694, -110.720



**Figure 2.2: Stockpile and natural site locations at CNRL Wolf Lake.**

### 2.3.2. Field Methods

Three soil pits were dug on each stockpile and at each natural site, spaced approximately 20 m apart, to better capture the variability of soils on selected sites. Soil pits were dug by hand using

a shovel and pickaxe to a depth of 90 cm and a tape measure was placed along the sampling face of the soil pit to establish sampling depths at 0 to 10 cm, 20 to 30 cm, and 80 to 90 cm. These sampling depths were chosen to reflect potential inclusions and exclusions during soil salvage. The 0 to 10 cm depth would typically be the target of salvage, with the 20 to 30 cm depth possibly being taken as well. The deepest sampling depth, at 80 to 90 cm, would remain behind. These different sampling depths would have different soil properties largely relating to *in situ* pedogenic processes, differences that could impact stockpile soil suitability during reclamation.

Samples were taken from the sampling face, at each depth, in two forms. First, stainless steel soil cores, measuring 8 cm in diameter and 5 cm in height, were pounded into the sampling face using a block and hammer until the top of the core was flush with the pit surface. A soil knife was then used to carefully remove the core. Bulk soil samples were then collected from a cleaned sampling face, were removed using a trowel, and placed into large, labelled plastic bags. Refrigerators set at 5°C were used to store the samples at field sites and while in storage in Edmonton. All soil samples were kept in coolers during transport to the laboratory.

### **2.3.3. Laboratory Methods**

#### **2.3.3.1. Site Descriptor Variables**

Bulk soil samples were used for determining gravimetric water content, pH, electrical conductivity (EC), and soil texture (section 2.3.3.2.) (Table 2.3). Field moist soils were passed through a 2 mm sieve to remove organic debris and coarse fragments. Samples were then placed in a drying oven set at 60°C for two days or until a constant weight was obtained. Soils that were excessively clayey and challenging to sieve at field water contents were oven-dried as above, broken with a hammer, and then passed through the 2 mm sieve. Gravimetric water contents

were determined by estimating the weight of water lost during the drying process and dividing by the dry weight of the soil (Topp et al., 2008). An Accumet XL200 meter was used to determine pH and EC, using deionized water and a soil-to-water ratio of 1:2 for mineral soils and 1:4 for soils with higher organic content (Kalra and Maynard, 1991).

**Table 2.3: Mean values and standard errors for gravimetric water content (g/g), pH, and electrical conductivity (EC) ( $\mu\text{S}/\text{cm}$ ), as well as textural class, for each natural and stockpiled depth.**

	Water Content (g/g)	pH	EC ( $\mu\text{S}/\text{cm}$ )	Textural Class
<b>Natural</b>				
0-10 cm	$0.18 \pm 0.02$	$5.71 \pm 0.22$	$85.4 \pm 11.7$	Loam
20-30 cm	$0.16 \pm 0.01$	$6.03 \pm 0.26$	$102.8 \pm 12.6$	Clay Loam
80-90 cm	$0.17 \pm 0.02$	$6.82 \pm 0.23$	$270.3 \pm 106.5$	Clay Loam
<b>Stockpiled</b>				
0-10 cm	$0.42 \pm 0.09$	$6.45 \pm 0.21$	$215.2 \pm 22.3$	Loam
20-30 cm	$0.48 \pm 0.11$	$6.41 \pm 0.24$	$228.9 \pm 34.1$	Loam
80-90 cm	$0.52 \pm 0.15$	$6.57 \pm 0.21$	$256.8 \pm 36.2$	Loam

### 2.3.3.2. Soil Texture

Soil texture was measured on sieved, oven-dried bulk soils (section 2.3.3.1) utilizing the hydrometer method outlined in Kaddah (1974). Following dispersion of 50 g soil samples with a sodium hexametaphosphate solution in a rotary mixing, the slurry was suspended in a 1-litre sedimentation cylinder. Hydrometer readings of the suspended mixture were taken at 0.5, 1, 2, 3, 4, 5, 10, 30, 60, and 120 minutes, as well as the following day, after suspension.

### 2.3.3.3. Bulk Density

Bulk density was determined using the soil core samples used for measurement of water retention curves (section 2.3.3.5). Following equilibration at -100 cm of water, the cores were

broken apart in smaller homogenized subsamples. A portion of this was placed in an oven at 105°C for two days, or until a constant weight was obtained. This water content was used to determine a dry weight for the whole core, which was then divided by core volume.

#### **2.3.3.4. Total Soil Carbon**

Total soil carbon was measured in sieved, oven-dried bulk soils (section 2.3.3.1). The dried soil was ground in a Retsch MM200 ball grinder at a frequency of 30 Hz for one minute and placed into labelled vials. These ground samples were sent to the Natural Resources Analytical Lab (NRAL) at the University of Alberta, and were analysed using a ThermoFinnigan Delta V Advantage Continuous Flow Isotope Ratio Mass Spectrometer.

#### **2.3.3.5. Water Retention**

Water retention curves were measured by equilibrating soil samples at known potentials using a tension table and pressure plate extractor as described in Reynolds and Topp (2007). External surfaces of the soil cores were first trimmed to remove any excess soil. In order to ensure hydraulic contact between the soil in the core and the tension table and pressure plates, saturated soil paste, made from the Ah horizon from Ellersie Research Center in Edmonton, was applied to the base of the soil core and covered with nylon mesh. Then, additional paste was applied to the bottom outer surface of the nylon mesh. The cores were set on a tension table at tensions of 0, -10, and -40 cm of water and allowed one week to equilibrate. The cores were then removed from the tension table, the paste on the outside of the nylon sheets was removed, and the cores were weighed. A new layer of paste was applied before the cores were placed back on the tension table. Water contents were estimated for the paste applied directly to the soil cores and to the nylon sheets according to the drying procedure outlined in section 2.3.3.3. For the tension

table and pressure plates, it was assumed that the equilibrium water content of the paste between the soil in the core and the inside of the nylon mesh was equal to the equilibrium water content of the paste on the outside of the nylon mesh.

Following equilibration at -40 cm of water, intact cores were moved to a 0.5 bar, high flow ceramic pressure plates (Soil Moisture Equipment Corp., Santa Barbara, CA) and placed in a pressure plate extractor set to a pressure of -100 cm of water and allowed to equilibrate for one week. The cores were weighed after one week, the soil was removed from the core, homogenized, and then subsampled to determine both bulk density (section 2.3.3.3) and to fill small rings measuring 5 cm in diameter by 1 cm in height. These rings were moved to ceramic plate extractors set at -500, -1000, -5000, and -15000 cm of water for one week.

Differences in weight from the start and end of the one-week equilibration were used to determine a weight of water, which, along with bulk density was used to estimate volumetric water content for each pressure level. The values were imported into PTC Mathcad version 15 to generate data points for a SWRC, with a double exponential model being used to generate the curve itself (Equation 2.1; Table 2.4) The Levenburg-Marquardt algorithm was used to improve the fit of the curve and identify parameters in the model that best fit the imported measurements.

$$\theta_v(h) = C + A_1 e^{(-h/h_1)} + A_2 e^{(-h/h_2)} \quad [2.1]$$

**Table 2.4: Description of the double exponential model parameters used to generate the majority of the water retention curves (Dexter et al., 2008; Ding et al., 2016).**

Parameter	Description	Parameter	Description
$\theta_v$	Soil volumetric water content ( $\text{cm}^3/\text{cm}^3$ )	$A_2$	Matrix porosity
C	Residual porosity	$h_1$	Suction to empty structural pores (hPa)
$A_1$	Structural porosity	$h_2$	Suction to empty matrix pores (hPa)



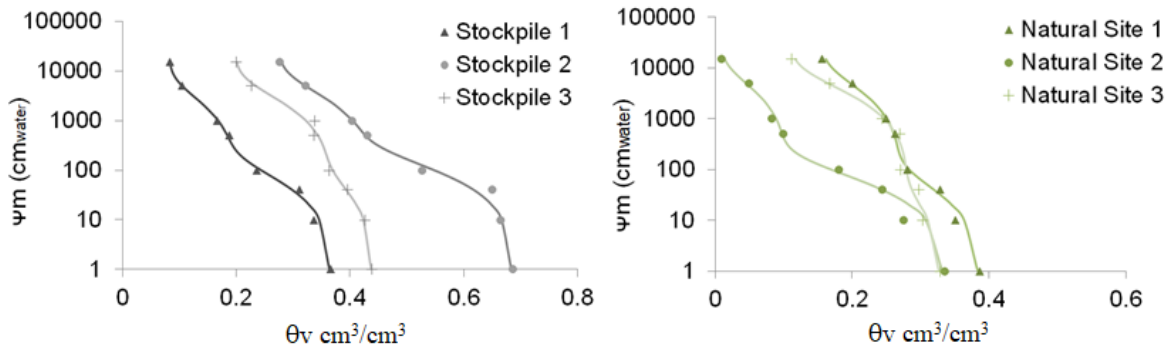
The double exponential model, however, was not appropriate for a few samples due to the unimodal nature of the SWRC. As such, the van Genuchten model was used for these samples instead (Equation 2.2; Table 2.5).

$$\theta_v(h) = \theta_r + \frac{\theta_s - \theta_r}{[1 + |\alpha h|^n]^m} \quad [2.2]$$

**Table 2.5: Description of the van Genuchten model parameters used to generate water retention curves for a select batch of soils sampled (van Genuchten, 1980).**

Parameter	Description	Parameter	Description
$\theta_v$	Soil volumetric water content ( $\text{cm}^3/\text{cm}^3$ )	H	Soil water potential (cm)
$\theta_r$	Soil residual water content ( $\text{cm}^3/\text{cm}^3$ )	A	Scale parameter inversely proportional to the mean pore diameter
$\theta_s$	Soil saturated water content ( $\text{cm}^3/\text{cm}^3$ )	n/m	Shape parameters of soil water characteristic

Examples of SWRCs generated by the above models can be seen in Figure 2.3.



**Figure 2.3: Soil water retention curves for three stockpiles and three natural sites.**

A number of parameters, including FC, AC, PAWC, macroporosity, S,  $A_a$ , and  $WR_a$  were calculated from these SWRCs. These parameters are described in greater detail in Table 2.6, and, where possible, shown on a hypothetical SWRC in Figure 2.4.

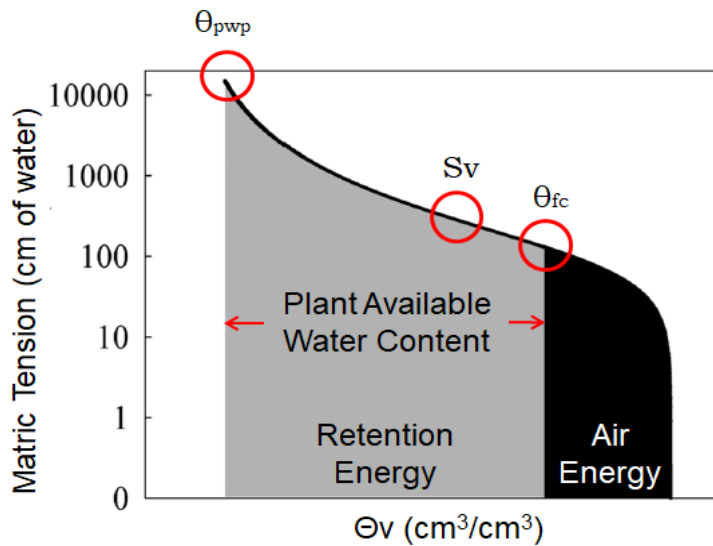
**Table 2.6: Description of parameters calculated from the soil water retention curves.**

<b>Parameter</b>	<b>Description of Parameter</b>
Field Capacity (FC) ( $\text{cm}^3 \text{ cm}^{-3}$ )	Volumetric water content of gravity drained soil (Reynolds et al., 2009).
Air Capacity (AC) ( $\text{cm}^3 \text{ cm}^{-3}$ )	The volume of soil pores filled with air; estimated by finding the difference between saturated water content and soil field capacity (gravity drained) (White, 2006; Reynolds et al., 2009).
Plant Available Water Content (PAWC) ( $\text{cm}^3 \text{ cm}^{-3}$ )	Represents the ability of the soil to store and provide water for plant roots; estimated by finding the difference in volumetric water content between field capacity and permanent wilting point (White, 2006; Reynolds et al., 2009).
Macroporosity ( $\text{cm}^3 \text{ cm}^{-3}$ )	Indicates the ability of a soil to quickly drain excess water; estimated by finding the difference in saturated volumetric water content and the “saturated” volumetric water content of the soil matrix exclusive of macropores (Reynolds et al., 2008; Reynolds et al., 2009).
S ( $\text{cm}^3 \text{ cm}^{-3} \text{ cm}^{-1}$ )	The slope of a soil water retention curve at the inflection point (Dexter, 2004; Reynolds et al., 2009). The inflection point is equal to the matric potential at which the second derivative is at a local minimum value.
Air Energy ( $A_a$ ) ( $\text{cm} \text{ cm}^3 \text{ cm}^{-3}$ )	The absolute integral energy associated with the gravitational potential causing water to drain from saturation to field capacity. Air enters the soil as water is lost during drainage (Armino and Wendroth, 2016).
Retention Energy ( $WR_a$ ) ( $\text{cm} \text{ cm}^3 \text{ cm}^{-3}$ )	The absolute integral energy that has to be applied by the soil to hold pore water from field capacity to the permanent wilting point (Armino and Wendroth, 2016).

It is important to note that Dexter (2004) utilizes S values, estimated using gravimetric water content as a function of matric potential (units of  $\text{g g}^{-1} \text{ cm}^{-1}$ ) whereas S values, estimated using volumetric water content as a function of matric potential (units of  $\text{cm}^3 \text{ cm}^{-3} \text{ cm}^{-1}$ ), were determined for this study. However, as outlined in both Reynolds et al. (2009) and Armino and Wendroth (2016), these two values can be related to each other by the bulk density of each sample. Pore volume distributions, intricately linked to volumetric S values, have been identified as being a stronger measure of SPQ than gravimetric S, and so the volumetric S values remained

the focus of this study and, where relevant, will be distinguished using physical units (Reynolds et al., 2009; Armindo and Wendroth, 2016).

It should be noted that the model chosen to represent the SWRC will influence the value of the S index. The most common model used to calculate the S index is the van Genuchten (1980) model (i.e., VG model; Eq. [2.2]) and the S index thresholds categorizing SPQ as good, poor or moderate have been developed using values of the S index calculated from SWRCs represented by the VG model. The main difference between the VG model and double exponential model, again, is that the former assumes a unimodal soil pore-size distribution and the latter assumes a bimodal pore-size distribution (Dexter and Richard, 2009). Therefore, while the majority of the S index values for the soils in this work will not be directly comparable to those presented in other works, the trends will be similar.



**Figure 2.4: Hypothetical soil water retention curve showing the relationship between field capacity ( $\theta_{fc}$ ) ( $\text{cm}^3 \text{cm}^{-3}$ ), permanent wilting point ( $\theta_{pwp}$ ) ( $\text{cm}^3 \text{cm}^{-3}$ ), plant available water content ( $\text{cm}^3 \text{cm}^{-3}$ ), S ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ), retention energy ( $\text{cm cm}^3 \text{cm}^{-3}$ ) (gray shaded area) and air energy ( $\text{cm cm}^3 \text{cm}^{-3}$ ) (black shaded area) parameters discussed in Table 2.6 (modified from Armindo and Wendroth, 2016).**

### **2.3.4. Statistical Analyses**

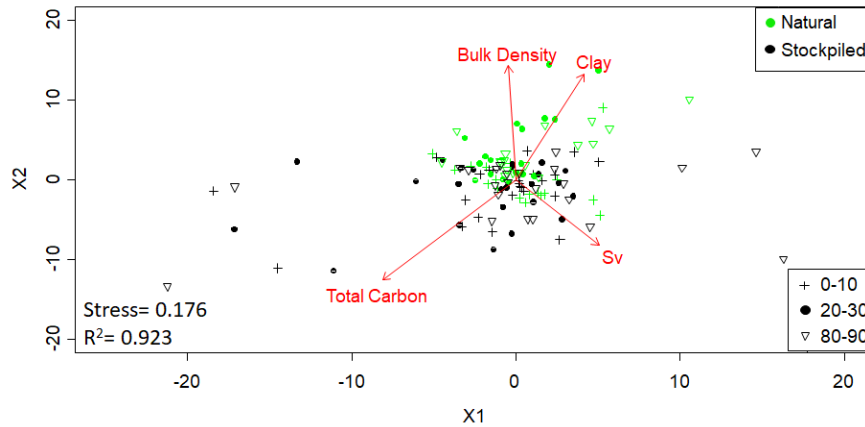
All statistical analyses were carried out in R Studio. Non-metric multidimensional scaling (NMDS) ordinations, using the distance function from the *ecodist* package, were first used to visualize differences in response variables by disturbance type (natural or stockpiled) and depth category (0 to 10 cm, 20 to 30 cm, or 80 to 90 cm). Significant differences between groups were assessed using a perMANOVA with pairwise comparisons, generated with the *adonis* and *pairwise.perm.manova* functions from the *vegan* and *RVAideMemoire* packages respectively.

NMDS is a multivariate technique; therefore, it is not possible to differentiate significant differences at a single variable level. As such, linear mixed models were applied to better identify any significant differences between natural and stockpiled soils and what the magnitude of that difference was. This required the use of the *lme* and *emmeans* functions from the *nlme* and *emmeans* packages. Linear mixed models were chosen for this task due to the blocking effects that were present between the two sampling locations, Horizon and Wolf Lake, as well as within stockpiles and individual soil pits. These blocking effects would lead to a covariance of samples at each level of the study's nested hierarchy, rendering a normal linear model ineffective.

## **2.4. RESULTS**

### **2.4.1. Site-Depth Comparisons**

Clay contents and bulk densities of the sampled soils were higher in natural soils, whereas total soil carbon and the S index were higher in stockpiled soils (Figure 2.5). For the four ordinated parameters, natural and stockpiled soils differed only at the 20 to 30 cm depth ( $p=0.015$ ) and the 80 to 90 cm depths ( $p=0.020$ ) (Table 2.7).



**Figure 2.5: NMDS ordination for soil clay content (%), bulk density ( $\text{g cm}^{-3}$ ), total soil carbon (%), and the S index ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ) by site type and sampling depth.**

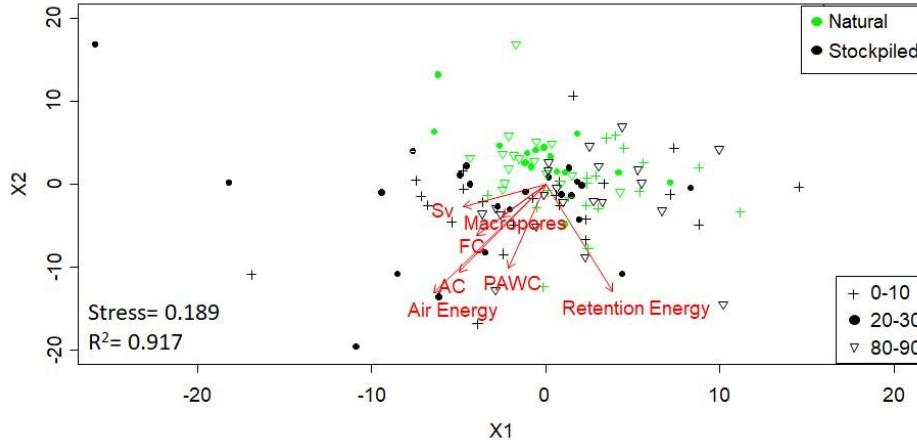
Within stockpiled sites, no significant differences between sampling depths were observed for clay content, bulk density or soil carbon ( $p=1.000$ ), but within natural sites these properties were significantly different in the 0 to 10 cm depth from the 20 to 30 cm depth ( $p=0.027$ ) and the 80 to 90 cm depth ( $p=0.015$ ) (Table 2.7).

**Table 2.7: perMANOVA pairwise comparisons (p-values) for soil clay content (%), bulk density ( $\text{g cm}^{-3}$ ), total soil carbon (%), and the S index ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ) parameters ordinated in Figure 2.5. Significant differences were declared for  $p<0.05$  (bolded in table).**

Comparisons					
	<b>0-10 Natural</b>	<b>0-10 Stockpiled</b>	<b>20-30 Natural</b>	<b>20-30 Stockpiled</b>	<b>80-90 Natural</b>
<b>0-10 Stockpiled</b>	0.540	-	-	-	-
<b>20-30 Natural</b>	<b>0.027</b>	<b>0.015</b>	-	-	-
<b>20-30 Stockpiled</b>	0.357	1.000	<b>0.015</b>	-	-
<b>80-90 Natural</b>	<b>0.015</b>	<b>0.015</b>	1.000	<b>0.015</b>	-
<b>80-90 Stockpiled</b>	0.815	1.000	0.192	1.000	<b>0.020</b>

All capacity-based indicators (FC, PAWC, AC, and macroporosity) were higher in stockpiles, as were air and water retention energies, which corresponded with a higher S index (Figure 2.6).

For the seven ordinated parameters, natural and stockpiled sites were observed to differ only between the 20 to 30 cm ( $p=0.015$ ) and 80 to 90 cm depths ( $p=0.033$ ) (Table 2.8).



**Figure 2.6: NMDS ordination for field capacity (FC) ( $\text{cm}^3 \text{cm}^{-3}$ ), plant available water content (PAWC) ( $\text{cm}^3 \text{cm}^{-3}$ ), air capacity (AC) ( $\text{cm}^3 \text{cm}^{-3}$ ), macropores ( $\text{cm}^3 \text{cm}^{-3}$ ), air energy ( $\text{cm cm}^3 \text{cm}^{-3}$ ), retention energy ( $\text{cm cm}^3 \text{cm}^{-3}$ ), and the S index ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ) by site type and sampling depth.**

**Table 2.8: perMANOVA pairwise comparisons (p-values) field capacity (FC) ( $\text{cm}^3 \text{cm}^{-3}$ ), plant available water content (PAWC) ( $\text{cm}^3 \text{cm}^{-3}$ ), air capacity (AC) ( $\text{cm}^3 \text{cm}^{-3}$ ), macropores ( $\text{cm}^3 \text{cm}^{-3}$ ), air energy ( $\text{cm cm}^3 \text{cm}^{-3}$ ), retention energy ( $\text{cm cm}^3 \text{cm}^{-3}$ ), and the S index ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ) parameters ordinated in Figure 2.6. Significant differences were declared when  $p < 0.05$  (bolded in table).**

Comparisons					
	0-10 Natural	0-10 Stockpiled	20-30 Natural	20-30 Stockpiled	80-90 Natural
<b>0-10 Stockpiled</b>	0.516	-	-	-	-
<b>20-30 Natural</b>	<b>0.033</b>	<b>0.015</b>	-	-	-
<b>20-30 Stockpiled</b>	0.315	1.000	<b>0.015</b>	-	-
<b>80-90 Natural</b>	<b>0.033</b>	<b>0.015</b>	1.000	<b>0.015</b>	-
<b>80-90 Stockpiled</b>	0.810	1.000	0.128	1.000	<b>0.033</b>

Within stockpiled sites, no significant differences were observed when any of the sampling depths were compared ( $p=1.000$ ), while within natural sites, significant differences were only observed in the 0 to 10 cm depth when compared to either the 20 to 30 cm depth ( $p=0.033$ ) or the 80 to 90 cm depth ( $p=0.033$ ) (Table 2.8).

#### 2.4.2. Soil Texture

Clay contents were higher in natural sites (overall mean = 31.7%) than in stockpiles (overall mean = 19.8%) across all sampled depths; however, this difference was only significant for the 20 to 30 cm ( $p=0.0002$ ) and the 80 to 90 cm ( $p=0.0001$ ) depths (Table 2.9).

**Table 2.9: Estimated marginal means and standard errors for soil clay content (%), bulk density ( $\text{g cm}^{-3}$ ), total soil carbon (%), and the S index ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ). For each property, uppercase letters denote significant differences between natural and stockpiled soils at a particular depth. Lower case letters represent significant differences within natural or stockpiled site types between difference depths. Significant differences were declared when  $p<0.05$ .**

	Clay Content (%)	Bulk Density ( $\text{g cm}^{-3}$ )	Total Soil Carbon (%)	S ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ )
<b>Depth=0-10 cm</b>				
Natural	20.7 ± 9.6 Aa	1.14 ± 0.10 Aa	0.96 ± 2.09 Aa	0.022 ± 0.006 Aa
Stockpiled	19.4 ± 9.5 Aa	0.96 ± 0.09 Aa	1.49 ± 1.78 Ba	0.034 ± 0.005 Aa
<b>Depth=20-30 cm</b>				
Natural	35.4 ± 9.6 Ab	1.43 ± 0.10 Ab	0.84 ± 2.06 Aa	0.020 ± 0.006 Aa
Stockpiled	18.4 ± 9.5 Ba	0.96 ± 0.09 Ba	1.50 ± 1.78 Ba	0.035 ± 0.005 Ba
<b>Depth=80-90 cm</b>				
Natural	39.1 ± 9.6 Ab	1.33 ± 0.10 Ab	0.85 ± 2.06 Aa	0.016 ± 0.006 Aa
Stockpiled	21.5 ± 9.6 Ba	0.91 ± 0.09 Ba	1.30 ± 1.83 Bb	0.037 ± 0.006 Ba

No significant differences were observed within stockpiled soils for clay content between any sampling depth ( $p>0.200$ ), whereas lower clay content was observed within natural sites at the 0 to 10 cm depth when compared to the 20 to 30 cm ( $p<0.0001$ ) and 80 to 90 cm depth ( $p<0.0001$ ) (Table 2.9).

### **2.4.3. Bulk Density**

The same patterns as noted for soil texture also existed for bulk density. Natural sites had higher bulk density across all sampling depths (overall mean =  $1.30 \text{ g/cm}^3$ ) than stockpiled soils (overall mean =  $0.94 \text{ g/cm}^3$ ), although this difference was only significant at the 20 to 30 cm ( $p=0.0044$ ) and 80 to 90 cm ( $p=0.0106$ ) depths (Table 2.9). Within stockpiled soils, no significant differences in bulk density were observed between depths; however, within natural sites, a lower bulk density was observed at the 0 to 10 cm depth when compared to either the 20 to 30 cm ( $p=0.0006$ ) or 80 to 90 cm depth ( $p=0.0421$ ) (Table 2.9).

### **2.4.4. Total Soil Carbon**

Stockpiles had higher amounts of soil carbon (overall mean = 1.43%) than natural soils (overall mean = 0.88%) across the 0 to 10 cm, 20 to 30 cm, and 80 to 90 cm depths ( $p=0.0061$ ,  $p=0.0014$ ,  $p=0.0147$ , respectively) (Table 2.9). Within natural sites, no significant differences in total carbon were observed between depths ( $p>0.250$ ), whereas within stockpiles, significantly lower total carbon quantities were observed in the 80 to 90 cm depth than in the 0 to 10 cm ( $p=0.0238$ ) or the 20 to 30 cm depths ( $p=0.0175$ ) (Table 2.9).



## 2.4.5. Soil Water Retention Curves

### 2.4.5.1. Capacity-Based Measurements

FC, PAWC, AC, and macroporosity were all higher in stockpiles (overall mean =  $0.35 \text{ cm}^3/\text{cm}^3$ ,  $0.18 \text{ cm}^3/\text{cm}^3$ ,  $0.16 \text{ cm}^3/\text{cm}^3$ ,  $0.07 \text{ cm}^3/\text{cm}^3$ , respectively) than in natural sites (overall mean =  $0.26 \text{ cm}^3/\text{cm}^3$ ,  $0.10 \text{ cm}^3/\text{cm}^3$ ,  $0.08 \text{ cm}^3/\text{cm}^3$ ;  $0.05 \text{ cm}^3/\text{cm}^3$ , respectively); however, FC values were only significantly higher in stockpiled soils at the 0 to 10 cm depth ( $p=0.0478$ ) (Table 2.10). PAWC and AC were significantly higher in stockpiles at the 20 to 30 cm ( $p=0.0010$ ,  $p=0.0002$  respectively) and 80-90 cm depths ( $p=0.0004$ ,  $p=0.0149$  respectively), while macropores were significantly higher only at the 20 to 30 cm depth ( $p=0.0021$ ) (Table 2.10).

**Table 2.10: Estimated marginal means and standard errors for field capacity (FC) ( $\text{cm}^3 \text{ cm}^{-3}$ ), plant available water content (PAWC) ( $\text{cm}^3 \text{ cm}^{-3}$ ), air capacity (AC) ( $\text{cm}^3 \text{ cm}^{-3}$ ), and macropores ( $\text{cm}^3 \text{ cm}^{-3}$ ). For each property, uppercase letters denote significant differences between natural and stockpiled soils at a particular depth. Lower case letters represent significant differences within natural or stockpiled site types between difference depths. Significant differences were declared when  $p<0.05$ .**

	FC ( $\text{cm}^3 \text{ cm}^{-3}$ )	PAWC ( $\text{cm}^3 \text{ cm}^{-3}$ )	AC ( $\text{cm}^3 \text{ cm}^{-3}$ )	Macropores ( $\text{cm}^3 \text{ cm}^{-3}$ )
<b>Depth= 0-10 cm</b>				
Natural	$0.24 \pm 0.06$ Aa	$0.14 \pm 0.02$ Aa	$0.11 \pm 0.02$ Aa	$0.06 \pm 0.01$ Aa
Stockpiled	$0.35 \pm 0.05$ Ba	$0.18 \pm 0.01$ Aa	$0.16 \pm 0.02$ Aa	$0.08 \pm 0.01$ Aa
<b>Depth= 20-30 cm</b>				
Natural	$0.27 \pm 0.06$ Aa	$0.08 \pm 0.02$ Ab	$0.05 \pm 0.02$ Ab	$0.03 \pm 0.01$ Ab
Stockpiled	$0.35 \pm 0.05$ Aa	$0.17 \pm 0.01$ Ba	$0.16 \pm 0.02$ Ba	$0.07 \pm 0.01$ Ba
<b>Depth= 80-90 cm</b>				
Natural	$0.27 \pm 0.06$ Aa	$0.09 \pm 0.02$ Ab	$0.09 \pm 0.02$ Aab	$0.05 \pm 0.01$ Aa
Stockpiled	$0.34 \pm 0.05$ Aa	$0.18 \pm 0.02$ Ba	$0.15 \pm 0.02$ Ba	$0.06 \pm 0.01$ Aa

No significant difference in FC, PAWC, AC, or macroporosity values were observed within stockpiles soils between sampling depths ( $p>0.550$ ,  $p>0.500$ ,  $p>0.450$ ,  $p>0.150$ , respectively). Within natural soils, no significant difference was observed for FC between depths ( $p>0.450$ ), though both the 0 to 10 cm and 80 to 90 cm depths had significantly higher air capacities ( $p<0.0001$ ,  $p=0.0197$ , respectively) and volumes of macropores ( $p=0.0020$ ,  $p=0.0261$  respectively) when compared to the 20 to 30 cm depth. PAWC was significantly higher in the 0 to 10 cm depth when compared to the 20 to 30 cm ( $p<0.0001$ ) and the 80 to 90 cm depth ( $p<0.0001$ ) (Table 2.10).

Higher capacity-based measurements across all depths in stockpiled soils, when compared to natural sites, corresponded with similarly higher S values for stockpiled sites (overall mean =  $0.035 \text{ cm}^3 \text{ cm}^{-3} \text{ cm}^{-1}$ ) than natural soils (overall mean =  $0.019 \text{ cm}^3 \text{ cm}^{-3} \text{ cm}^{-1}$ ) across all depths (Table 2.9). However, these differences were only significant when stockpiled and natural soils were compared at the 20 to 30 cm ( $p=0.0076$ ) and the 80 to 90 cm depth ( $p=0.0044$ ). No significant differences were observed between sampling depths within natural ( $p>0.150$ ) and stockpiled sites ( $p>0.950$ ) (Table 2.9).

#### **2.4.5.2. Energy Parameters**

Air and water retention energies were higher in stockpiled soils (overall mean =  $8.52 \text{ cm}^3/\text{cm}^3$ ;  $435 \text{ cm}^3/\text{cm}^3$ , respectively) than in natural sites (overall mean =  $3.60 \text{ cm}^3/\text{cm}^3$ ;  $318 \text{ cm}^3/\text{cm}^3$ , respectively). Air energies were found to be significantly higher in stockpiled soils at all depths ( $p=0.0402$ ;  $p=0.0002$ ;  $p=0.0038$ ), whereas retention energies were only higher in stockpiles at the 80 to 90 cm depth ( $p=0.0066$ ) (Table 2.11).

Within stockpiled soils, no significant differences in air energies ( $p > 0.800$ ) or water retention energies ( $p > 0.150$ ) were observed between depths (Table 2.11). Within natural sites, higher air energies were observed in the 0 to 10 cm depth than the 20 to 30 cm ( $p < 0.0001$ ) and 80 to 90 cm ( $p = 0.0367$ ) depths. Air energies were also higher in the 80 to 90 cm depth than in the 20 to 30 cm depth ( $p = 0.0497$ ). Water retention energies within natural sites were only found to be higher in the 0 to 10 cm depth than in the 80 to 90 cm depth ( $p = 0.0072$ ) (Table 2.11).

**Table 2.11: Estimated marginal means and standard errors for air energies ( $\text{cm cm}^3 \text{cm}^{-3}$ ) and water retention energies ( $\text{cm cm}^3 \text{cm}^{-3}$ ). For each property, uppercase letters denote significant differences between natural and stockpiled soils at a particular depth. Lower case letters represent significant differences within natural or stockpiled site types between difference depths. Significant differences were declared when  $p < 0.05$ .**

	Air Energy ( $\text{cm cm}^3 \text{cm}^{-3}$ )	Retention Energy ( $\text{cm cm}^3 \text{cm}^{-3}$ )
<b>Depth=0-10 cm</b>		
Natural	5.40 ± 1.17 Aa	424 ± 67 Aa
Stockpiled	8.55 ± 1.00 Ba	445 ± 58 Aa
<b>Depth=20-30 cm</b>		
Natural	2.04 ± 1.14 Ab	300 ± 64 Aa
Stockpiled	8.70 ± 1.00 Ba	363 ± 58 Aa
<b>Depth=80-90 cm</b>		
Natural	3.37 ± 1.14 Ac	229 ± 64 Aba
Stockpiled	8.32 ± 1.04 Ba	496 ± 61 Ba

## 2.5. DISCUSSION

### 2.5.1. Utilizing Basic Soil Properties to Apply the S Theory

Stockpiled soils had lower clay contents and bulk densities, and higher total carbon, than natural soils in the two subsurface depths, results that are largely contrary to the original hypotheses of this study and those of many other stockpiled studies (Gee and Bauer, 1976; Abdul-Kareem and

McRae, 1984; Schwenke et al., 2000a; b; Ghose, 2004; Wick et al., 2009). Within stockpiles, no differences in clay content or bulk density were observed between depths, whereas within natural sites, increases in clay content and bulk density were observed between surface and subsurface depths. These results supported the original hypotheses of this study. In contrast, no changes in total carbon between depths within natural sites, and declines in total carbon between subsurface and surface depths in stockpiles, refutes the original hypotheses of this study.

Natural soils of the sampling area were predominately Gray Luvisols (Downing and Pettapiece, 2006). As such, lower clay contents throughout the stockpile profile are likely due to the inherent characteristics of the LFH and A horizons targeted for soil salvage. The LFH layer lacks clay almost entirely as it is an organic layer that remains on the soil surface, not mixing with underlying mineral horizons as seen in other soil orders (Lavkulich and Arocena, 2011; Pennock et al., 2011; Smith et al., 2011). Luvisolic A horizons are eluviated, with the majority of the clay originally present being translocated downwards with percolating water (Lavkulich and Arocena, 2011). Therefore, due to the homogenization effects of soil salvage and storage, clay contents throughout the entire stockpile profile would be uniform and similar to those observed in surface depths of natural soils, observations that were made in this study. The relatively young age of many stockpiles likely means that stratification associated with clay eluviation is also not yet observable, preventing increases in clay contents with depth as seen in natural Luvisolic soils.

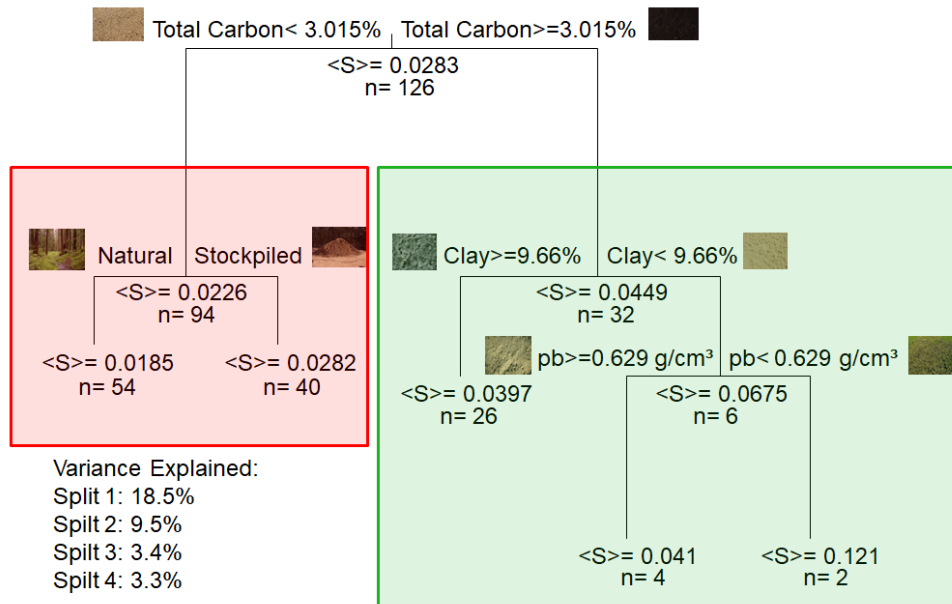
Lower levels of clay in the targeted salvage material would likely have implications on bulk density as well, largely by minimizing soil plasticity and alterations to soil porosity following disturbance (Chaudhari et al., 2013). This would provide some explanation for the consistently lower bulk densities observed in stockpiles. Distribution of clays also would explain the trends observed within the stockpile and natural soil profiles; stockpiles, with clay contents

homogenized during placement, would experience less compaction issues between all sampled depths. Natural sites, which have undergone eluviation, would have greater clay contents in subsoil layers, layers comprised of till parent material that already have elevated bulk densities.

Soil organic matter, in turn, also likely influenced bulk density due to its role in the development of soil structure. Unlike the natural soils of Alberta's boreal forest, where the organic LFH layer remains on the soil surface, salvaging and stockpiling would see the integration of organic and mineral materials (Lavkulich and Arocena, 2011; Smith et al., 2011). In this scenario, the LFH may have acted similar to an organic amendment, facilitating aggregation, improving soil porosity, reducing bulk density, and increasing soil carbon throughout the entire stockpile profile (Boyle et al., 1989; Larney and Angers, 2012). Homogenization of total soil carbon quantities within stockpiles, as discussed above, likely further explains a lack of bulk density changes between stockpile depths. Natural soils, which exhibited no change in total soil carbon quantities with increasing depth, would have potentially lacked increases in soil porosity as discussed above, contributing to increased bulk densities.

Differences in clay contents, bulk densities, and total soil carbon between natural and stockpiled sites should be reflected by the S index. The S index was developed originally for agricultural zones; however, it integrates a number of soil parameters and has been shown to represent SPQ in soils from multiple different geographic zones, soil types, and climates (Dexter, 2004). As such, it has a wide applicability for measuring SPQ, and so has been applied to natural and stockpiled soils in this study. The S index, in theory, should increase with increasing total soil carbon, and decline with increasing clay content and bulk density (Dexter, 2004).

As can be seen in Figure 2.7, higher levels of soil carbon, lower clay content, and lower bulk density results in the highest S values, all of which correspond with stockpiled soils (Table 2.9). Natural sites, which exhibited lower soil carbon, higher clay contents, and higher bulk densities, consequently had the lowest S values (Figure 2.5; Table 2.9; Figure 2.7).



**Figure 2.7: Regression tree showing the relationship between S ( $\text{cm}^3 \text{ cm}^{-3} \text{ cm}^{-1}$ ), the dependent variable, and the independent variables clay content (%), bulk density ( $\text{g cm}^{-3}$ ), total soil carbon (%), and site type. <S> represent the mean S values for each split, and n is the number of samples that fall into each split category. Variance explained by each split is also shown.**

Dexter (2004) suggested a S threshold of  $S > 0.035 \text{ hPa/g/g}$  as representing good SPQ. Using this threshold as a rough guideline for this study's similar (although not directly comparable) S values, not only do natural soils fall below this threshold, they also fall below Dexter's (2004) poor soil physical threshold of  $S < 0.020 \text{ hPa/g/g}$  (Dexter, 2004). Figure 2.7 also shows that, while clay contents and bulk densities are important in influencing S, it is soil carbon that determines whether a soil will exceed or fall below the poor SPQ S threshold. Having sufficient

levels of soil carbon, to promote the formation of more stable soil aggregates or, at least, to improve the porosity of the soil, creates better SPQ.

While the experimental data are consistent with the S theory of Dexter (2004), higher S values noted in stockpiles contradict the original hypothesis of lower SPQ in stockpiled soils and seems counterintuitive. Natural sites support mature, functioning forests. Soil stockpiles, at the time of sampling, were either bare soil, colonized mainly by weedy species, or were just becoming colonized by native forbs and shrubs. As such, the S index can be compared to capacity-based measures also derived from SWRCs to see if alternative implications can be found.

### **2.5.2. Relating Capacity Based Indicators to S and Overall Soil Physical Quality**

Stockpiled soils had higher FC, PAWC, AC, and macroporosity than natural sites, primarily in the two subsurface horizons, which again is contrary to the original hypotheses of this project and the findings of similar studies on disturbed soils (Schwenke et al., 2000b; Soon et al., 2000; Ghose, 2004; Banning et al., 2008). Within stockpiles, no differences were observed for these variables between sampling depths. Within natural sites, declines in PAWC, AC, and macroporosities were also observed, with no differences observed for FC. Both of these observations largely support the original hypotheses of this study.

Stockpiled soils in this study contained high overall total carbon, sourced from a combination of integrated LFH and inherent organic material in the initially salvaged soil. These higher levels of soil carbon would improve the macropore structure of the stockpiled soils, creating a larger amount of structural pores. These structural pores influence the SWRC and a number of capacity-based measurements derived from it, including FC, AC, and PAWC, with further implications on S and SPQ (Dexter, 2004).

Work has been done on relating these capacity-based measures to SPQ, primarily by applying thresholds to classify a soil as having either good or poor SPQ (Table 2.12). These thresholds, like the S index, were originally created for agricultural zones. Also like the S index, they have been shown to remain consistent across a number of different land management systems, soil types, and climate regimes, and so have been applied to natural and stockpiled soils in this study (Cockroft and Olsson, 1997; Drewry et al., 2008).

**Table 2.12: Summary of capacity based threshold values to assess soil physical quality (Hall et al., 1977; Carter, 1988; Doran et al., 1990; Cockroft and Olsson, 1997; White, 2006; Reynolds et al., 2008; Reynolds et al., 2009)**

<b>Parameter</b>	<b>Threshold Value for Good SPQ</b>
Air Capacity	$> 0.10 \text{ cm}^3/\text{cm}^3$
Plant Available Water Content	$> 0.20 \text{ cm}^3/\text{cm}^3$
Relative Field Capacity (RFC)	$0.6 < \text{RFC} < 0.7$
Macroporosity	$> 0.05 - 0.10 \text{ cm}^3/\text{cm}^3$

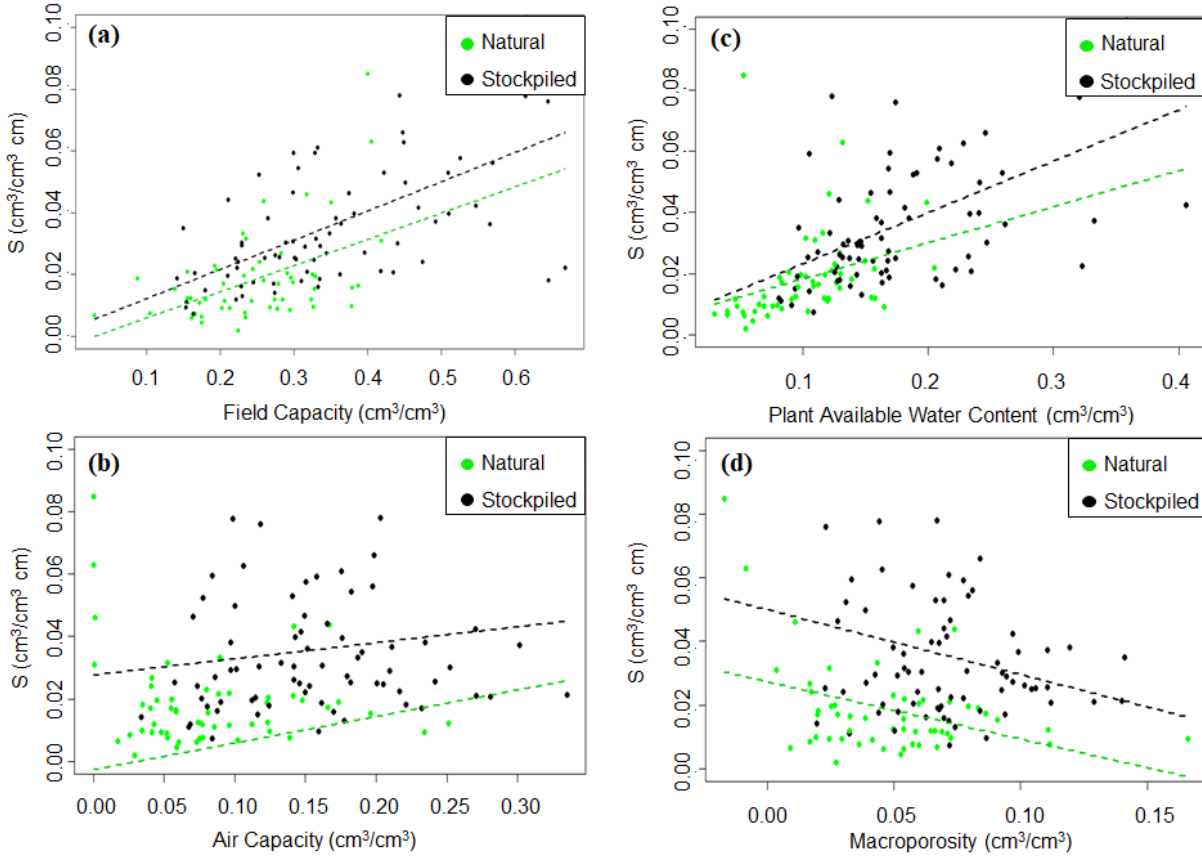
Stockpiled soils often met these threshold values regardless of depth, especially for AC and macroporosity, whereas natural sites typically only met the threshold at the soil surface (Table 2.10; Table 2.12). While both stockpiled and natural sites failed to surpass the PAWC threshold, stockpiled sites were much closer to doing so across all depths than natural sites (Table 2.10; Table 2.12). RFC was not used as a parameter in this study; however, it was calculated to provide quick reference to the threshold listed in Table 2.12. Both natural and stockpiled soils exceeded the minimal RFC value regardless of depth.

Considering the thresholds, along with the basic soil parameters discussed in section 2.5.1, leads to some interesting ecological implications for stockpiled and natural soils. While both natural and stockpiled sites were able to hold a sufficient amount of water against free drainage, demonstrated by high and uniform field capacity values between depths, a larger amount of this



water remains unavailable for plant uptake in natural sites. This is likely due to higher clay contents and bulk densities characteristic of natural soils, leading to an increasing preferential creation of small textural pores rather than the larger macropores seen uniformly throughout the stockpiled soils (Salter and Williams, 1965). Small textural pores dominate water retention characteristics as a soil dries toward the PWP; the increasingly close contact of water to soil colloids in these textural pores would create very low (more negative) matric potentials unable to be overcome by plant roots (Salter and Williams, 1965; Rawls et al., 1991). This would reduce PAWCs while leaving a larger proportion of soil pore spaces filled with water. Coupled with poor levels of macroporosity in natural soils, due to low organic matter content and high bulk density, AC values would be reduced throughout the natural soil profile as well, limiting soil aeration and proper root respiration (Pallardy, 2008). These reductions in PAWC, AC, and macroporosities are especially pronounced when surface and subsurface depths within the natural soil profile are compared, owing to the increased bulk density caused by larger quantities of illuviated clay alongside consistent levels of total carbon.

Reynolds et al. (2009) makes further use of these capacity-based measurements, comparing them to the S index. As both the capacity-based measures and S are based on the soil pore-size distribution, capacity-based measures should be correlated with S and, therefore, SPQ (Dexter, 2004; Reynolds et al., 2009). To test this connection, the S index was graphed alongside a number of different capacity based measures, shown in Figure 2.8.



**Figure 2.8: Scatter plot panel showing the relationship between  $S$  ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ), field capacity ( $\text{cm}^3 \text{cm}^{-3}$ ), plant available water content ( $\text{cm}^3 \text{cm}^{-3}$ ), air capacity ( $\text{cm}^3 \text{cm}^{-3}$ ), and macroporosity ( $\text{cm}^3 \text{cm}^{-3}$ ) parameters for both natural and stockpiled soils.**

A positive linear relationship was seen with  $S$  for all the capacity-based measurements, with the exception of macroporosity, regardless of site type (Figure 2.8; Table 2.13). As such, this supports, for the most part, the theory of Reynolds et al. (2009) and suggests that these capacity-based measurements are largely related to  $S$  and SPQ. Figure 2.8 also provides additional support that the stockpiles of this study have a better SPQ than natural sites, due to, on average, higher capacity-based measurements correlating with higher  $S$ -index values in stockpiled soils.

**Table 2.13: Regression equations, correlation coefficients, and p-values for each scatterplot panel in Figure 2.8. Overall regression trends were determined on all data points regardless of site type (natural or stockpiled) and individual regressions were also determined for natural and stockpiled sites separately.**

Panel (a)		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
-0.004+0.11*x	0.32	0.00
<b>Natural</b>		
Equation	R <sup>2</sup>	P
-0.003+0.08*x	0.22	0.00
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
0.003+0.09*x	0.24	0.00

Panel (b)		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
0.019+0.07*x	0.04	0.01
<b>Natural</b>		
Equation	R <sup>2</sup>	P
-0.003+0.09*x	0.22	0.00
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
0.028+0.05*x	0.01	0.27

Panel (c)		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
0.002+0.18*x	0.27	0.00
<b>Natural</b>		
Equation	R <sup>2</sup>	P
0.006+0.12*x	0.08	0.02
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
0.006+0.17*x	0.18	0.00

Panel (d)		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
0.033-0.08*x	0.01	0.22
<b>Natural</b>		
Equation	R <sup>2</sup>	P
0.027-0.18*x	0.13	0.00
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
0.049-0.20*x	0.04	0.05

### 2.5.3. Alternative Indicators of Soil Physical Quality

Based on the findings of this study, using either basic soil parameters such as clay content, bulk density, and total soil carbon, or capacity-based measurements such as FC, AC, PAWC, and macroporosity, leads to similar findings for S and reflects higher SPQ in the stockpiled soils.

However, S as a summary of SPQ may not be truly representative of individual site conditions.

Reviews have been done on the validity of the S index and, while it may be a suitable measure of

SPQ at a landscape scale, it may not be capturing the full variability present on a localized per-site basis, variability that would be shown through separate SWRCs (Bacher et al., 2019).

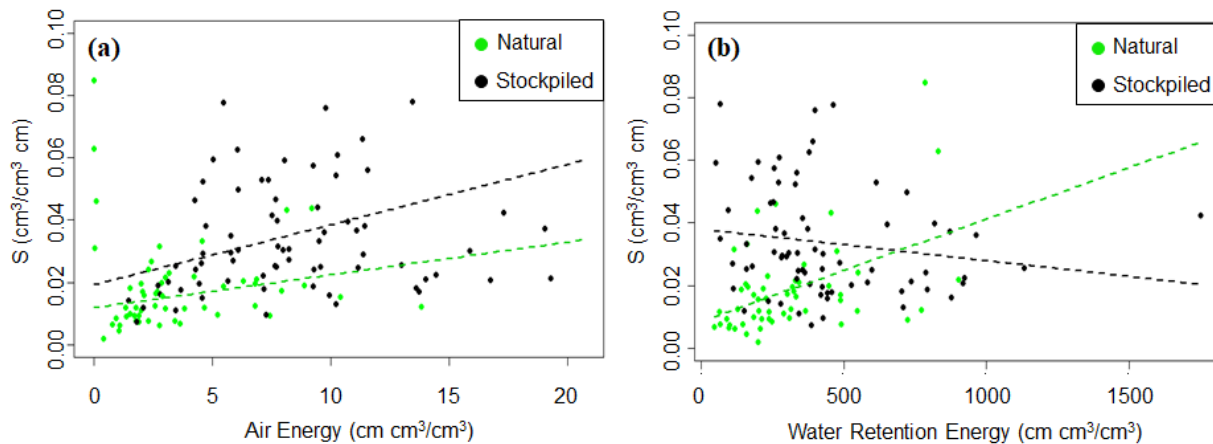
This is demonstrated in Bacher et al. (2019) and reflects a major shortcoming of S and the capacity-based measurements discussed above. Differences in macrostructure drive many of the soil water dynamics from saturation to the inflection point of a SWRC and it is the slope at the inflection point that determines S (Dexter, 2004). Stockpiled and natural soils, due to differences in soil structure, would likely have different SWRCs; however, it is possible that even if the SWRC differs between two sites, identical values could be generated for FC, PWP, and S, masking any structural differences between sites.

To overcome this obstacle, absolute air ( $A_a$ ) and water retention energies ( $WR_a$ ) can be used instead (Armino and Wendroth, 2016). These two parameters have been shown to be more sensitive to differences in soil structure on a per-site basis, and so may better represent overall SPQ (Bacher et al., 2019). This added sensitivity comes from the fact that these energy parameters are not based on single points such as FC, PWP, and S; instead, they represent the area under specific intervals of a SWRC, revolving around the FC of any given soil (Figure 2.4). It is possible that identical FC values can still be found between different soils; however, the shape of the curve, from saturation to FC and from FC to PWP, may differ drastically due to soil structure. This would change the underlying area of the curve, resulting in different air and retention energy values.

In this study, natural sites had lower air and water retention energies than stockpiles at the two subsurface depths, results that support the original project hypotheses. While there is no literature that uses these indices as a means to assess stockpile SPQ, a previous study compared

several managed soils to natural sites across a variety of different climates. In that study, natural soils typically had lower  $WR_a$  values when compared to managed soils, though  $A_a$  trends were inconsistent (Armando and Wendroth, 2016). No differences in air or retention energies were observed within stockpiles between sampling depths, whereas air and retention energies were found to decrease with increasing depth within natural sites, results that support and refute, respectively, the original hypotheses of this study.

The results of these two energy indices are interesting because, at first glance, they show a different trend than both the basic soil parameters and capacity-based measurements. Figure 2.9 shows the comparison between each energy parameter and  $S$  with there typically being a positive linear relationship between each energy parameter and  $S$  regardless of site type, and larger energy values in stockpiles than in natural sites (Figure 2.9; Table 2.14).



**Figure 2.9: Scatter plot panel showing the relationship between  $S$  ( $\text{cm}^3 \text{ cm}^{-3} \text{ cm}^{-1}$ ), air energy ( $\text{cm}^3 \text{ cm}^{-3}$ ), and water retention energy ( $\text{cm}^3 \text{ cm}^{-3}$ ) parameters for both natural and stockpiled soils.**

**Table 2.14: Regression equations, correlation coefficients, and p-values for each scatterplot panel in Figure 2.9. Overall regression trends were determined on all data points regardless of site type (natural or stockpiled) and individual regressions were also determined for natural and stockpiled sites separately.**

<b>Panel (a)</b>			<b>Panel (b)</b>		
<b>Overall</b>			<b>Overall</b>		
Equation	R <sup>2</sup>	P	Equation	R <sup>2</sup>	P
0.015+0.002*x	0.15	0.00	0.027+0.00004*x	0.00	0.59
<b>Natural</b>			<b>Natural</b>		
Equation	R <sup>2</sup>	P	Equation	R <sup>2</sup>	P
0.012+0.001*x	0.09	0.02	0.008+0.00003*x	0.19	0.00
<b>Stockpiled</b>			<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P	Equation	R <sup>2</sup>	P
0.019+0.002*x	0.10	0.00	0.038-0.00001	0.01	0.16

Lower values of the  $A_a$  and  $WR_a$  parameters are generally associated with greater SPQ than higher values. When AC is equal, in the soil with a lower  $A_a$ , aeration and oxygen availability is superior. Air can rapidly permeate the soil profile, a process critical for normal root respiration (Pallardy, 2008). Likewise, in soils with equal PAWC, in the soil with a lower  $WR_a$ , plants have to exert lower amounts of energy to extract water from soil pores (Armino and Wendroth, 2016). Both of these situations denote better growing conditions for plants. However, lower  $A_a$  and  $WR_a$  values of the natural soils of this study correspond to lower S values, giving contradictory results with respect to SPQ. As well, air and water retention energies are intimately linked to AC and PAWC, both of which were lower in natural soils. Therefore, it is not immediately clear whether low energy parameters in natural soils are truly due to higher SPQ, or to lower capacity-based measurements.

Explanations for these results likely tie largely to the role soil organic matter plays in water retention within a given soil. Soil organic matter has been identified as a player in influencing

both air and retention energies, as they will ultimately influence the shape of a SWRC (Armando and Wendroth, 2016). As well, organic matter is capable of retaining large quantities of water (Chen and Schnitzer, 1976). Stockpiles, which exhibited consistently higher and relatively uniform levels of soil carbon, had higher FC and PAWC, likely due to the adsorption of water to *in situ* SOM. This SOM subsequently created a more porous soil, increasing AC and macroporosities. However, water adsorbed to soil organic matter is held tightly, preventing it from easily draining through the profile. Further, AC and PAWC do not give any information on pore continuity and connectivity. As such, it is possible that the stockpiled soils could remain saturated for longer periods of time, subsequently increasing the amount of time it takes for a soil to drain and air to permeate the profile. This would lead to higher air energies. Likewise, the tight bond formed between SOM and water molecules and lack of pore continuity would require significantly more energy required for plant roots to extract water from the soil.

This relationship between soil organic matter also explains the trends observed within natural and stockpiled soils. Stockpiles, homogenized at placement with relatively uniform distributions of soil carbon, exhibited no change in air or retention energies with depth as the soil carbon was able to consistently hold water against drainage or plant uptake. Natural sites however, while not exhibiting a decrease in total carbon with depth, likely saw a difference in the composition of that carbon pool with an increase in depth. At the surface, the majority of the carbon would be plant based, which would, like in stockpiles, retain a large amount of water against drainage and plant uptake. With increasing depth however, the carbon pool would likely switch to one dominated by microbial-based carbon, which may not adsorb water as strongly.

Nonetheless, when the results of the basic soil properties, capacity-based measures, S, and energy parameters are all considered together, it is no longer apparent whether stockpiled sites ultimately have better SPQ than natural sites. More work on this area, as a result, may be needed.

## **2.6. CONCLUSIONS**

Stockpiled soils differed from natural sites when using basic soil properties, capacity-based measurements, S, and energy parameters, in ways that are largely contrary to much of the current literature base available. As well, the majority of these groups of SPQ indicators show that stockpiled materials, regardless of depth, have not suffered a decline in SPQ following disturbance; in fact, stockpiled materials actually have an improved SPQ when compared to their natural soil counterparts. This consistency seems to suggest that these groups of SPQ indicators are suitable to assess differences between stockpiled and natural soils in Alberta's boreal forest. They also largely demonstrate that there may be little concern with stockpiled soils with regards to reclamation suitability and success based on SPQ alone. However, it is worth noting that potential contradicting results may still exist when comparing energy parameters to S, capacity-based measurements, and basic soil properties.



### **III. INFLUENCE OF STOCKPILING ON SOIL AGGREGATE STRUCTURE, CARBON DISTRIBUTION, AND CARBON DYNAMICS**

#### **3.1. INTRODUCTION**

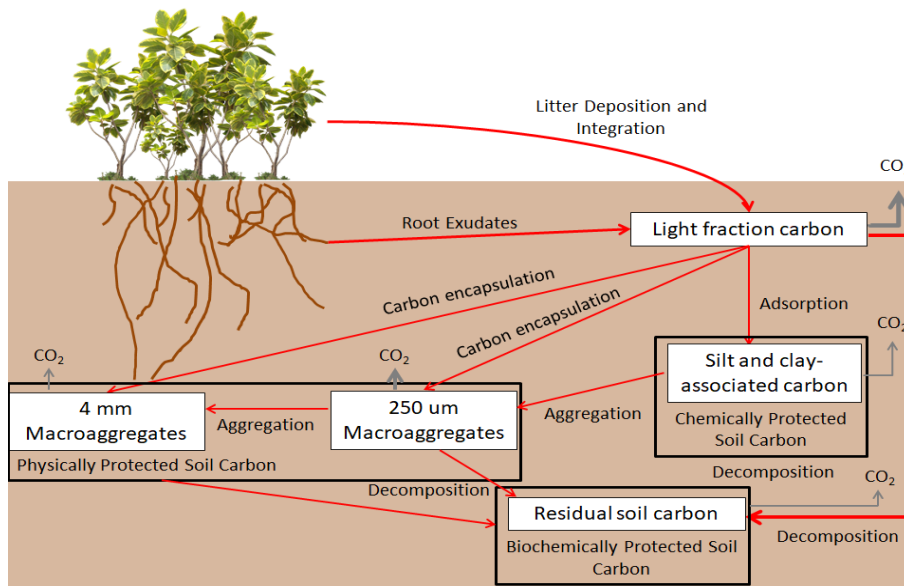
Oil sands developments in Alberta's boreal forest represent a major anthropogenic disturbance to the landscape. To mitigate this footprint over the long term, these disturbed areas must be reclaimed in accordance with provincial legislation. Land reclamation in Alberta focuses on equivalent land capability, meaning that reclaimed land needs to be able to support similar land uses as to what existed prior to disturbance (Government of Alberta, 1993). However, there is oftentimes an interest in trying to not only return a similar function to the landscape, but to create a self-sustaining and functional forest similar in both composition and trajectory to surrounding native areas (Alberta Environment, 2010; Pinno and Hawkes, 2015).

As such, using the appropriate soil is crucial to reclamation success. The most preferable resource to use is salvaged topsoil, which contains adequate plant propagules, soil carbon, mineralizable nutrients, and microorganism function to promote the re-establishment of native species (MacKenzie, 2011; Government of Alberta, 2013). One important source of salvaged topsoil is that which was collected prior to the onset of industrial operations, could not be directly placed, and so was stockpiled for future use (MacKenzie, 2011; Naeth et al., 2013).

Changes in soil structure, as well as the distribution and lability of soil organic matter (SOM), can occur during stockpiling, thereby impacting overall soil quality (Wick et al., 2009a). Soil quality can be divided into physical, chemical, and biological components and while each component has an influence on overall soil quality, it is also important to recognize the cascading effects and feedback cycles which link all three components together (Bacher et al., 2019).

### 3.1.1. Interactions between the Three Soil Quality Components

Structure and the distribution of SOM are two distinctly different, though strongly linked, components of soil. Soil structure is a major driver in determining overall soil physical quality (Oades, 1984; Dexter, 2004; Rabot et al., 2018). It also plays a pivotal role in influencing soil carbon dynamics, primarily due to its stabilization effects on SOM (Oades, 1984; Six et al., 2002). Several SOM stabilization processes have been identified in soils, which in turn relate to different soil physical properties (Figure 3.1). Soil texture influences the chemical stabilization mechanisms of SOM; increased proportions of primary silt and clay particles promote additional adsorption of SOM into mineral surfaces, increasing its chemical stabilization (Hassink, 1997; Six et al., 2002; Cotrufo et al., 2013).



**Figure 3.1: Diagram showing the different soil carbon pools, processes, and fluxes that influence the movement and stabilization of carbon in undisturbed natural soils.**

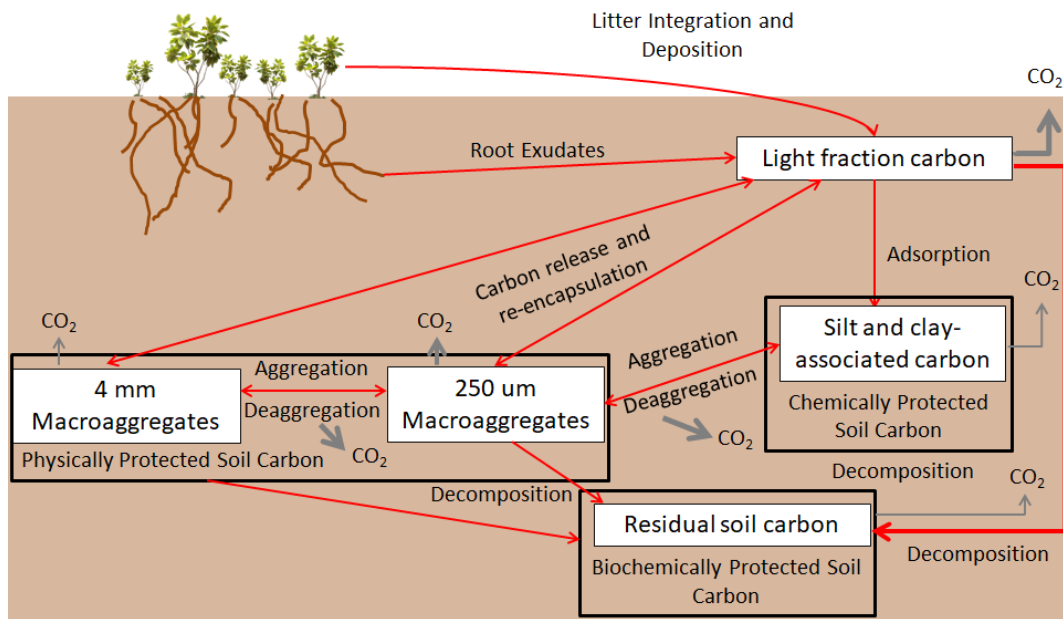
Soil aggregation provides physical protection of SOM by shielding it from microorganisms and their decomposition enzymes (Figure 3.1). Soil aggregates are divided into two categories:

microaggregates ( $< 250 \mu\text{m}$  diameter), which represent a consolidation of primary silt and clay particles, and macroaggregates ( $\geq 250 \mu\text{m}$  diameter), which result from the consolidation of these separate microaggregates (Tisdall and Oades, 1982; Oades, 1984). Shielding of SOM occurs in both aggregate sizes, although the extent of shielding is related to the pore size distribution of the soil and the aggregates themselves. Smaller pores, characteristic of microaggregates, are able to size-exclude a larger segment of the microbial population, potentially increasing the amount of carbon associated with microaggregates and smaller macroaggregates (Killham et al., 1993). However, as macroaggregates are formed primarily from the influence of organic binding agents, higher levels of labile organic material may be contained in macroaggregates, despite lower carbon quantities (Yamashita et al., 2006; Tan et al., 2007).

SOM that is not associated with silt or clay particles, or remains on the outside of microaggregates or larger macroaggregates, can become increasingly biochemically stabilized (Figure 3.1). This is because this SOM is exposed to the influence of microorganisms and their digestive enzymes, thereby advancing the stage of SOM decomposition, creating or increasing the proportion of more chemically complex functional groups, and increasing the inherent recalcitrance of the material (Hattori, 1989; Golchin et al., 1994; Six et al., 2002; Cotrufo et al., 2013). It is important to note that more recent work questions the importance of biochemical stabilization; while the chemical composition of SOM does influence decomposition rates of soil carbon, it plays more of a secondary role when compared to chemical and physical stabilization mechanisms on overall carbon stability and turnover (Schmidt et al., 2011).

Regardless, the way soil carbon is stabilized likely influences how it responds to different types of disturbance (Figure 3.2). While carbon associated with free silt and clay particles could

remain largely unchanged due to chemical stabilization mechanisms, large destructive disturbances can break apart both the macroaggregate and microaggregate structure of a soil (Figure 3.2). This increases the amount of free silt and clay particles, while simultaneously liberating carbon originally protected within the aggregate structure (Six et al., 2002; Tan et al., 2007; Wick et al., 2009a). Microorganisms would then be able to break down this exposed carbon, reducing total carbon levels and biochemically stabilizing residual SOM (Figure 3.2; Hunter and Currie, 1956; Six et al., 2002; Tan et al., 2007). Reduced carbon quantities would reduce aggregation potential through the loss of organic binding agents, reducing structural integrity (Oades, 1984). This feedback loop would have subsequent implications on the suitability of stockpiled soils for reclamation, due to changes in overall soil physical quality and altered nutrient cycling processes critical for native overstory and understory vegetation species (Oades, 1984; Wick et al., 2009a; Pinno and Errington, 2015; Macdonald et al., 2015).



**Figure 3.2: Diagram showing the different soil carbon pools, processes and fluxes that influence the movement and stabilization of carbon in stockpiled soils.**

### 3.1.2. Measures of Carbon Dynamics in Soil

The interconnection between soil structure, overall soil physical quality, and carbon stabilization highlights the importance of understanding *in situ* carbon dynamics. One of the most direct ways to assess carbon dynamics is to measure soil microbial respiration, which represents the amount of carbon dioxide emitted during oxidative decomposition processes (Schlesinger and Andrews, 2000). Greater levels of microbial activity would typically correspond to higher basal respiration levels measured for a given soil. Higher levels of soil carbon, on the other hand, do not necessarily imply the same. Different carbon pools have different overall carbon lability, resulting in different respiration rates from the microbes that decompose them. A number of parameters can be measured that relate to the lability of soil carbon: light fraction carbon proportions, C:N ratios, and stable carbon isotopes, such as  $^{13}\text{C}$ .

Determining light fraction carbon (LFC) quantities provides an idea of the relative proportions of the more labile and less humified plant-based carbon available for microbial decomposition. LFC has been defined as organic material with a specific density  $< 2.0 \text{ g cm}^{-1}$ , oftentimes representing only a small proportion of the overall soil mass (Gregorich and Janzen, 1996). However, it is a critically important component of total soil carbon as it is the site of large amounts of *in situ* decomposition due to its enrichment in labile carbohydrates and available nitrogen (Gregorich and Janzen, 1996; Tan et al., 2007).

The C:N ratio can be used to determine two complementary pieces of information. First, it can give an idea as to the amount of nitrogen available for microbial use; lower C:N of litter and other plant-based materials in soil indicates a greater availability of nitrogen, important in terrestrial ecosystems that are often nitrogen limited (Gregorich and Janzen, 1996; LeBauer and

Treseder, 2008). Microbes have very low C:N ratios due to their high metabolic requirements for nitrogen; as such, they will more actively decompose plant materials with a lower C:N (Enríquez et al., 1993; Gregorich and Janzen, 1996; Fernandez et al., 2003; Garten Jr. et al., 2007; Cotrufo et al., 2013). Secondly, the C:N ratio can provide information relating to the extent of SOM decomposition, utilizing the concept of substrate use efficiency. Substrate use efficiency is the amount of a substrate used for growth and enzyme production *versus* the amount that is mineralized or respired (Lekkerkerk et al., 1990). More labile plant materials, with a lower C:N ratio, correspond to a higher substrate use efficiency, indicating that a greater proportion of the original material is absorbed by microorganisms and converted to biomass or products such as enzymes. The continuous decomposition of the original plant matter would see increasing C:N ratios for the residual plant material; however, the overall soil can retain or develop lower C:N ratios, as plant-based carbon is continuously respired from the soil and nitrogen, mineralized during decomposition, is retained in the soil by the microbial population. In turn, this would suggest an advanced state of decomposition for *in situ* SOM (Cotrufo et al., 2013).

Stable heavy isotopes, such as  $^{13}\text{C}$ , can also provide an idea as to the extent of decomposition and rates of carbon turnover (Sulzman, 2007). Differences in the amount of  $^{13}\text{C}$  can result from alterations in vegetation type (C3 versus C4) following disturbance; however, if the type of vegetation remains consistent pre- and post-disturbance, then differences in isotopic compositions can be the result of fractionation mechanisms during microbial processes (Fernandez et al., 2003; Garten Jr. et al., 2007). This entails a preferential release of  $^{13}\text{C}$  depleted carbon dioxide during respiration, subsequently enriching the remaining soil carbon pool with heavy carbon isotopes (Quideau et al., 2003; Fernandez et al., 2003; Wynn et al., 2006).

These three properties of SOM (LFC, C:N and  $^{13}\text{C}$ ) reflect the lability and extent of decomposition of SOM. It is possible that by considering these three parameters together, a link can be made to soil respiration, allowing measures of carbon lability to serve as a proxy for carbon dynamics in a given soil.

### **3.1.3. Current State of Understanding in the Literature**

A number of studies have been conducted on structural alterations in stockpiles, primarily through changes to aggregate distributions. Abdul-Kareem and McRae (1984) found that, when compared to undisturbed natural soils, stockpiled material had drastic declines in macroaggregate proportions. Similar results were found by Hunter and Currie (1956), where macroaggregate structure could not be assessed because of the larger amount of free silt and clay particles present. Wick et al. (2009) also found declines in the proportion of macroaggregates present in stockpiled soil. Few studies have been done that looked at carbon quantities associated with different aggregate sizes, as well as aggregate associated and whole soil carbon quality, in stockpiled materials (Wick et al., 2009b). In one study, organic carbon concentrations across all aggregate size classes were been found to be significantly lower in stockpiled than natural soils (Wick et al., 2009b).

Various results have been found regarding microbial respiration rates in both reclaimed and stockpiled materials. When newly salvaged reclamation substrates were compared to the same substrates that had been previously stockpiled, the stockpiled substrates had higher basal respiration levels (Gupta et al., 2019). Visser et al. (1984) found that microbial activity was lowest at the stockpile surface that subsequently increased with depth, while Williamson and Johnson (1990) found no changes in respiration levels with increases in stockpile depth. When

comparing mining disturbed soils to nearby reference sites, Visser et al. (1983) found lower respiration levels in the disturbed soils.

Light fraction carbon has been found to decrease in stockpiles when compared to natural reference sites (Wick et al., 2009b). Similar results have been noted in a number of different studies, where, as a result of disturbance, a portion of labile *in situ* organic matter is either completely lost through microbial respiration or converted into less active, more recalcitrant forms (Hunter and Currie, 1956; Schwenke et al., 2000b; a; Turcotte et al., 2009).

Banning et al. (2008) found that C:N ratios in rehabilitated forest sites following mining were lower than in nearby reference sites. Opposite findings can be found in Wick et al. (2009a), where reclaimed sites had much higher C:N than comparable natural reference sites. However, Ingram et al. (2005) found that C:N did not differ between natural and reclaimed sites, while Williamson and Johnson (1990) found that C:N also remained consistent with stockpile depth.

Little work has been done using stable carbon isotopes to assess carbon quality in stockpiles. One study done found that stockpiled soils did not experience changes in  $^{13}\text{C}$  concentrations over a three year period, despite additions of new organic matter (Wick et al., 2009b). Similar results were found when disturbed and undisturbed boreal forest sites were compared to each other (Turcotte et al., 2009).

The above literature shows that there has been limited work on characterizing stockpiling impacts on aggregate distributions and associated aggregate carbon, as well as assessing carbon quality indicators such as basal respiration rates, LFC proportions, C:N, and stable carbon isotopes. Furthermore, there is a large variability in the findings of these studies, and little work has been done in synthesizing carbon quality indicators in an effort to develop a relationship with



soil respiration rates. There has also been minimal effort to link soil carbon attributes, soil carbon dynamics, and structural changes in stockpiles to get a more comprehensive picture on what alterations are occurring to topsoil quality following disturbance and storage.

### **3.2. RESEARCH OBJECTIVES**

The objective of this chapter is to quantify the impacts of stockpiling on soil carbon distribution and dynamics, linking these to changes in soil aggregate structure. The focus will be on mineral top soils from the Alberta boreal forest, particularly those associated with both open pit mining and *in situ* SAGD operations. Soils will be assessed using alterations in aggregate and carbon distributions, light fraction carbon proportions, basal respiration rates, C:N ratios, and stable isotopic concentrations, between natural and stockpiled soils, over a variety of depths.

#### **3.2.1. Research Questions**

Throughout this chapter, the following research questions will be explored:

1. To what extent does stockpiling, over a variety of depths, shift soil aggregates from larger to smaller macro-aggregates, alter aggregate associated carbon quantities, and shift whole soil light fraction carbon proportions when compared to natural reference sites?
2. How do soil respiration rates differ between stockpiled and natural reference sites across a variety of depths?
3. How do measurements of soil carbon quality such as C:N ratios, light fraction carbon proportions, and stable isotope concentrations ( $^{13}\text{C}$ ), considered alongside aggregate carbon attributes ( $^{13}\text{C}$ , C:N), relate to soil respiration?

### 3.2.2. Hypotheses

Stockpiling is expected to result in a shift from larger macroaggregates to smaller macroaggregates when compared to natural sites, as these aggregates will be broken up by the destructive forces of soil salvage, transport, and storage. Aggregate associated carbon quantities and light fraction carbon proportions are expected to be lower in stockpiled material than natural sites, due to the fracturing of soil aggregates and subsequent exposure of previously shielded organic matter to microbial decomposition enzymes. However, greater quantities of carbon will remain associated with smaller macroaggregates regardless of soil type, due to size-exclusion mechanisms acting on microbial communities.

Increased exposure of previously sheltered organic material to the microbial community and their decomposition enzymes, along with higher total carbon quantities in stockpiles sourced from integrated LFH material during salvage and storage, will likely result in elevated basal respiration rates in stockpiled soils compared to natural sites. This would then be accompanied by a decrease in C:N ratios and an enrichment (less negative values) in  $^{13}\text{C}$ , as decomposition of more labile soil carbon proceeds.

It is expected that greater proportions of large macroaggregates, aggregate associated carbon quantities, and light fraction carbon quantities will be observed within both the stockpile and natural site surface depths than at subsurface depths, due to the influence of re-establishing and existing vegetation on soil organic matter inputs and the dominant role that soil carbon plays in large macroaggregate formation. As a result, within both the stockpiles and natural soils, C:N ratios will decrease and  $^{13}\text{C}$  concentrations will increase with depth, at both the aggregate and

whole soil scale. Respiration levels are also expected to decline with depth within both the stockpiles and natural soils, due to the lack of labile carbon inputs at depth.

### **3.3. METHODS**

#### **3.3.1. Field Site Description**

This study took place on Canadian Natural Resources Limited's (CNRL) Horizon and Wolf Lake sites. Description of the vegetation and soils can be found in section 2.3.1.

#### **3.3.2. Field Methods**

The field site plot layout and sampling design are described in greater detail in section 2.3.2. In short, eight stockpiles and six natural sites were sampled between CNRL Horizon and Wolf Lake. Three soil pits were dug on each stockpile and natural site to a depth of 90 cm, with three sampling depths being established at 0 to 10 cm, 20 to 30 cm, and 80 to 90 cm. Samples were taken from each depth in the form of a soil core and bulk soil collection, which were kept refrigerated and chilled while on site, during transport, and when stored in the lab.

#### **3.3.3. Laboratory Methods**

##### **3.3.3.1. Site Descriptor Variables**

Bulk soils were analyzed for gravimetric water content, pH, electrical conductivity (EC), and texture (section 2.3.3.1; Kalra and Maynard, 1991; Topp et al., 2008).

##### **3.3.3.2. Water Stable Aggregates and Associated Carbon**

Aggregate size distributions were quantified on bulk soil samples using a large soil washing apparatus and the methodology outlined in Mehuys et al. (2007). Water levels were set to

completely submerge aggregates in the top sieve during the lowest part of the washing cycle. Aggregates were washed for 10 minutes, removed from each sieve, transported to drying tins, weighed, and placed in an oven at 105°C for 24 hours. After this time, tins were weighed again and the aggregates transferred to a beaker. Aggregates were sonicated using a Q700 QSonica ultrasonic probe for one minute at an amplitude of 23 A (Mentler et al., 2004). Samples were then run through their respective original sieve size, the primary particles and aggregates were collected into drying tins, and were dried as above. The tins were re-weighed and the samples bagged. Collected aggregates were ground in a Retsch MM200 ball grinder at a frequency of 30 Hz for one minute. They were then analysed at the Natural Resources Analytical Lab (NRAL) at the University of Alberta for total carbon, total nitrogen, and <sup>13</sup>C abundance analyses using a ThermoFinnigan Delta V Advantage Continuous Flow Isotope Ratio Mass Spectrometer.

### **3.3.3.3. CLPP and Basal Respiration Rates**

To determine basal respiration rates, incubations were run in sets of 20 samples per day three days a week. Bulk soil samples were sieved at field water contents through an 8 mm sieve, with 100 g of soil being added to a plastic bag. 100 g of sterile sand was added to improve soil workability. The bags were agitated to mix the soil and sand and were incubated at 60% of field capacity, determined using pressure plate extractors, at 25°C for two weeks. Anaerobic test strips were placed in 25% of the bags to monitor for aerobic conditions, with bags being vented every two days for 10 minutes to maintain conditions. Following the two week period, each batch of 20 was removed from the incubator and prepped for community-level physiological profiling (CLPP), using the method and equipment similar to the ones described in Campbell et al. (2003) and Howell and Mackenzie (2017). A six hour incubation period was used to determine basal carbon mineralization rates for 1.1 g of soil on average in 50 µl of deionized water.

#### **3.3.3.4. Light Fraction Carbon**

Light fraction carbon (LFC) proportions were determined using the methodology outlined in Gregorich and Beare (2008). 15 g of mineral soil, or 5 g for soils that visually contained more organic material such as peat, along with 40 mL of 1.7 g/cm<sup>3</sup> sodium iodide, was placed into plastic Nalgene bottles. Bottles were loaded onto a mechanical shaker for 30 minutes at a low speed, and then were set aside to stand for one day at room temperature. The separated LFC was removed using a vacuum apparatus, with the collected LFC being allowed to air dry for 48 hours. Dried LFC was ground using a Retsch MM200 ball grinder at a frequency of 30 Hz for a period of 15 seconds, and returned to NRAL for total carbon, total nitrogen, and C<sup>13</sup> measurements using a ThermoFinnigan Delta V Advantage Continuous Flow Isotope Ratio Mass Spectrometer.

#### **3.3.4. Statistical Analysis**

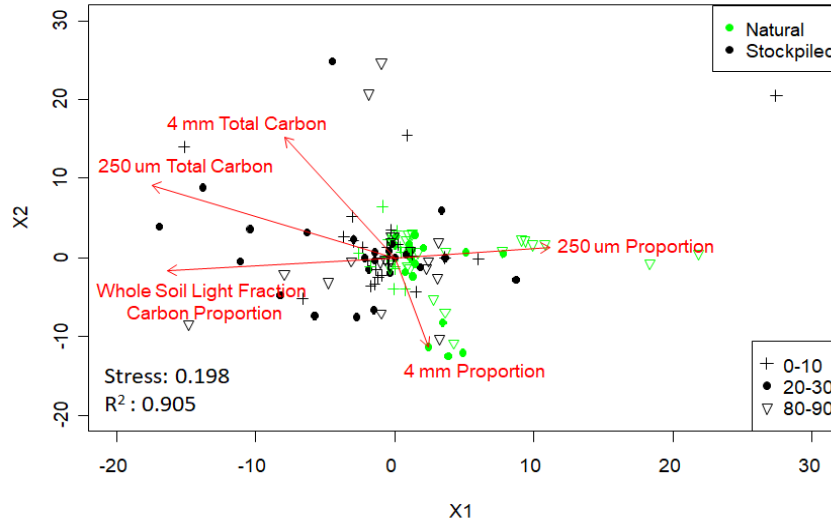
A detailed outline for the statistical analyses used for this project can be found in section 2.3.4. In short, variables were ordinated using the multivariate non-metric multidimensional scaling (NMDS) technique by both site type and depth, with significant differences being assessed using a perMANOVA and pairwise comparisons. Linear mixed models were then applied to better identify if and by how much the groups differed from each other. These results were then synthesized into summary plots and figures to visualize points made in the discussion.

### **3.4. RESULTS**

#### **3.4.1. Site-Depth Comparisons**

The proportions of 4 mm and 250 µm aggregates were higher in natural sites than in stockpiles. In contrast, total carbon quantities for both aggregate size classes, as well as whole soil LFC

proportions, were higher in stockpiled soils (Figure 3.3). For the five parameters ordinated in Figure 3.3, natural and stockpiled soils differed only at the 20 to 30 cm ( $p=0.032$ ) and 80 to 90 cm ( $p=0.015$ ) sampling depths (Table 3.1).



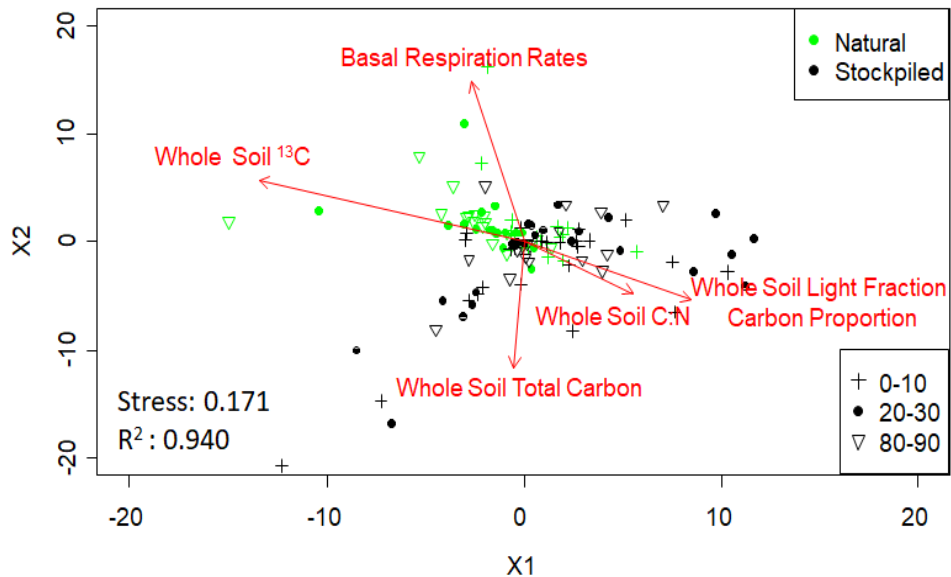
**Figure 3.3: NMDS ordination for the 4 mm and 250  $\mu$ m water stable aggregate proportions, aggregate associated total carbon quantities (%), and whole soil light fraction carbon proportions by site type and sampling depth.**

**Table 3.1: perMANOVA pairwise comparisons for 4 mm and 250  $\mu$ m water stable aggregate proportions, aggregate associated total carbon quantities (%), and whole soil light fraction carbon proportions ordinated in Figure 3.3. Significant differences were declared for  $p<0.05$  (bolded in table).**

Comparisons	0-10 Natural	0-10 Stockpiled	20-30 Natural	20-30 Stockpiled	80-90 Natural
0-10 Stockpiled	0.273	-	-	-	-
20-30 Natural	<b>0.015</b>	<b>0.020</b>	-	-	-
20-30 Stockpiled	0.288	1.000	<b>0.032</b>	-	-
80-90 Natural	<b>0.015</b>	<b>0.015</b>	1.000	<b>0.027</b>	-
80-90 Stockpiled	0.350	1.000	<b>0.015</b>	1.000	<b>0.015</b>

Within stockpiled sites, no significant differences among sampling depths was observed for any of the five ordinated parameters ( $p=1.000$ ); however, within natural sites, these properties were significantly different in the 0 to 10 cm depth from the 20 to 30 cm depth ( $p=0.015$ ) and the 80 to 90 cm depth ( $p=0.015$ ) (Table 3.1).

Basal respiration rates and whole soil  $^{13}\text{C}$  concentrations were higher in natural sites, whereas whole soil total carbon, light fraction proportions, and C:N ratios were higher in stockpiled soils (Figure 3.4). For the five parameters ordinated in Figure 3.4, natural and stockpiled sites only differed at the 0 to 10 cm ( $p=0.022$ ) and 20 to 30 cm ( $p=0.015$ ) depths (Table 3.2). Within stockpiled and natural sites, no significant differences were observed for these five parameters between any of the sampling depths ( $p=1.000$ ) (Table 3.2).

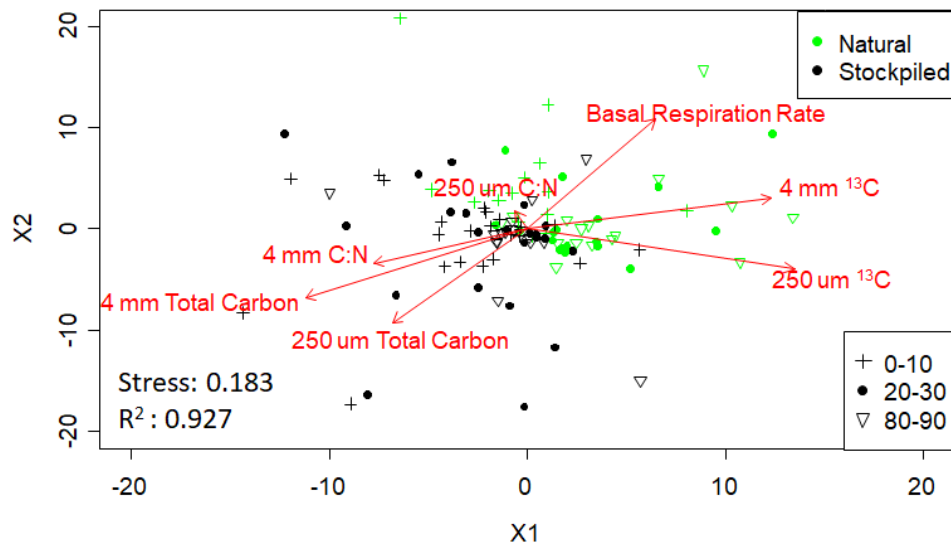


**Figure 3.4: NMDS ordination for basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ), whole soil total carbon (%), light fraction carbon proportions, C:N ratios, and  $^{13}\text{C}$  isotopic concentrations by site type and sampling depth.**

**Table 3.2: perMANOVA pairwise comparisons basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ), whole soil total carbon (%), light fraction carbon proportions, C:N ratios, and  $^{13}\text{C}$  isotopic concentration parameters ordinated in Figure 3.4. Significant differences were declared for  $p < 0.05$  (bolded in table).**

Comparisons	0-10 Natural	0-10 Stockpiled	20-30 Natural	20-30 Stockpiled	80-90 Natural
0-10 Stockpiled	<b>0.022</b>	-	-	-	-
20-30 Natural	1.000	<b>0.015</b>	-	-	-
20-30 Stockpiled	<b>0.030</b>	1.000	<b>0.015</b>	-	-
80-90 Natural	1.000	<b>0.015</b>	1.000	<b>0.015</b>	-
80-90 Stockpiled	1.000	1.000	1.000	1.000	1.000

4 mm and 250  $\mu\text{m}$  aggregate  $^{13}\text{C}$  concentrations were higher in natural sites, corresponding with higher basal respiration rates, while C:N ratios were higher in stockpiled sites, corresponding with higher aggregate total carbon quantities (Figure 3.5).



**Figure 3.5: NMDS ordination for basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) and 4 mm and 250  $\mu\text{m}$  aggregate total carbon (%), C:N ratios, and stable carbon isotope ( $^{13}\text{C}$ ) parameters by site type and sampling depth.**



**Table 3.3: perMANOVA pairwise comparisons for basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) and 4 mm and 250  $\mu\text{m}$  aggregate total carbon, C:N ratios, and stable carbon isotope ( $^{13}\text{C}$ ) parameters ordinated in Figure 3.5. Significant differences were declared for  $p < 0.05$  (bolded in table).**

Comparisons					
	<b>0-10 Natural</b>	<b>0-10 Stockpiled</b>	<b>20-30 Natural</b>	<b>20-30 Stockpiled</b>	<b>80-90 Natural</b>
<b>0-10 Stockpiled</b>	<b>0.022</b>	-	-	-	-
<b>20-30 Natural</b>	1.000	<b>0.015</b>	-	-	-
<b>20-30 Stockpiled</b>	<b>0.022</b>	1.000	<b>0.015</b>	-	-
<b>80-90 Natural</b>	1.000	<b>0.015</b>	1.000	<b>0.015</b>	-
<b>80-90 Stockpiled</b>	1.000	1.000	1.000	1.000	1.000

For the seven parameters ordinated in Figure 3.5, natural and stockpiled sites only differed at the 0 to 10 cm ( $p=0.022$ ) and 20 to 30 cm ( $p=0.015$ ) depths (Table 3.3). Within stockpiled and natural sites, no significant differences were observed for these five parameters between any of the sampling depths ( $p=1.000$ ) (Table 3.3).

### **3.4.2. Aggregate Size Distributions and Associated Carbon Contents**

4 mm (overall mean = 0.28) and 250  $\mu\text{m}$  (overall mean = 0.15) aggregate proportions were higher in natural sites at the 0 to 10 cm and 20 to 30 cm depth, whereas stockpiled sites had higher levels of both aggregate sizes (overall mean = 0.23; 0.04, respectively) at the 80 to 90 cm depth. However, these differences were not significant for any depth ( $p > 0.05$ ) (Table 3.4).

Within stockpiled sites, no significant differences in aggregate proportions between depths, for both the 4 mm and 250  $\mu\text{m}$  ( $p > 0.450$ ) aggregates, were observed. Within natural sites however, significantly higher proportions of 4 mm aggregates were observed in the 20 to 30 cm depth

when compared to either the 0 to 10 cm ( $p=0.0134$ ) or the 80 to 90 cm ( $p=0.0337$ ) depth, while significantly lower proportions of 250  $\mu\text{m}$  aggregates were observed in the 80 to 90 cm depth when compared to the 0 to 10 cm ( $p=0.0002$ ) and the 20 to 30 cm ( $p=0.0027$ ) depths (Table 3.4).

**Table 3.4: Estimated marginal means and standard errors for 4 mm and 250  $\mu\text{m}$  water stable aggregate proportions, total carbon associated with each aggregate size fraction (%), and whole soil light fraction carbon (LFC) proportions. For each property, uppercase letters denote significant differences between natural and stockpiled soils at a particular depth. Lower case letters represent significant differences within natural or stockpiled site types between difference depths. Significant differences were declared for  $p<0.05$ .**

	4 mm Aggregate Proportion	250 $\mu\text{m}$ Aggregate Proportion	4 mm Aggregate Carbon (%)	250 $\mu\text{m}$ Aggregate Carbon (%)	LFC Proportion
<b>Depth=0-10 cm</b>					
Site= Natural	0.24 $\pm$ 0.07 Aa	0.16 $\pm$ 0.01 Aa	1.55 $\pm$ 1.59 Aa	3.87 $\pm$ 2.31 Aa	0.20 $\pm$ 0.06 Aa
Site= Stockpiled	0.20 $\pm$ 0.06 Aa	0.04 $\pm$ 0.01 Aa	5.78 $\pm$ 1.37 Ba	9.04 $\pm$ 1.96 Aa	0.39 $\pm$ 0.05 Ba
<b>Depth=20-30 cm</b>					
Site= Natural	0.38 $\pm$ 0.07 Ab	0.26 $\pm$ 0.01 Aa	0.70 $\pm$ 1.56 Ab	1.25 $\pm$ 2.26 Ab	0.08 $\pm$ 0.05 Ab
Site= Stockpiled	0.20 $\pm$ 0.06 Aa	0.03 $\pm$ 0.01 Aa	5.22 $\pm$ 1.37 Ba	9.82 $\pm$ 1.96 Ba	0.41 $\pm$ 0.05 Ba
<b>Depth=80-90 cm</b>					
Site= Natural	0.26 $\pm$ 0.07 Aa	0.03 $\pm$ 0.01 Ab	0.76 $\pm$ 1.56 Ab	0.97 $\pm$ 2.26 Ab	0.06 $\pm$ 0.05 Ab
Site= Stockpiled	0.29 $\pm$ 0.06 Aa	0.06 $\pm$ 0.01 Aa	4.44 $\pm$ 1.42 Ba	8.09 $\pm$ 2.03 Ba	0.34 $\pm$ 0.05 Ba

Carbon contents for the 4 mm aggregate size class were significantly higher at all three sampling depths ( $p=0.0173$ ,  $p=0.0026$ ,  $p=0.0048$ ) in stockpiled soils (overall mean = 5.15%) than in natural sites (overall mean = 1.00%). A similar pattern was found for the 250  $\mu\text{m}$  aggregate size proportions in stockpiled (overall mean = 8.98%) and natural soils (overall mean = 2.03%), though significant differences were only found at the 20 to 30 cm ( $p=0.0016$ ) and 80 to 90 cm ( $p=0.0008$ ) depths (Table 3.4). Within stockpiled soils, no significant differences in aggregate

associated carbon content were observed between depths for both the 4 mm ( $p>0.200$ ) and 250  $\mu\text{m}$  ( $p>0.950$ ) aggregate sizes. Within natural sites, however, significantly higher carbon contents, for both the 4 mm and 250  $\mu\text{m}$  aggregate sizes, were observed in the 0 to 10 cm depth when compared to either the 20 to 30 cm ( $p=0.0264$ ,  $p=0.0001$ , respectively) or 80 to 90 cm ( $p=0.0282$ ,  $p<0.0001$ , respectively) depths (Table 3.4).

### **3.4.3. Light Fraction Carbon Proportions**

Light fraction carbon was measured as a proportion of overall total soil carbon. Stockpiled soils had a greater proportion of soil carbon contained in the light fraction (overall mean = 0.38) than natural sites (overall mean = 0.11) across all depths sampled ( $p=0.0334$ ,  $p=0.0002$ ,  $p=0.0003$ ) (Table 3.4). Within stockpiled soils, no significant differences in light fraction carbon proportions were observed with depth ( $p>0.131$ ), whereas higher light fraction proportions were observed within natural sites when the 0 to 10 cm depth was compared to either the 20 to 30 cm ( $p=0.0007$ ) or the 80 to 90 cm ( $p<0.0001$ ) depths (Table 3.4).

### **3.4.4. Basal Respiration Rates**

Basal respiration rates were significantly higher in natural (overall mean = 222  $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) than in stockpile soils (overall mean = 113  $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) for all three of the depths ( $p=0.0144$ ,  $p=0.0049$ ,  $p=0.0111$ ). However, within both stockpiled and natural sites, no significant differences were observed between depths ( $p>0.300$ ) (Table 3.5).

**Table 3.5: Estimated marginal means and standard errors for basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) as well as whole soil C:N and  $^{13}\text{C}$  concentrations. For each property, uppercase letters denote significant differences between natural and stockpiled soils at a particular depth, while lower case letters represent significant differences within natural or stockpiled site types between difference depths. Significant differences were declared for  $p < 0.05$ .**

	<b>Basal Respiration (<math>\mu\text{gC gC}^{-1} \text{h}^{-1}</math>)</b>	<b>Whole Soil C:N</b>	<b>Whole Soil <math>^{13}\text{C}</math></b>
<b>Depth=0-10 cm</b>			
Site= Natural	210 $\pm$ 45 Aa	17.0 $\pm$ 2.1 Aa	-26.4 $\pm$ 0.4 Aa
Site= Stockpiled	84 $\pm$ 38 Ba	19.3 $\pm$ 1.9 Aa	-26.6 $\pm$ 0.4 Aa
<b>Depth=20-30 cm</b>			
Site= Natural	212 $\pm$ 43 Aa	13.0 $\pm$ 2.1 Ab	-25.6 $\pm$ 0.4 Aa
Site= Stockpiled	89 $\pm$ 38 Ba	20.1 $\pm$ 1.9 Ba	-27.0 $\pm$ 0.4 Ba
<b>Depth=80-90 cm</b>			
Site= Natural	245 $\pm$ 43 Aa	14.2 $\pm$ 2.1 Ab	-24.0 $\pm$ 0.4 Ab
Site= Stockpiled	166 $\pm$ 40 Ba	18.8 $\pm$ 2.0 Ba	-27.0 $\pm$ 0.4 Ba

### 3.4.5. Whole Soil C:N

C:N ratios of the whole soil were consistently lower in natural sites (overall mean = 14.7) than in stockpiled sites (overall mean = 19.4) for all depths sampled, though this difference was only significant at the 20 to 30 cm ( $p=0.0028$ ) and 80 to 90 cm ( $p=0.0301$ ) depths (Table 3.5). No significant differences were observed for C:N ratios within stockpiles between any of the sampling depths ( $p>0.350$ ), whereas within natural sites, significantly higher C:N ratios were observed when the 0 to 10 cm depth was compared to either the 20 to 30 cm ( $p=0.0015$ ) or the 80 to 90 cm depth ( $p=0.0305$ ) (Table 3.5).

### 3.4.6. Whole Soil $^{13}\text{C}$ Isotopic Composition

$^{13}\text{C}$  concentrations were less negative (more enriched) in the natural sites (overall mean = -25.3) at all three depths when compared to stockpiled soils (overall mean = -26.9), though this difference was only significant at the 20 to 30 cm ( $p=0.0246$ ) and 80 to 90 cm depths ( $p=0.0003$ ) (Table 3.5). Within stockpiled sites, no significant differences were observed in  $^{13}\text{C}$  concentrations between sampling depths ( $p>0.600$ ), whereas within natural sites, a greater enrichment of  $^{13}\text{C}$  was observed in the 80 to 90 cm depth when compared to either the 0 to 10 cm ( $p=0.0001$ ) or the 20 to 30 cm ( $p=0.0165$ ) depths (Table 3.5).

### 3.4.7. Aggregate C:N and Isotopic Composition

C:N ratios were generally lower in both the 4 mm and 250  $\mu\text{m}$  aggregate sizes of the natural sites (overall mean = 15.1; overall mean = 19.1, respectively) than in stockpiled soils (overall mean = 20.2; overall mean = 20.1, respectively), except in the 0 to 10 cm depth for the 250  $\mu\text{m}$  sized aggregates. However, these differences were only significant in the 20 to 30 cm ( $p=0.0055$ ) and 80 to 90 cm ( $p=0.0249$ ) depths, and only for the 4 mm sized aggregates (Table 3.6). No significant differences in C:N ratios were observed within stockpiled soils, between sampling depths, for both the 4 mm ( $p>0.500$ ) and 250  $\mu\text{m}$  ( $p>0.150$ ) aggregate sizes. In contrast, significantly higher C:N ratios were observed within natural sites, in both the 4 mm and 250  $\mu\text{m}$  aggregate sizes, when the 0 to 10 cm depth was compared to the 20 to 30 cm ( $p=0.0014$ ,  $p<0.0001$ , respectively) and 80 to 90 cm ( $p=0.0177$ ,  $p<0.0001$ , respectively) depths (Table 3.6).

**Table 3.6: Estimated marginal means and standard errors for C:N and <sup>13</sup>C concentrations for the 4 mm and 250 μm water stable aggregate proportions. For each property, uppercase letters denote significant differences between natural and stockpiled soils at a particular depth. Lower case letters represent significant differences within natural or stockpiled site types between difference depths. Significant differences were declared for p<0.05.**

	4 mm C:N	250 μm C:N	4 mm <sup>13</sup> C	250 μm <sup>13</sup> C
<b>Depth=0-10 cm</b>				
Site= Natural	18.0 ± 2.8 Aa	24.3 ± 2.3 Aa	-26.2 ± 0.5 Aa	-25.3 ± 0.6 Aa
Site= Stockpiled	21.3 ± 2.7 Aa	19.0 ± 2.1 Aa	-26.4 ± 0.4 Aa	-26.0 ± 0.5 Aa
<b>Depth=20-30 cm</b>				
Site= Natural	13.0 ± 2.8 Ab	16.7 ± 2.3 Ab	-25.6 ± 0.4 Aa	-23.7 ± 0.6 Aab
Site= Stockpiled	20.0 ± 2.7 Ba	20.1 ± 2.1 Aa	-26.1 ± 0.4 Aa	-26.3 ± 0.5 Ba
<b>Depth=80-90 cm</b>				
Site= Natural	14.4 ± 2.8 Ab	16.4 ± 2.3 Ab	-23.4 ± 0.4 Ab	-23.3 ± 0.6 Ab
Site= Stockpiled	19.4 ± 2.7 Ba	21.2 ± 2.2 Aa	-26.4 ± 0.4 Ba	-25.3 ± 0.6 Ba

<sup>13</sup>C levels were less negative (more enriched) in both the 4 mm and 250 μm aggregate sizes in natural soils (overall mean = -25.1; overall mean = -24.1 respectively) than in stockpiled soils (overall mean = -26.3; overall mean = -25.9 respectively) for all sampled depths. However, these differences were only significant at the 80 to 90 cm depth for the 4 mm aggregate size (p=0.0004), and at the 20 to 30 cm and 80 to 90 cm depths for the 250 μm aggregate size (p=0.0037, p=0.0195 respectively) (Table 3.6). Isotopic concentrations did not change between depths within stockpiled soils for both the 4 mm and 250 μm aggregate sizes (p>0.750, p>0.250 respectively). However, a greater enrichment in <sup>13</sup>C was observed in natural sites at the 80 to 90 cm depth in the 4 mm aggregate size when compared to either the 0 to 10 cm (p<0.0001) or 20 to

30 cm ( $p=0.0003$ ) depths. Only the 0 to 10 cm and 80 to 90 cm depths were different in natural sites for the 250  $\mu\text{m}$  aggregate size ( $p=0.0211$ ) (Table 3.6).

### **3.5. DISCUSSION**

#### **3.5.1. Linkage between Structural Stability and Soil Carbon Quantity**

Proportions of 4 mm and 250  $\mu\text{m}$  water stable aggregates were often higher in natural sites than stockpiles, though these differences were not statistically significant. The similarity in aggregate proportions between the two site types is contrary to the original hypothesis of this study and to the findings of many other stockpile studies (Hunter and Currie, 1956; Abdul-Kareem and McRae, 1984; Wick et al., 2009b). Proportions of each aggregate size also varied with depths in natural sites, whereas no differences were observed in stockpiles, results that also support and refute, respectively, the original hypotheses of this project.

Despite the non-significant statistical difference, natural soils generally had a greater amount of 4 mm aggregates than stockpiles, likely due to the lack of a disturbance that would disrupt and break down soil structure. Stockpiled soils, having undergone such a disturbance, likely did experience a decline in macroaggregate proportions initially. These proportional declines were not noted here, likely due to the elevated levels of soil carbon found in the sampled stockpiles.

Stockpiles were found to have a greater amount of total carbon, aggregate associated carbon, and higher proportions of light fraction carbon than natural soils, results that refute the original hypotheses of this project and the finding of many other studies (Hunter and Currie, 1956; Abdul-Kareem and McRae, 1984; Visser et al., 1984).

Elevated LFC proportions in stockpiled soils would likely allow for consistent, uniform aggregation across all stockpile sampling depths, minimizing differences within the stockpile profile and eliminating natural-stockpile site differences in terms of aggregate structure. This is because macroaggregates represent the consolidation of individual microaggregates through the influence of organic binding agents, a component of SOM that is more recently deposited in the soil and/or less decomposed (Tisdall and Oades, 1982; Yamashita et al., 2006; Tan et al., 2007). Greater levels of LFC in stockpiles likely provided sufficient sources of the organic binding agents necessary to facilitate larger macroaggregate formation, leading to higher than expected macroaggregation proportions overall.

Higher levels of 250  $\mu\text{m}$  aggregates in natural soils likely also relate to the abundance and availability of LFC. While natural soils did not experience any structural disturbance, they exhibited much lower availabilities of total carbon and LFC than their stockpiled counterparts. This reduced the availability of organic binding agents to facilitate small macroaggregate consolidation throughout the natural soil profile. This effect would be especially pronounced in natural sites with an increase in depth, where a lack of new carbon inputs could first prevent the formation of larger macroaggregates, but also the formation of macroaggregates altogether if organic binding agents are sufficiently scarce (Lavkulich and Arocena, 2011; Smith et al., 2011).

Higher quantities of carbon were found in the 250  $\mu\text{m}$  sized aggregates than the 4 mm aggregates, supporting the hypotheses of this project and the theory proposed by others (Killham et al., 1993; Yamashita et al., 2006; Tan et al., 2007). This is likely indicative of multiple stabilization mechanisms acting in both soil types and aggregate size fractions (Six et al., 2002).



At the 250  $\mu\text{m}$  macroaggregate scale, primary stabilization mechanisms that could explain larger quantities of aggregate associated carbon would likely relate to the physical protection of SOM, through the size exclusion of microorganisms, due to the smaller intra-aggregate pore spaces present than in the 4 mm macroaggregates. This would represent a compartmentalization of microorganisms and SOM, decreasing the extent of decomposition by limiting SOM exposure to microbial enzymes (Killham et al., 1993; Cotrufo et al., 2013). At the same time, small pore spaces would limit the speed of oxygen diffusion into the intra-aggregate space, creating a low oxygen environment and reducing the rate of aerobic respiration by *in situ* micro-organisms even further (Sexstone et al., 1985). These mechanisms would, in turn, reduce both the number of microbes within the aggregate pore spaces and overall microbial activity, thereby stabilizing the soil carbon pool associated with these small macroaggregates in both soil types (Six et al., 2002).

The shielding mechanisms at the 4 mm aggregate scale would largely relate to the compartmentalization of SOM and microorganisms as well. The carbon that binds these large macroaggregates together, carbon that would typically be exposed to soil microbes on the aggregate surface, would become interred in the intra-aggregate space. Pore sizes in the 4 mm aggregates would permit a larger portion of the microbial community to enter the aggregate itself; however, spatial separation between the majority of the labile SOM and microbial decomposition enzymes would still serve as sufficient protection to prevent further decomposition (Yamashita et al., 2006; Tan et al., 2007).

Quantities of aggregate associated carbon and light fraction carbon proportions were not found to be different within stockpiled soils with increasing depth, refuting the original hypotheses of this project, whereas declines were observed within natural sites supporting the original hypotheses of this study. Alterations in the quantity of 4 mm and 250  $\mu\text{m}$  aggregate associated carbon, along

with LFC proportions between depths within natural and stockpiled sites is likely due to differences in organic matter integration at each respective depth, rather than differences in the extent of physical stabilization mechanisms discussed above. As stockpiles were homogenized at placement, soil organic matter would have been integrated at all depths within the stockpile, leading to similar levels of aggregate associated carbon and LFC proportions between sampling depths. Natural sites on the other hand, lacked a deep soil disturbance. Minimal inputs of new organic material to subsoil depths, largely the result of poor LFH-mineral soil integration, coupled with *in situ* microbial decomposition, is likely why declines in aggregate associated carbon quantities and LFC proportions were observed between surface and subsurface depths of the natural soils (Lavkulich and Arocena, 2011; Smith et al., 2011).

### **3.5.2. Soil Carbon Dynamics**

The above section highlights the importance of soil carbon in influencing soil structure, and the reciprocal role soil structure has on influencing aggregate associated carbon quantities. This influence can be scaled up, in an effort to look at the whole soil and *in situ* soil carbon dynamics that are taking place. In this study, soil carbon dynamics were directly measured through basal respiration rates. Stockpiled soils had lower basal respiration rates than natural soils across all sampled depths, findings which are contrary to the original hypothesis of this project but have been observed in other stockpile studies (Visser et al., 1984). Respiration rates also did not differ within natural and stockpiled soils between sampling depths, results that are contrary again to the original hypotheses of this study.

Lower levels of microbial respiration in stockpiled soils when compared to natural sites, as well as similar levels of respiration between depths within natural and stockpiled sites, could be the

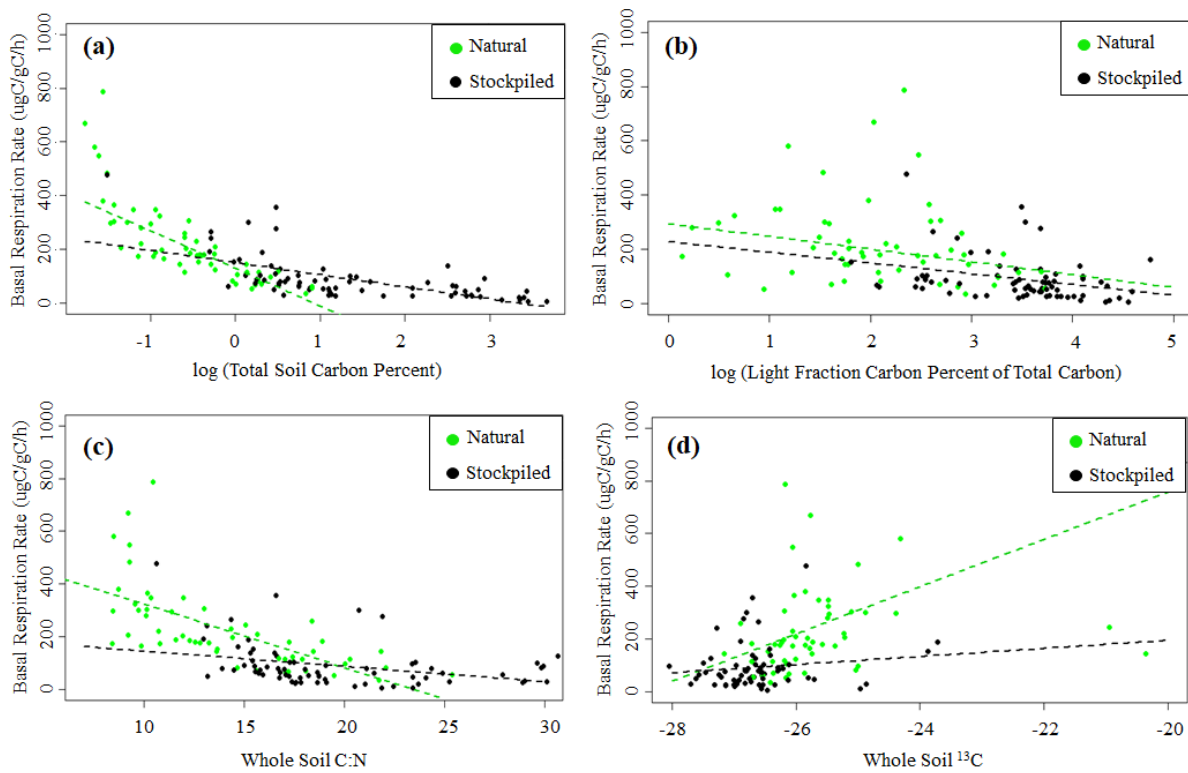
result of a number of factors. Many previous studies reported a marked drop in stockpile carbon levels compared to surrounding natural soils across all depths, resulting from the dilution of topsoil, often higher in organic material, with underlying subsoil during salvaging and stockpiling operations (Visser et al., 1984; Wick et al., 2009b). Upland natural soils in the boreal forest also exhibit “dilutions” of soil carbon with increasing depth, due to limited carbon inputs to subsurface layers (Lavkulich and Arocena, 2011; Smith et al., 2011). Having abundant quantities of available carbon is important for soil microorganisms, which need carbon not only for respiratory activities to produce energy, but also for cellular growth and development (Gougoulis et al., 2014). As such, the dilution of available carbon could limit the ability for microorganisms to meet their metabolic needs, subsequently reducing their respiratory activity.

The results of this study highlight three interesting points tied to this concept: (1) to maintain similar respiration rates at all depths within the natural sites, microbial communities likely decompose more plant-based materials at the soil surface and more microbial related products in deeper subsoil layers (2) elevated, uniform levels of LFC across stockpile sampling depths likely account for the similar respiratory levels observed, due to an abundance of carbon sources needed to sustain a residual microbial community, and (3) ultimately, carbon dilution is likely not the cause of low respiration rates in stockpiles when compared to natural sites. This would be because, again, stockpiles contained much higher levels of total carbon than natural sites, likely sourced from the integrated LFH material.

An alternative explanation to low stockpile respiration rates could be declines in the overall size or function of *in situ* microbial populations. While not measured in this study, microbial biomass has been found to be lower in stockpiled soils, especially at the soil surface where weather extremes can further stress *in situ* microorganisms (Visser et al., 1984; Harris et al., 1989;

Johnson et al., 1991). Building on this concept, differences in soil carbon quantity and lability between site types could further stress microbial communities, potentially hindering growth and development.

Work has been done on using measures of soil carbon quantity and lability as a proxy for soil respiration and carbon turnover by comparing previously reclaimed lands with natural reference sites (Ingram et al., 2005). As such, a similar comparative approach was applied here, for the stockpiled and natural reference sites, using a number of measures pertaining to SOM and carbon quantity and lability (Figure 3.6).



**Figure 3.6: Scatter plot panel showing the relationship between basal respiration ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ), total soil carbon (%), light fraction carbon proportions, whole soil C:N, and whole soil  $^{13}\text{C}$  isotopic concentrations for both natural and stockpiled soils.**

**Table 3.7: Regression equations, correlation coefficients, and p-values for each scatterplot panel in Figure 3.6. Overall regression trends were determined on all data points regardless of site type (natural or stockpiled) and individual regressions were also determined for natural and stockpiled sites separately.**

Panel (a)- log (Total Carbon)		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
196.96-77.54*x	0.12	0.00
<b>Natural</b>		
Equation	R <sup>2</sup>	P
129.20-126.00*x	0.49	0.00
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
150.38-45.31*x	0.39	0.00

Panel (b)- log (Light Fraction Carbon)		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
303.99-51.72*x	0.10	0.00
<b>Natural</b>		
Equation	R <sup>2</sup>	P
294.46-24.02*x	0.12	0.01
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
227.80-39.40*x	0.08	0.01

Panel (c)- Whole Soil C:N		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
414.70-15.00*x	0.18	0.00
<b>Natural</b>		
Equation	R <sup>2</sup>	P
564.19-24.02*x	0.42	0.00
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
202.80-5.78*x	0.09	0.01

Panel (d)- Whole Soil <sup>13</sup> C		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
493.19+12.76*x	0.01	0.13
<b>Natural</b>		
Equation	R <sup>2</sup>	P
2550.33+89.65*x	0.10	0.02
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
502.68+15.38*x	0.01	0.29

Stockpiled soils consistently exhibited higher soil carbon levels and greater proportions of LFC than natural sites; stockpiled soils also had higher whole soil C:N ratios and were more depleted (more negative) in <sup>13</sup>C than natural sites (Figure 3.6; Table 3.4; Table 3.5). All of these findings largely contradict the original hypotheses of the project and the findings of many alternative studies conducted on reclaimed and natural soils (Williamson and Johnson, 1990; Schwenke et al., 2000b; Ingram et al., 2005; Banning et al., 2008; Wick et al., 2009a; Turcotte et al., 2009; Wick et al., 2009b). Within natural sites, the declines observed for whole soil C:N ratios with increasing depth, accompanied by an enrichment (less negative) in <sup>13</sup>C, support the original

hypotheses of this project. However, the lack of change in these same variables within stockpiles soils refutes the original hypotheses of this project.

The relationship between the above mentioned carbon parameters and basal respiration rates demonstrate some important points. A negative exponential relationship (subsequently log transformed) was observed to exist between basal respiration, on a per gram of carbon basis, total soil carbon, and light fraction carbon proportions for both site types (Figure 3.6; Table 3.7). This is surprising, as carbon is an important component in microbial growth and development as discussed above. As well, LFC sources are more readily decomposed by microorganisms to meet their carbon requirements due to its less chemically complex nature (Janzen et al., 1992; Bremer et al., 1994).

This indicates that perhaps there is something that is preventing microorganisms from actively utilizing the carbon sources available to them in stockpiled soils. This could relate to a limited availability of soil nitrogen, typical for many terrestrial upland environments (Gregorich and Janzen, 1996; LeBauer and Treseder, 2008). A negative linear relationship was found between respiration rates and soil C:N ratios, indicating that a greater abundance of carbon, or proportionally less nitrogen, corresponds with lower microbial activity and respiration (Figure 3.6; Table 3.7).

Incorporating considerations of nitrogen availability seems to provide a more representative idea of the *in situ* microbial metabolic requirements. Microorganisms need to maintain a very low C:N ratio to survive; as such, preferential decomposition patterns are created where microbes break down plant residues that have a lower C:N (Enríquez et al., 1993; Gregorich and Janzen, 1996; Fernandez et al., 2003; Cotrufo et al., 2013). As a result of this preferential decomposition

process, more carbon is lost from the soil, resulting in an accumulation of nitrogen within the residual SOM pool mainly immobilized within the microbial community a trend that is being observed in natural soils.

The continuous breakdown of SOM to acquire nitrogen can be demonstrated by looking at the final parameter of soil carbon quality in Figure 3.6,  $^{13}\text{C}$  isotopic concentrations. A positive, linear relationship between basal respiration rates and  $^{13}\text{C}$  was found for both soil types, though this relationship was only significant for the natural soils (Figure 3.6; Table 3.7). Nonetheless, this would suggest that an enrichment in  $^{13}\text{C}$  corresponds with greater microbial activity. This is because  $^{13}\text{C}$  becomes increasingly concentrated in microbial communities and the bulk SOM with increasing stages of decomposition through respiratory fractionation mechanisms (Quideau et al., 2003; Fernandez et al., 2003; Wynn et al., 2006; Garten Jr. et al., 2007; Sulzman, 2007).

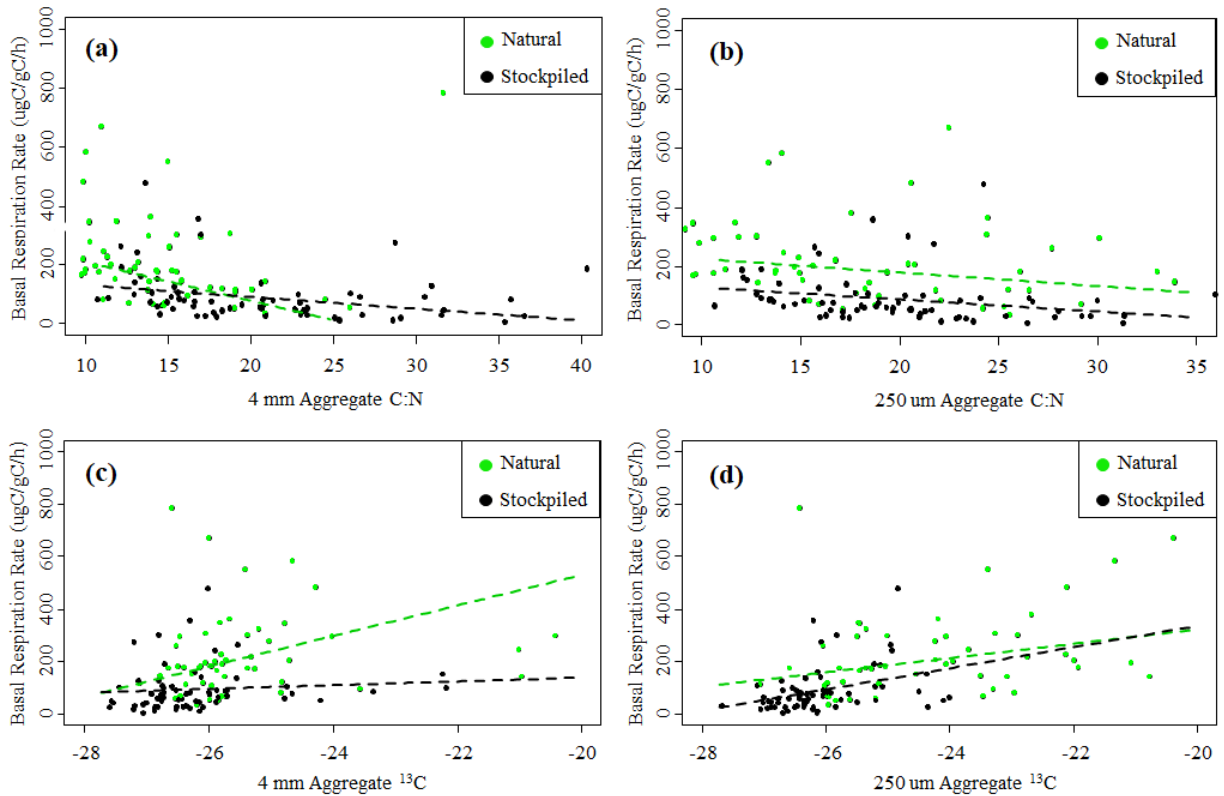
Higher concentrations of heavy isotopes in natural soils, especially with increasing depth, indicate that a greater level of decomposition has occurred and is likely still occurring.

Preferential decomposition of low C:N plant materials, found largely within the LFC pool, would result in an increased concentration of nitrogen immobilized within the microbial community, subsequently lowering whole soil C:N ratios and elevating the C:N ratio of the residual LFC pool. Greater nitrogen accumulation, likewise, means greater microbial community growth, elevated metabolic activity, higher respiration rates, and increased carbon turnover rates when compared to stockpiled soils, explaining how natural soils have lower carbon levels with correspondingly high respiratory activity on a per gram of carbon basis.

Minimal differences in these same parameters between sampling depths within stockpiled soils likely links again to the abundant distribution of LFC throughout the entire stockpile profile, but

also a smaller, less active microbial community previously impacted by the soil salvaging and storage disturbances.

While the above results are for the whole soil, it is interesting to note that these trends are also being demonstrated at the individual aggregate level (Figure 3.7).



**Figure 3.7: Scatter plot panel showing the relationship between basal respiration ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ), 4 mm aggregate, and 250  $\mu\text{m}$  aggregate C:N and  $^{13}\text{C}$  isotopic concentrations for both natural and stockpiled soils.**



**Table 3.8: Regression equations, correlation coefficients, and p-values for each scatterplot panel in Figure 3.8. Overall regression trends were determined on all data points regardless of site type (natural or stockpiled) and individual regressions were also determined for natural and stockpiled sites separately.**

<b>Panel (a)- 4 mm C:N</b>		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
273.24-6.49*x	0.05	0.01
<b>Natural</b>		
Equation	R <sup>2</sup>	P
337.46-12.99*x	0.23	0.00
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
169.52-3.96*x	0.08	0.01

<b>Panel (b)- 250 um C:N</b>		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
206.75-2.43*x	0.00	0.37
<b>Natural</b>		
Equation	R <sup>2</sup>	P
269.08-4.59*x	0.08	0.03
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
166.28-4.13*x	0.09	0.01

<b>Panel (c)- 4 mm <sup>13</sup>C</b>		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
252.33+3.68*x	0.00	0.62
<b>Natural</b>		
Equation	R <sup>2</sup>	P
1690.96+58.09*x	0.13	0.01
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
271.65+6.84*x	0.00	0.49

<b>Panel (d)- 250 um <sup>13</sup>C</b>		
<b>Overall</b>		
Equation	R <sup>2</sup>	P
602.43+17.63*x	0.05	0.01
<b>Natural</b>		
Equation	R <sup>2</sup>	P
861.84+27.08*x	0.12	0.01
<b>Stockpiled</b>		
Equation	R <sup>2</sup>	P
1141.10+40.30*x	0.12	0.00

Negative, linear relationships were found to exist between basal respiration and the C:N ratio for both aggregate sizes (Figure 3.7; Table 3.8). Positive, linear relationships were found to exist between respiration and <sup>13</sup>C concentrations, though this relationship was not significant for stockpiled 4 mm aggregates (Figure 3.7; Table 3.8). Therefore, even at the single aggregate-size scale, the importance of the *in situ* microbial community becomes evident; natural sites, which would likely host a larger, more active, and persistent microbial community than stockpiles,

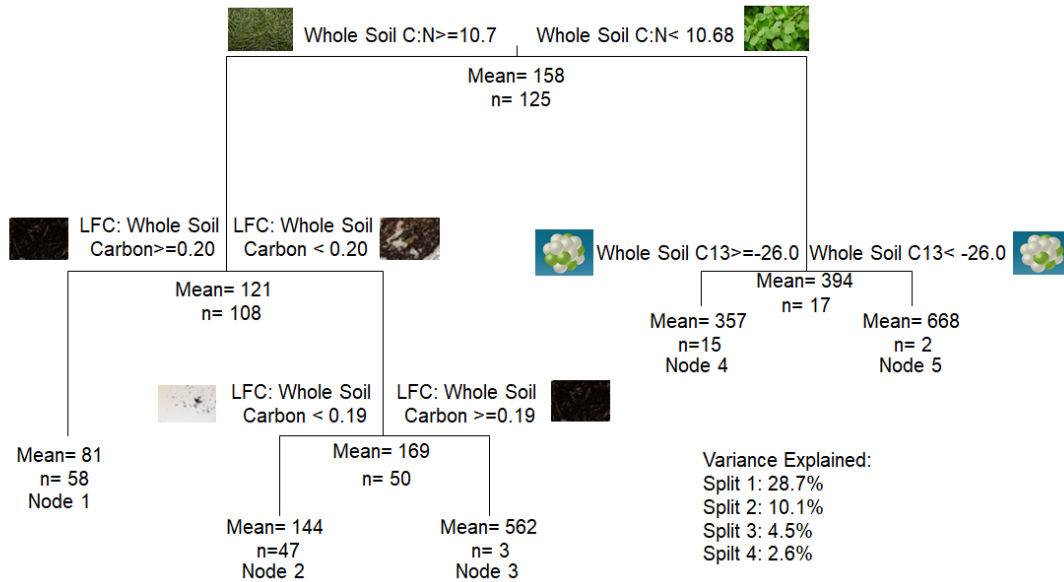
would be capable of breaking down a larger amount of organic material not physically shielded within soil aggregates (Six et al., 2002).

It is also important to note that while aggregate C:N ratios declined and  $^{13}\text{C}$  concentrations increased with increasing depth within the natural sites, stockpiled soils did not show the same trend. Natural sites likely experienced these changes due to the limited input of new litter material and increased decomposition stage at depth. Stockpiled soils, heavily and uniformly disturbed during soil salvage, would have a much smaller and less active microbial community that, even at the aggregate level and regardless of sampling depth, would not be able to breakdown *in situ* carbon resources to the same extent as was noted in the natural soils.

The similarities observed between the aggregate size classes and whole soil in relation to soil carbon quantity, quality, and respiration rates highlights the importance of soil structure and availability of nutrients on driving soil carbon turnover for the greater soil body. It also seems to suggest that there may be a disproportionate influence of these soil carbon quality parameters on basal respiration rates, which is shown in Figure 3.8.

It can be seen that the whole soil C:N ratio has the largest influence of overall soil respiration rates. Natural soils likely have a larger, more consistently functional microbial community than stockpiled soils, communities which would not have been adversely impacted by soil salvage and storage operations (Visser et al., 1984; Harris et al., 1989; Johnson et al., 1991). The increased proportion of nitrogen fixed in microbial communities and their products would result in lower whole soil C:N ratios and allow for a more maintained SOM decomposition process in natural soils, indicated by the  $^{13}\text{C}$  isotopic concentrations. A larger microbial community coupled with less decomposed SOM (more negative  $^{13}\text{C}$  values) would then produce the highest basal

respiration rates (Table 3.13, Figure 3.8). The more the SOM becomes decomposed (less negative  $^{13}\text{C}$  values), the more respiration declines (Table 3.12, Figure 3.8).



**Figure 3.8: Regression tree showing the relationship between basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ), whole soil C:N, light fraction carbon proportions, and whole soil  $^{13}\text{C}$  isotopic concentrations. Mean values represent the average basal respiration rate for each split and  $n$  is the number of samples that fall into each split category. Variance explained by each split is also shown. Node values correspond to Tables 3.9-3.13.**

The regression tree in Figure 3.8 also establishes an optimal threshold for LFC. In soils characterized by a higher C:N, high levels of labile carbon remain insufficient to properly stimulate microbial respiration. This could be the result of a lack of other essential nutrients such as nitrogen needed for growth, or, perhaps more likely, due to a decline in overall soil microbial utilization of the carbon sources available in the soil due to smaller community sizes (Table 3.9). However, when LFC proportions are too low, SOM pools may be too chemically complex to decompose, thereby limiting overall rapid microbial activity, an issue present in both natural and stockpiled soils (Table 3.10; Figure 3.8). The optimal threshold established in Figure 3.8, and the subsequently high respiration rate observed, seems to suggest an adequate balance between labile

carbon quantities, an abundance of other nutrients needed for growth, and a microbial community size capable of utilizing the present resources, and stresses the importance of creating and maintaining this balance for overall soil function (Table 3.11; Figure 3.8).

**Table 3.9: Frequency of occurrence by site type and depth for Node 1.**

Average Respiration: 81 ugC gC <sup>-1</sup> h <sup>-1</sup>		
Site Type	Depth (cm)	n
Natural	0-10	5
Natural	80-90	1
Stockpiled	0-10	19
Stockpiled	20-30	19
Stockpiled	80-90	15

**Table 3.10: Frequency of occurrence by site type and depth for Node 2.**

Average Respiration: 144 ugC gC <sup>-1</sup> h <sup>-1</sup>		
Site Type	Depth (cm)	n
Natural	0-10	10
Natural	20-30	11
Natural	80-90	10
Stockpiled	0-10	4
Stockpiled	20-30	5
Stockpiled	80-90	7

**Table 3.11: Frequency of occurrence by site type and depth for Node 3.**

Average Respiration: 562 ugC gC <sup>-1</sup> h <sup>-1</sup>		
Site Type	Depth (cm)	n
Natural	20-30	1
Stockpiled	0-10	1
Stockpiled	80-90	1

**Table 3.12: Frequency of occurrence by site type and depth for Node 4.**

Average Respiration: 357 ugC gC <sup>-1</sup> h <sup>-1</sup>		
Site Type	Depth (cm)	n
Natural	0-10	1
Natural	20-30	6
Natural	80-90	7
Stockpiled	80-90	1

**Table 3.13: Frequency of occurrence by site type and depth for Node 5.**

Average Respiration: 668 ugC gC <sup>-1</sup> h <sup>-1</sup>		
Site Type	Depth (cm)	n
Natural	0-10	2

### 3.6. CONCLUSIONS

Stockpiled soils differed from natural soils when looking at aggregate-associated and whole soil carbon quantity and lability criteria, as well basal respiration rates. However, stockpiled and natural soils remained similar in terms of their aggregate size distributions. These results are largely contrary to much of the current literature base and also reflect that stockpiled soils, regardless of depth, have not suffered a decline in chemical quality, pertaining in this case to

organic carbon quantities and lability, as a result of soil salvage and storage. In fact, these stockpiled soils actually have an improved chemical quality over their natural soil counterparts, meaning that there may be little concern regarding the suitability of stockpiled soils for use in reclamation based on chemical quality alone.

It is critical to note, however, that the biological quality of stockpiled material is lower than their natural counterparts, relating to the lower basal respiration rates that were observed in this study. This is an important observation to make, as it shows that despite improvements in some aspects of soil quality, stockpiled soils are also experiencing declines in others. Lower levels of biological activity could have some important consequences when assessing stockpiled soils for potential substrates to use in reclamation; lower amounts of carbon turnover could have cascading effects not just on carbon mineralization, but the release of other essential nutrients that could influence the success of native plant communities post-reclamation.

## IV. SYNTHESIS

### 4.1. RESEARCH SUMMARY

The overall objective of this project was to assess the impacts of stockpiling on salvaged topsoil, in Alberta's boreal forest, associated with oil and gas mining and *in situ* operations. Stockpiling effects were assessed over a variety of depths first with regards to alterations in a number of basic soil properties and general soil physical quality, and secondly, on changes to soil aggregation, carbon distributions, and carbon quality at aggregate and whole soil scales.

These main objectives were explored in two data chapters, each with specific goals to address the broader objectives above. The first data chapter explored how stockpiling impacted soil texture, bulk density, and total soil carbon, linking these changes to those noted for a number of water retention curve parameters such as field capacity, plant available water contents, air capacities, and macroporosity. Together, these two sets of variables were compared to an index of soil physical quality, the S index, to demonstrate their ability to act as proxies for soil physical quality. A third set of soil physical quality indicators, air and water retention energies, were then presented as an alternative to using the S index.

The second data chapter addressed a specific component of soil physical quality, soil structure in the form of soil aggregates, and assessed how stockpiling shifted aggregate size distributions and changed aggregate carbon and whole soil light fraction carbon quantities. Basal respiration rates were measured to estimate soil carbon turnover rates in both natural and stockpiled soils over different depths. In turn, additional measures such as total soil carbon, light fraction carbon proportions, C:N, and  $^{13}\text{C}$  isotopic concentrations, at both the aggregate- and whole-soil-scale

where applicable, were compared to basal respiration rates to assess whether these measures could serve as proxy variables for carbon dynamics in soil.

#### **4.2. LINKING BASIC SOIL PROPERTIES AND SOIL PHYSICAL QUALITY TO SOIL CARBON QUANTITIES AND QUALITY**

Stockpiled soils consistently showed lower clay contents and bulk densities, and higher levels of total soil carbon, than natural soils. These results were associated with larger field capacity values, greater plant available water contents, and higher macroporosities and air capacities in stockpiled soils. Stockpiled soils, as a result, had higher soil physical quality than their natural soil counterparts when assessed using the S index (Dexter, 2004; Reynolds et al., 2009). In contrast, when air and water retention energy parameters were used to assess soil physical quality, natural sites potentially had more favorable soil physical quality than stockpiled soils, though this was inconclusive (Armando and Wendroth, 2016; Bacher et al., 2019).

Further surprising results were found when the aggregate size distributions were measured; no differences were found, across any of the sampling depths, between natural and stockpiled soils. Aggregation of stockpiled material following soil salvage and storage disturbances was likely due to the influence of soil carbon, more specifically light fraction carbon, which was in a greater abundance in stockpiled soils than in natural soils (Yamashita et al., 2006; Tan et al., 2007). The lack of soil disturbance likely also explains the higher basal respiration rates measured in natural soils. While having less overall soil carbon and lower proportions of light fraction carbon that was of poorer quality, natural soils still had much lower C:N and greater proportions of  $^{13}\text{C}$  than stockpiles did. Advanced levels of decomposition, indicated by higher  $^{13}\text{C}$  levels, could denote greater amounts of nitrogen mineralization, with subsequently higher levels of nitrogen being immobilized by the microbial population. This would lower whole soil C:N while

simultaneously creating higher respiration values due to a growing, more active microbial population. Stockpiled soils, in contrast, would likely have had a microbial community biomass much smaller or a species composition different from that present at the natural sites as a result of disturbance, reducing their overall capacity to utilize abundant *in situ* carbon sources and subsequently lowering respiration rates.

When looking at the differences between depths within natural and stockpiled profiles separately, it was interesting to note that for all depth comparisons made, no differences were observed in basic soil properties, water retention parameters, S index values, air and retention energies, aggregate size proportions, carbon quantities, and carbon lability, with the exception of total soil carbon, for stockpiled soils. This reinforces the homogenization effects of soil salvage, transport, and storage and the general lack of internal pedogenic processes in stockpiled material due to recent disturbance. On the contrary, differences in texture, bulk densities, water retention parameters, and aggregate size proportions between depths in natural soils highlights the influence of pedogenic processes such as clay eluviation on these natural soil profiles.

Differences in aggregate and whole soil carbon quantities and lability observed with depth denotes the lack of LFH integration into the mineral profile, with differences in total carbon quantities in particular likely having a larger and more cascading influence on aggregation, soil physical quality, and microbial activity than other measured parameters such as texture.

These results ultimately indicate that while largely looked at in two separate data chapters, it was not possible to fully separate soil physical properties and carbon characteristics from each other; in soil, these two parameters are highly linked, and so feedbacks between them should be considered. Lower levels of soil respiration and subsequent carbon turnover would have facilitated the retention of greater proportions of soil carbon throughout the stockpile profile.



Higher levels of soil carbon, in turn, would have then contributed to higher than expected quantities of macroaggregates in stockpiles following disturbance and an increase in the extent to which physical carbon stabilization could occur. When considered alongside diluted levels of fine textured materials, lower bulk densities and improved soil porosity observed in stockpiles would be expected. Improvements in macroaggregation would subsequently explain improvements to macroporosity and air capacities, increased water retention, and improved overall soil physical quality for stockpiled materials, creating more suitable growing conditions for vegetation.

### **4.3. RECLAMATION IMPLICATIONS**

When all of the results of this project are taken into consideration, it becomes apparent that they do in fact differ greatly from the majority of the information that is present in the current literature base. This was a conclusion mentioned at the end of each data chapter and ultimately is an important finding in the context of reclamation practice; it demonstrates the importance of assessing soil quality for the soils that have been impacted by salvage and storage and will be used in the reclamation of disturbed sites. Variation in the results of this project from the current literature base likely is due to the fact that the original natural soils in Alberta's boreal forest, from which the evaluated stockpiles were created from, are unique compared to those present in other regions of the world where stockpile quality assessments have been done. Differences between these soils would be the result of different soil forming factors acting on and pedogenic processes occurring within the soil profile, with these two aspects dictating how susceptible a particular soil is to high-intensity disturbance. Soils more susceptible to these kinds of disturbances may then create unforeseen challenges to reclamation success.

Looking more specifically at the project results as a whole, not only is it important to assess soil quality for stockpiled soils intended for use, but to ensure that all components are being assessed, the physical, chemical, and biological aspects of soils, rather than one or two of these components in isolation of the others. From a reclamation perspective, this would allow operators and reclamation practitioners to get the most comprehensive idea on the quality of the soils they will rely on to meet reclamation requirements and create equivalent land capability. For example, looking at basic soil properties, the S-index, and a number of capacity based measurements, it is apparent that stockpiled soils have actually undergone improvements in SPQ, regardless of the depth being looked at. With similar proportions of macroaggregates compared to natural soils, it is likely that these stockpiled soils are actually of little concern regarding their physical suitability for use in reclamation. Broadening the scope of factors being considered, taking into account soil carbon quantities and lability across all sampling depths, associated with chemical quality in this study, similar conclusions can be drawn; compared to natural soils, stockpiles have an improved chemical quality which would not likely present significant barriers to reclamation success.

Limiting assessments to these two aspects of soil quality could ultimately lead to management decisions that are not wholly representative of actual soil quality conditions. In this study, stockpile quality as a whole is being constrained by the biological quality of the soil. Lower respiration rates observed across all stockpile sampling depths, when compared to natural soils, indicate that based on the available soil carbon pool, stockpile microbial communities are not utilizing the available carbon substrates to the fullest extent possible. This would indicate that smaller proportions of the soil carbon pool are being mineralized, and it is this mineralization process that not only provides nutrients to support *in situ* microbial populations, but also re-

establishing native plant communities. Native species have adapted to specific nutrient cycling pathways; lower levels of nutrient mineralization may not be conducive to supporting the establishment and survival of many native overstory and understory vegetation species. This in turn, could create challenges in meeting equivalent land capability requirements and postponing reclamation certification. These findings therefore stress the importance of using a holistic approach in the assessment of stockpile soil quality

#### **4.4. PROJECT LIMITATIONS**

The ability to truly assess stockpiling impacts on soil physical properties and *in situ* carbon characteristics was constrained by some project limitations. Soil sampling depths were based on using the mineral soil surface as the point of reference. While not present on stockpiles, natural sites of the area were characterized by a thin overlaying LFH layer. This layer was excluded from the natural site sampling design to maintain consistency. This would have artificially reduced carbon quantities in natural soils on a depth-by-depth basis, due to minimal integration of LFH material into the mineral profiles of natural soils. However, this exclusion was assumed to have a minimal impact, as stockpiling would have led to a dilution of the thin LFH layer in large volumes of salvaged mineral soils.

One of the primary criteria used for stockpile selection was whether or not it was classified as topsoil. While all stockpiles in this project had that classification, it became apparent that some parts of the stockpiles were more of a topsoil-peat transitional material than straight mineral topsoil. This was noticed only after the soil pits were dug and so, due to a limited availability of sites, time constraints on equipment, and the inconsistency of depth in which transitional soils were identified, these sites were still sampled for this project. This would have increased total

soil carbon values in stockpiled materials compared to natural soils, with potential implications observed through lower bulk densities, higher capacity-based measurements, greater extents of macroaggregation, and increased basal respiration values. .

In an effort to address the variability in topsoil composition for each stockpile, each of the sampled stockpiles had three soil pits dug on them, with the same types of samples being collected from each of the different sampling depths. While this did seem to help address the variability to some extent, their proximity to each other was assumed to have created spatial correlation between the samples. As such, the samples taken from the three pits at each respective depth were viewed as pseudo-replicates, reducing the number of statistical sampling points on each stockpile at each depth to a single value. This, in turn, reduced the number of comparable data points available for statistical analysis.

It was also challenging to find a suitable number of similarly aged stockpiles between the two sampling sites, especially at CNRL Horizon. As such, the sampled stockpiles ended up being a range of different ages, some of which differed by more than 10 years. It is possible that some of the trends that were observed in this study are due to the influence of time on the recovery of stockpile properties, rather than revolving around current *in situ* carbon pools as proposed in this project. However, this time component can be partially addressed with the project data at hand, as will be discussed below.

#### **4.5. FUTURE RESEARCH**

In this study, stockpiles of various ages were sampled and while large age ranges of stockpiles were available, it was not possible to replicate each stockpile age sufficiently to allow for quantitative statistical measures to be carried out on the data. Assessing the impact of stockpile

age on topsoil quality is a critical component in trying to determine the suitability of stockpiled topsoil for use in reclamation. For this study specifically, the influence of time on stockpile properties may be a confounding factor, reducing the level of certainty that the changes observed here can be attributed to soil organic matter pools, more specifically light fraction carbon, and its role in soil structure stabilization.

While carrying out full quantitative statistical measures, as seen in the two data chapters of this project, was not able to be done to address the time component, it was still possible to create exploratory graphics and figures to see, qualitatively, whether it appears that time is playing a major role in influencing the following key soil parameters: bulk density, total soil carbon, S, whole soil light fraction carbon proportions, whole soil C:N, whole soil  $^{13}\text{C}$ , 4 mm and 250  $\mu\text{m}$  aggregate proportions, 4 mm and 250  $\mu\text{m}$  C:N and  $^{13}\text{C}$ , and basal respiration rates. These variables were selected as they were identified as being the most likely to change over time. The exploratory figures can be seen in Appendix A and each age is divided into surface depths (0 to 10 cm) and subsurface depths (20 to 30 cm and 80 to 90 cm combined).

These exploratory figures highlight two interesting, preliminary findings. First off, there does not appear to be any strong influence of time on any of the parameters, seeming to suggest that the confounding influence of time is minimal in relation to the current project conclusions. For some parameters, such as total soil carbon, S, whole soil C:N, and light fraction proportions, there may be a weak negative relationship with time (Figure A.2-5). In contrast, for basal respiration rates, there may be a weak positive relationship (Figure A.10). However, the variability present in these five cases makes it challenging to confidently say that these trends exist at all. Secondly, the oldest stockpiles, particularly the 28 year old piles, seem to be the most similar to the natural soils for the some of the parameters graphed in Appendix A (Figure A.1-6; A.9) However, this

trend is not consistent and some younger stockpiles appear very similar to both the oldest stockpiles and natural sites, showing again that increasing age does not necessarily correspond with more natural soil conditions.

While appearing qualitatively insignificant, being able to confirm the statistical significance of these trends, and relating them perhaps to any biological significance that may arise, would provide additional insight on how time can change the suitability of stockpiled topsoil for use in reclamation. As such, it may be beneficial to incorporate time assessments into a cradle-to-grave approach, meaning that soils would be sampled in an area slotted for salvage immediately before it was disturbed, as soon as the soil was placed into stockpiles, in sequential years following storage, and perhaps even when the soils are placed in reclamation. This type of approach would allow one to assess how all these different stages of soil handling and disturbance are changing the physical, chemical, and biological components of soil quality, while also addressing a number of limitations that were present in this research project.

As salvage areas are typically quite large, it would be possible to establish numerous replicate stockpiles that were salvaged at the same time, allowing for quantitative statistical analyses to be carried out based on stockpile age. These replicated stockpiles would be created from the same salvaged material, allowing for more uniformity in substrate composition, and in the same way, maintaining consistency, as best as operationally possible, in how stockpiled soils are salvaged, loaded, transported, and stored. This type of study would provide additional, complimentary insights to the results of this project, further developing the literature base and informing industry practitioners on how to best handle and assess stockpiled topsoil to improve the chances of overall reclamation success.

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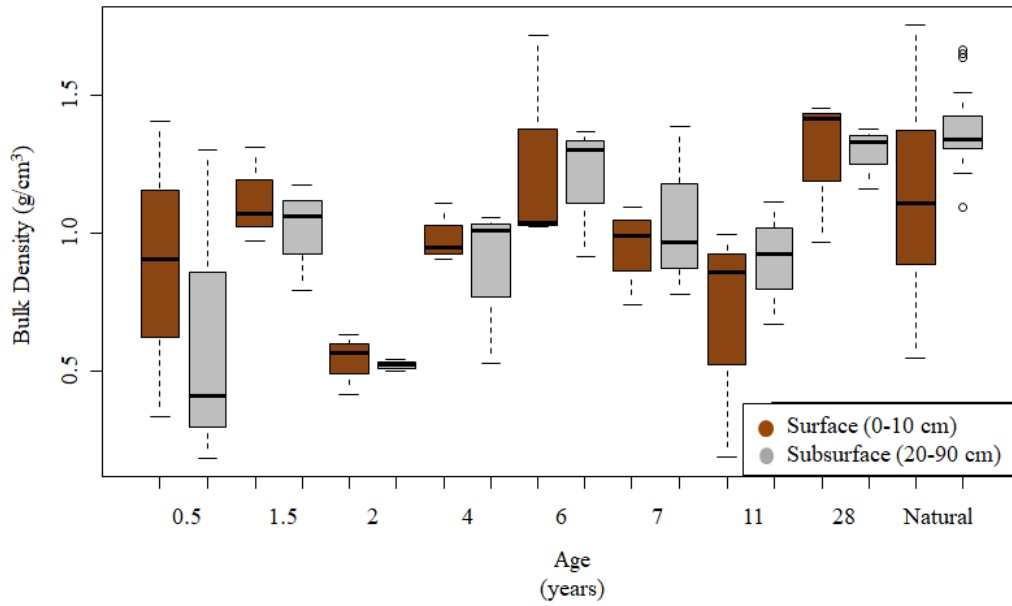
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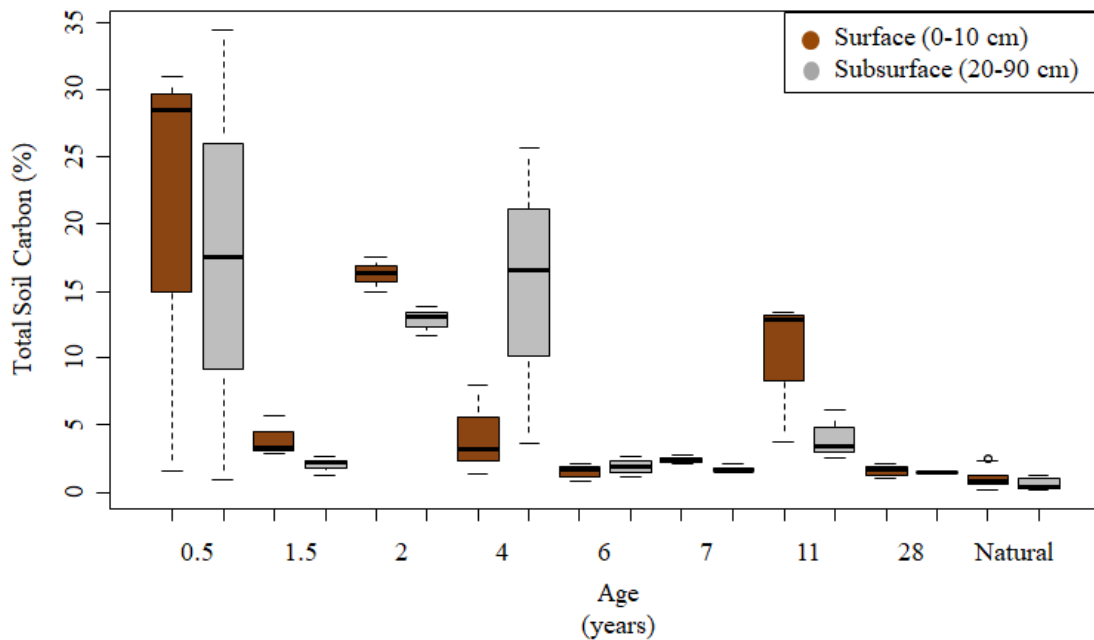
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## APPENDIX A

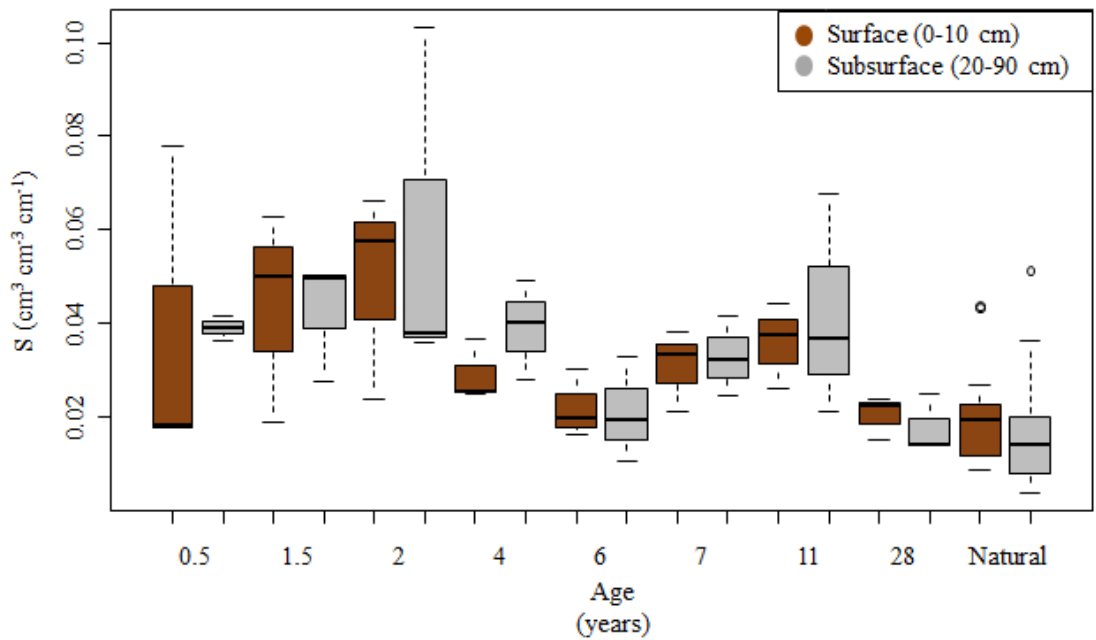


**Figure A.1: Boxplot showing bulk density ( $\text{g cm}^{-3}$ ) values for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.**

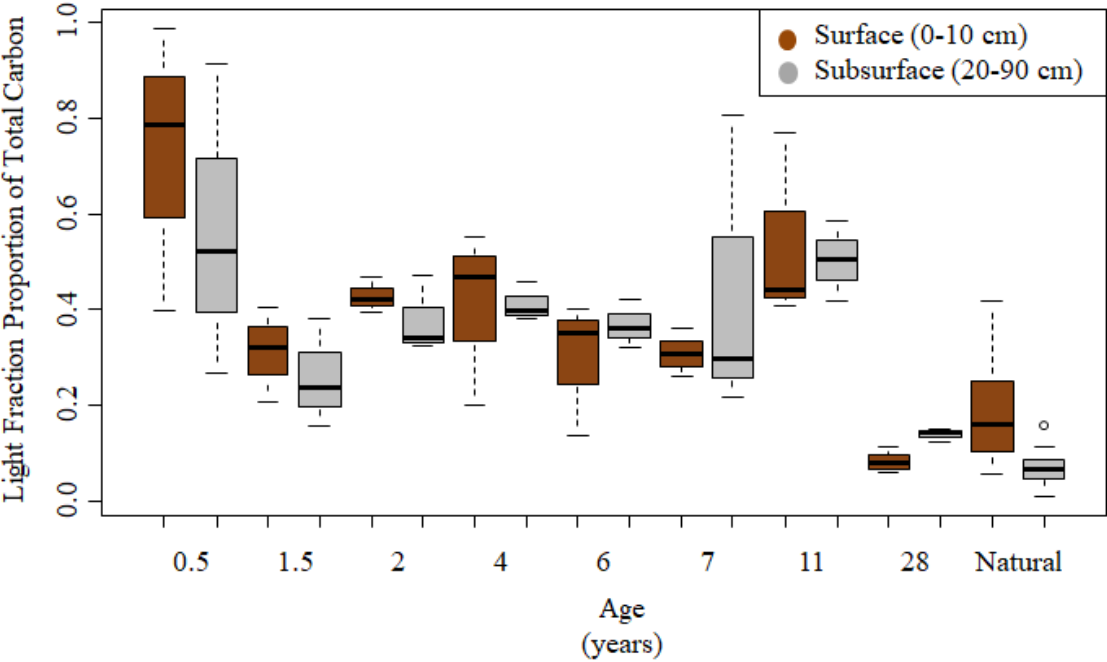


**Figure A.2: Boxplot showing total soil carbon values (%) for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.**

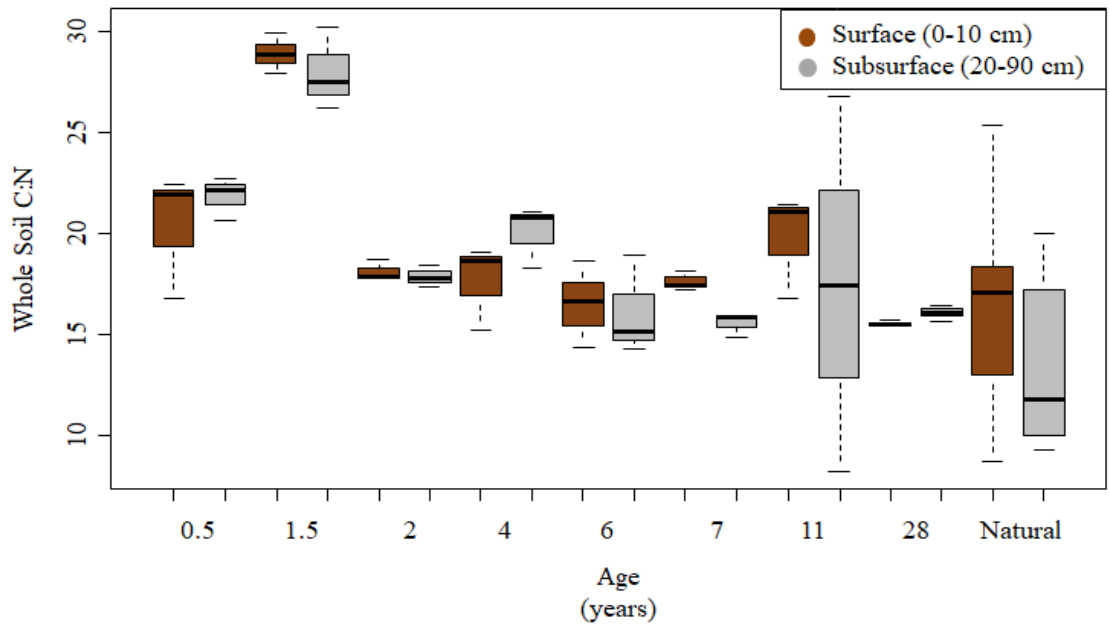




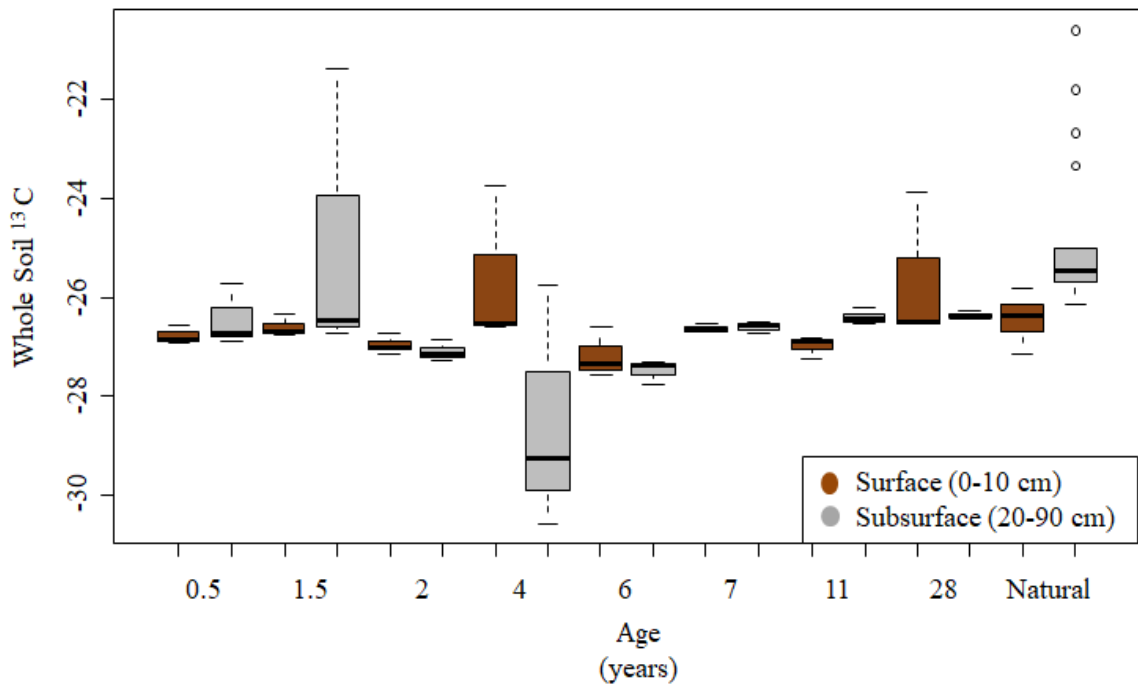
**Figure A.3:** Boxplot showing S index values ( $\text{cm}^3 \text{cm}^{-3} \text{cm}^{-1}$ ) for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.



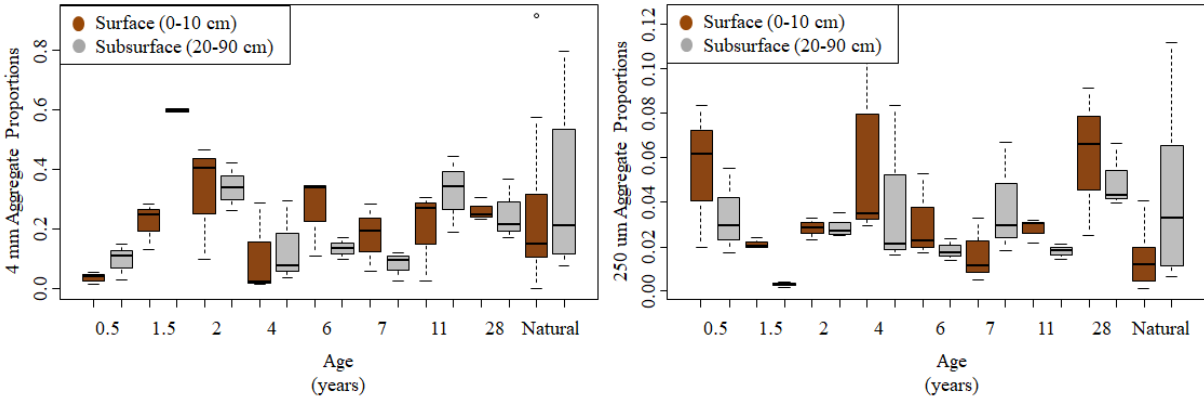
**Figure A.4:** Boxplot showing light fraction proportions values for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.



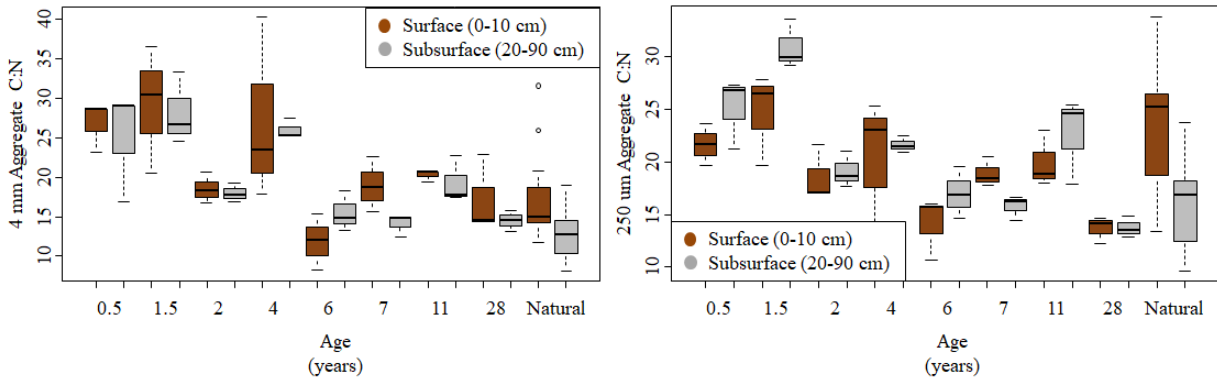
**Figure A.5:** Boxplot showing whole soil C:N values for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.



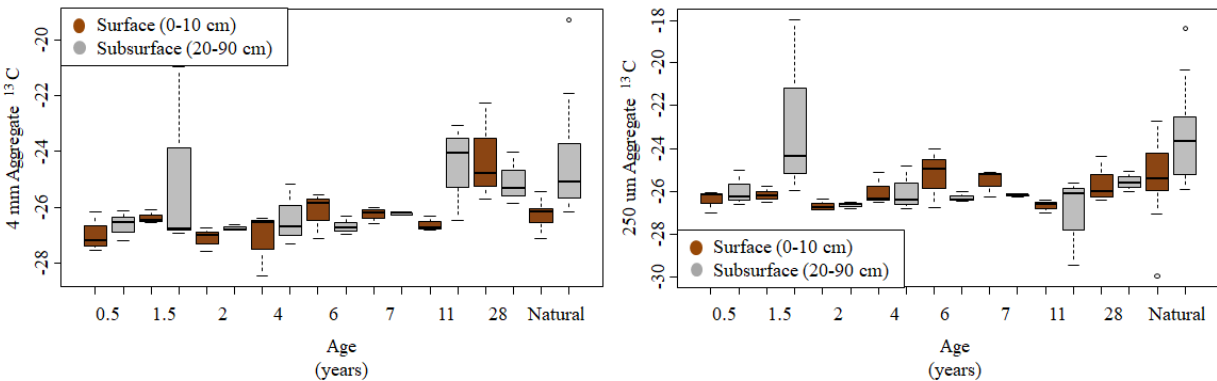
**Figure A.6:** Boxplot showing whole soil  $^{13}\text{C}$  values for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.



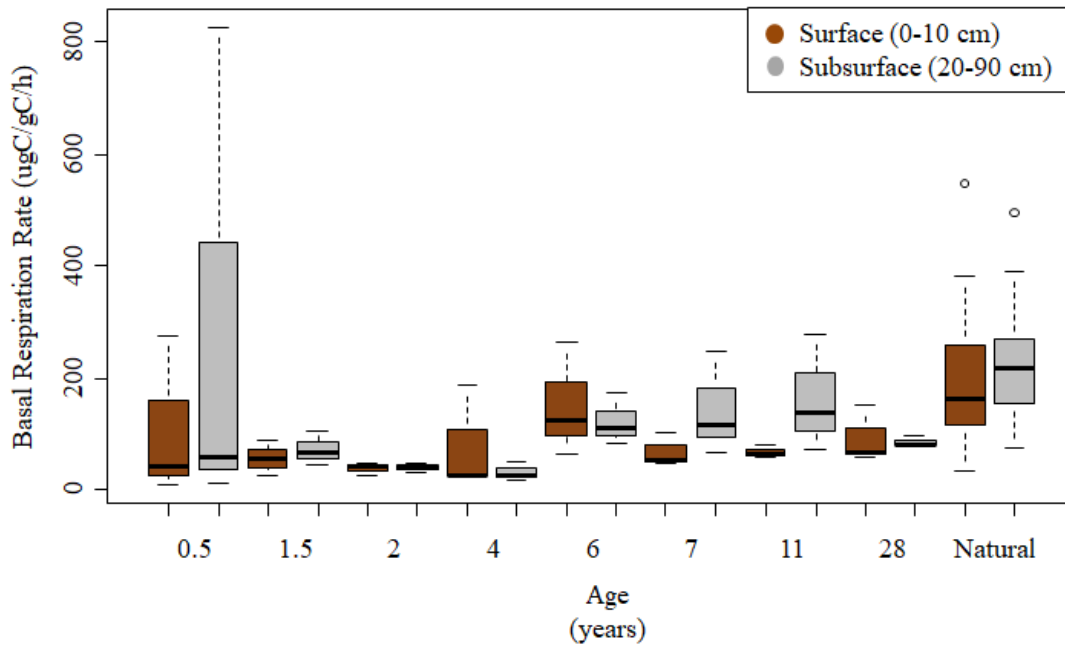
**Figure A.7: Boxplot showing 4 mm and 250 um aggregate size proportions for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.**



**Figure A.8: Boxplot showing 4 mm and 250 um aggregate C:N values for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.**



**Figure A.9: Boxplot showing 4 mm and 250 um aggregate  $^{13}\text{C}$  values for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.**



**Figure A.10: Boxplot showing basal respiration rates ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) for surface (0-10 cm) and subsurface (20-90 cm) depths by stockpile age and for natural reference sites.**

## APPENDIX B

**Table B.1: Estimated marginal means and standard errors for soil respiration rates for natural and stockpiled soils, by sampling depth, both normalized on a per gram of carbon basis ( $\mu\text{gC gC}^{-1} \text{h}^{-1}$ ) as presented in section 3 of this study and on a per gram of soil basis ( $\mu\text{gC g}^{-1} \text{h}^{-1}$ ) for comparison.**

	<b>Basal Respiration (<math>\mu\text{gC gC}^{-1} \text{h}^{-1}</math>)</b>	<b>Basal Respiration (<math>\mu\text{gC g}^{-1} \text{h}^{-1}</math>)</b>
<b>Depth=0-10 cm</b>		
Site= Natural	210 $\pm$ 45	1.34 $\pm$ 0.84
Site= Stockpiled	84 $\pm$ 38	3.61 $\pm$ 0.71
<b>Depth=20-30 cm</b>		
Site= Natural	212 $\pm$ 43	0.89 $\pm$ 0.83
Site= Stockpiled	89 $\pm$ 38	3.83 $\pm$ 0.71
<b>Depth=80-90 cm</b>		
Site= Natural	245 $\pm$ 43	1.05 $\pm$ 0.83
Site= Stockpiled	166 $\pm$ 40	3.09 $\pm$ 0.74