Hydrogeochemical soil dynamics relative to topography for forested land units undergoing reclamation in a post-mined landscape in the Athabasca Oil Sands Region, Alberta

by

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Author's Declaration

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

I understand that my thesis may be made electronically available to the public.

Abstract

Natural forest soils of the Western Boreal Forest rarely witness near-surface soil flushing events during the growing season due to the forest's excessive evapotranspiration demands and large unsaturated zone storage capacity. This leads to the accumulation of nutrients such as Soluble Reactive Phosphorus (SRP) and Total Inorganic Nitrogen (TIN) within the surface soils, increasing along a low-relief moisture gradient transitioning through upland forests, riparian zones and wetlands, influencing vegetation communities. In the post-mined landscape, decompressed overburden produce topographically elevated hillslopes with cover soils exhibiting poor transmissivity and hydrophobic properties, which are often subject to erosion. Reclamation projects are beginning to develop entire watersheds consisting of engineered wetlands, uplands and hillslopes, varying in elevation, to ensure a hydrologic connectivity that can support resiliency to moisture deficit during periodic stresses. To avoid undesirable interactions between land units, it is important to understand their hydrogeochemical connectivity. This study focuses on the interactions between a recently (i.e. three years) reclaimed low-relief upland and three encompassing hillslopes (aged five to nine-years since reclamation), located within a constructed fen watershed. The objectives were to determine if topographically driven moisture-nutrient gradients were being formed and how this would influence vegetation colonization.

No topographically driven moisture-nutrient gradient was detected within the lower-lying constructed upland, attributed to the heterogeneity of the cover soil placement and the lack of preferential flow paths, typically witnessed in newly reclaimed soils. Furthermore, the application of control release fertilizer likely hindered the detection of any topographic influence on ion mobility. Runoffs collectors suggest that fertilizer may lead to off-site movement immediately following application. Results also demonstrated that SRP is likely in excess within this system and susceptible to leaching following overland flow events. However, TIN is potentially a limiting nutrient and while immobilized at the surface, demonstrated greater susceptibility towards vertical flow, especially when groundwater recharge promoting structures are incorporated within the construction of forested land units. Sapling survival within the constructed upland appeared to be influenced by moisture stress over nutrient availability, re-

examining the need for fertilizer application when reclaimed soils still lack moisture absorbing properties.

The elevated hillslopes also did not demonstrate any topographically driven moisturenutrient gradient regardless of age since reclamation. The more mature hillslope was expected to demonstrate such a gradient, however the dry growing season likely hindered subsurface interflow downslope. The two younger hillslopes still demonstrated poor transmissivity attributed to their immaturity. TIN contributions towards the constructed upland proved to be minimal, however phosphorus inputs from erosion prone areas are likely to influence SRP availability following phosphate desorption processes within the constructed upland. Although our system demonstrated positive correlations of increased SRP on native species establishment, TIN availability demonstrated increased forb and non-native species colonization.

This study demonstrates how current forested upland reclamation practices might influence other land units when re-initiating hydrogeochemical connectivity throughout engineered landscapes. This study also demonstrates how contributions from topographically elevated land units might impact vegetation communities downslope, which is crucial for reestablishing the resiliency of the landscape. Current forest upland and hillslope reclamation practices will likely need to be re-evaluated when considering landscape scale hydrogeochemical connectivity.

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Chapter 1: General Introduction 1.1 Background and Rationale for Research

The Athabasca Oil Sands Region (AOSR) is located in north-eastern Alberta, Canada covering over 142,000 km² of the Boreal forest (Johnson and Miyanishi, 2008). This region is currently facing landscape scale disturbances mostly due to industrial development for bituminous oil sands extraction through in-situ recovery and open pit mining (Rooney et al., 2012). The AOSR contains the world's third largest proven crude oil deposit (~166 billion barrels), where an estimated 4800 km² has been deemed feasible for recovery of near-surface crude reserves by open pit mining, which requires the removal of vegetation and soil horizons to depths of about 70 meters (Government of Alberta, 2015; Rowland et al., 2009). Much of the habitat disturbed through these processes are wetlands (~65%), most notably, fen peatlands (~90%) (Khadka et al., 2016; Vitt et al., 1996).

Fen peatlands are a crucial component of the AOSR landscape, as they support vital ecosystem processes such as water storage and cycling, habitat biodiversity, and the storage of carbon and nutrients (Nwaishi et al., 2015a). The resiliency of this landscape in the sub-humid climate of the region is due to the hydrogeological connectivity present amongst land units, where upland forests and peatlands re-distribute moisture and solutes to one another through groundwater flow, and occasionally, surface runoff (Dimitrov et al., 2014; Westbrook et al., 2006). The Alberta government requires energy companies to reclaim all disturbed land to "equivalent capability", where functional eco-services can be re-established. However, achieving this regulatory requirement has been a major challenge due to the disruption of the natural hydrogeological connectivity that is characteristic of the pre-disturbance landscape (Johnson and Miyanishi, 2008; Price et al., 2010). Additionally, the chemical properties of the constructed landcape will be different because post-mined landscape consist of highly alkaline and sodic waste material as a result of the bitumen extraction process (MacKenzie and Quideau, 2012). Although peatlands may take thousands of years to develop, it is believed that re-creating proper hydrogeological conditions, combined with peatland restoration techniques, could make the development of peatlands possible within a few decades (Daly et al., 2012; Price et al., 2010). Therefore, the incorporation of constructed forested upland-peatland systems is essential for mine closure plans where peatland formation is required. There are currenly two constructed

systems in the AOSR where ongoing research and monitoring will dictate the potential for future fen reclamation (Ketcheson et al., 2016; Pollard et al., 2012).

Upland reclamation has been successful in the past (~0.15% of disturbed land), however these reclaimed systems vary considerably from their natural analogues (Audet et al., 2015; Wu, 2015). The natural uplands of the AOSR are categorized by a low relief (~7-12%) with deep glaciated substrates varying in water storage and transmissivity properties (Devito et al., 2005; Rowland et al., 2009). The variability in these hydrological properties in turn, influences how moisture and solutes are re-distributed amongst the land units. Differences in slope position and soil substrate create moisture gradients, which influence biogeochemical cycling and vegetation establishment (Dimitriu and Grayston, 2010). Despite this gradient generally increasing towards the lowlands of catchments, surface re-distribution of mositure is rare within this landscape as deep unsaturated zone storage, vegetation water demand and vertical flow dominate over lateral flow (Devito et al., 2005; Johnson and Miyanishi, 2008; MacKenzie and Quideau, 2010). As such, infrequent surface soil flushing leads to the accumulation of readily available nutrients such as inorganic nitrogen and phosphorus, which when mobilized can greatly alter ecosystems situated in topographic lows by inputting readily available sources of nutrients (Macrae et al., 2006; 2005).

Reclaimed upland soils differ from natural soils primarily due to their limited transmissivity, particularly in the first years after placement (Keshta et al., 2010; Ketcheson and Price, 2016a). Salvaged organic amandements, such as Forest Floor Material (FFM) or Peat-Mineral-Mix (PMM), are commonly used as cover substrates to prevent sodic-alkaline waste water from reaching the rooting zone, all while maintaining adequate moisture for the vegetation (Jung et al., 2014; Naeth et al., 2011). These substrates can demonstrate hydrophobic properties and relatively low permeability, leading to frequent surface runoff events and overland flow (Hunter, 2011; Ketcheson, 2015). Furthermore, the mixing and degradation of the salvaged material during placement disturbs the pre-excavation soil structure, presenting greater heterogeneity within reclaimed systems (Ketcheson and Price, 2016c; Macdonald et al., 2015b; Mackenzie, 2011). Additionally, given the abundance of overburden mine waste following excavation, newly constructed landscapes will inevitably possess greater topographic variability, inserting uncharacteristic hillslopes throughout the AOSR (Leatherdale et al., 2012; Rowland et al., 2009). The incorporation of topographically elevated landscapes with heterogeneous soil

structures and salvaged materials will inevitably alter biogeochemical dynamics throughout newly developed landscapes and therefore vegetation communities. Forested upland reclamation strategies are likely going to need to evolve when considering watershed engineering.

1.2 Monitoring upland reclamation in constructed watersheds

The purpose of reclamation is to accelerate successional processes, enabling pedogenic development and eventually return the disturbed land to self-sustaining conditions similar to predisturbance (Welham, 2013). Upland reclamation strategies are developed relative to soil characteristics and position (i.e. aspect, slope, soil type and texture), where soil moisture regimes and soil nutrient regimes determine attainable ecosites ranging from xeric to sub hydric moisture regimes and poor to rich nutrient regimes (Daly et al., 2012; Mackenzie, 2011). The designated ecosites are used to determine afforestation procedures, where future management practices (. i.e. fertilizer, erosion control) are conducted if required (Audet et al., 2015; Turcotte et al., 2009). Monitoring reclaimed systems typically consists of comparing novel ecosystems with natural forestlands, where edaphic characteristics (i.e. organic matter accumulation, vegetation colonization, microorganism communities, hydrophysical properties, physicochemical properties) are used as functional biometrics for assessing successional development (Macdonald et al., 2015b; McMillan et al., 2007; Turcotte et al., 2009). Although these studies have proven to be crucial in developing optimal reclamation strategies, they focus on isolated land units where landscape connectivity is not required.

Watershed reclamation is a new concept in the AOSR and studies are beginning to demonstrate the feasibility of engineering such systems. Ketcheson and Price (2016c) found that re-initiating the hydrogeological connectivity necessary to sustain fen-peatlands using salvaged mine residues was possible, concluding that the materials are continually evolving and therefore require long term monitoring. Similarly, Kessel (2016) found that the incorporation of groundwater recharge promoting structures can aid in enhancing hydrogeochemical connectivity, while minimizing the damaging effects of alkaline sodic mine residues towards both upland and wetland vegetation. Currently, no research has focused on nutrient mobility throughout constructed watersheds. Leatherdale (2008) looked at topographic differences in nutrient regimes along a reclaimed slope, as did MacKenzie and Quideau (2010) in a similar study, however both focused solely on the surface soils of isolated upland systems. Jung et al. (2014) determined that

capillary barriers formed by the textural discontinuity between cover soils and underlying overburden delayed nutrient losses through leaching. However, the poor moisture and nutrient retaining abilities of the tailing sands material led to considerable losses once nutrients moved past the interface, no longer accessible for the vegetation. As moisture-nutrient gradients possess a strong control on vegetation community dynamics, it is important to comprehend how these abiotic components are re-distributed throughout engineered landscapes. Given the influence that topographically elevated uplands and hillslopes are likely to have on water and solute re-distribution, an understanding of the chemical evolution of the landscape is essential when recreating watersheds containing fen peatlands.

1.3 Research Objectives

Upland and hillslope reclamation research have yet to determine how nutrient leaching from one land unit might influence the vegetation communities of the hydrologically connected downslope land unit. Hence, the overall objective of this research project is to gain a better understanding of how topography will influence nutrient dynamics in constructed fen watersheds. Specifically, the three main objectives are to:

- Assess spatial and temporal variability in the hydrophysical properties of a three-year-old reclaimed upland and to relate those properties to nutrient availability and vegetation community establishment.
- Assess spatial and temporal differences in hydrophysical and physicochemical properties between three reclaimed hillslopes and, to predict their hydrogeochemical connectivity with the constructed upland located at the topographic low of the slopes.
- 3) Synthesize the findings from the first two objectives to inform future management practices necessary when conducting upland reclamation within constructed watersheds.

1.4 Thesis Structure

This thesis consists of six chapters. The first chapter provided a general introduction to the themes covered in the thesis and outlines the goals and objectives of this thesis.

The second chapter is a literature review, intended to collect and synthesize the available information on: (1) the characteristics of natural forested uplands in the AOSR, (2) natural hydrogeochemical landscape connectivity throughout the AOSR, (3) reclamation procedures and, (4) soil development and plant colonization in post-mined landscape.

The third chapter contains an overview of the study site, as well a general description of the methodologies common to both research questions.

The fourth chapter addresses the first of the two primary research goals by investigating the influence topography might have on the hydrophysical and physicochemical properties of newly reclaimed cover soils, and the potential influence on vegetation communities.

The fifth chapter addresses the second of the two primary goals by investigating variability in hydrophysical and physicochemical properties between three reclaimed hillslopes varying in age since reclamation, and predicting their connectivity with a constructed upland situated at the toe of the slopes.

The sixth chapter addresses the third and final objectives and consists of a synthesis of the conclusions drawn from the two research chapters, which placed them in context for developing new research ideas, and recommendations for future studies of hydrogeochemical connectivity in reclaimed watersheds

Chapter 2: Literature Review

2.1 Introduction

Oil sands mining is projected to continue to impact the pristine boreal forests of northeastern Alberta, with an estimated 1400 km² of land set to be developed by 2023 (Alberta Government 2015). The Alberta Government requires oil companies to restore disturbed areas back to a state of equivalent capability (Alberta Government 2000). Although upland reclamation has been successfully accomplished in the past, upland land units make up only 23% of the territory set to be disturbed, while the remaining are wetlands, especially fen peatlands (Rooney et al., 2012). Price et al. (2010) developed a conceptual design for watershed reclamation that suggested that the creation of self-sustaining peatlands could be feasible if the fen is placed in a topographically lower point where adequate water supply can sustain the relative wetness required for the establishment of fen vegetation. The conceptual design (Price et al., 2010) relies on the hydrogeological connectivity between a constructed fen-upland system, where both groundwater flow from the upland, supplemented by surface runoff contribution from the surrounding hillslopes would maintain adequate moisture conditions for the fen (Price et al., 2010). While much research has been conducted to improve upland reclamation, most of these studies were conducted on isolated land units (Rooney et al. 2012). In the natural forested uplands of the WBF, the sub-humid climate and large moisture storage capacities of the soils render substantial runoff events infrequent (Brown et al., 2010; Redding and Devito, 2010). This leads to the accumulation of solutes within the surface soil, susceptible to large releases when wet conditions do occur (Macrae et al., 2006; 2005). Poor moisture retaining abilities, lack of vegetation and the inclusion of topographically elevated hillslopes on newly reclaimed landscapes generally make these systems prone to runoff and erosional processes, especially within the first few years after placement (Audet et al., 2015; Raab et al., 2012). As solute and nutrient loading into bodies of water or wetlands can lead to issues such as eutrophication and changes in vegetation communities (Lamers et al., 2012), it is important to understand how this might impact the development of constructed fen-peatland systems, as well as other land units incorporated within the designs.

2.2 Characteristics of natural forested uplands of the AOSR

Forested uplands are a crucial component of the AOSR as they convey multiple hydrogeological functions to the entire landscape (Devito et al., 2005; Ketcheson et al., 2016). These functions are a direct result of the geological formation of this area. The bitumen reserves of the AOSR are the delta deposits of an ancient boreal ocean, where organic matter and hydrocarbons accumulated and were subsequently compressed by extensive glacier formation. The marine deposits eventually formed the Clearwater shale deposits of the Athabasca, lying directly above the economically valuable bitumen (Mossop, 1980; Rowland et al., 2009). Pleistocene epoch deposits then formed a range of geological structures throughout the AOSR, each varying uniquely in hydrogeophysical properties (Lanoue, 2003). These deposits range in terms of particle size distribution and other physical characteristics from coarse-textured Orthic Luvisols with elevated water transmissivity, to veneer-textured Gleyed Luvisols with complex hydrological processes, to finally fine-textured Gleysolic soils with poor hydrological transmission (Lanoue, 2003; MacKenzie and Quideau, 2010; Norris et al., 2013). The impeded water flow induced by the increased clay content of the mineral subsoils led to the formation of several forested upland ecosites (a-g) ranging from a xeric to sub-hydric moisture regimes, rich to poor nutrient regimes and, from aerobic to increasingly anaerobic conditions (Rowland et al., 2009; Turchenek and Lindsay, 1983). Individual ecosites (Figure 2.1) contain unique controlling factors (e.g., soil moisture, nutrients, soil aeration, soil temperature, competition), which are representative of the dominant vegetation, as these communities have adapted in response to those limitations (Alberta Environment, 2009). Five site types (i.e., Dry, Wet-poor, Wet-rich, Moist-poor, Moist-rich) exist throughout the AOSR varying in Soil Moisture Regime and Soil Nutrient Regime (Alberta Environment, 2009; Kemball and Bielich, 2010). Because multiple ecosites exist within the landscape, varying along topographic position, it is difficult to generalize specific nutrient regimes of the AOSR. Boreal ecosystems have tightly regulated internal biogeochemical cycling between plants and soils, where organic matter composition, microbial communities and plant communities are strongly interlinked (Hemsley, 2006; Quideau et al., 2013). Nutrients, mostly in organic form, are returned to the forest floor as leaf litter and converted into plant-available forms primarily through mineralization (Huang and Schoenau, 1997). Therefore, nutrient regimes differ between stands in response to canopy composition (Jerabkova et al., 2006).



Figure 2-1 Description of ecosites in relation to their Soil Moisture Regimes (SMR)/ Soil Nutrient Regimes (SNR). Source: Geographic Dynamics Corp. (2006).

2.3 Hydrogeochemical landscape connectivity throughout the AOSR

2.3.1 Hydrological re-distribution

The AOSR of north-eastern Alberta (142,000 km²) covers a considerable portion of the Western Boreal Plains (WBP), an ecozone that extends over 650,000 km² (Turetsky et al., 2010). This landscape is a mosaic of wetlands, ponds and shallow lakes that are hydrologically connected with densely covered forested uplands and riparian zones (Dimitrov et al., 2014; Westbrook et al., 2006). The sub-humid climate of this ecozone is categorized by a long-term (~30 years) water deficit, where potential evapotranspiration (PET) is often greater than precipitation (P) (Brown et al., 2010). In this landscape, the interaction between climate and geological formation controls ecological functions and hydrologic re-distribution (Johnson and Miyanishi, 2008; Macrae et al., 2006, 2005). During the typical dry years of the climate cycle, most the annual precipitation (65-75%) arrives as rainfall over the growing season, when much of the forested uplands are water limited, and PET is at its highest and Actual Evapotranspiration (AET) depends on the confinement of the groundwater. Although precipitation during the non-

growing season (snowfall) is rather minimal (~30%), the dormancy of the plants, thus low PET results in a net surplus of water availability, where, depending on the geological formation and antecedent moisture conditions of the soil, can be available for re-distribution (Brown et al., 2010; Petrone et al., 2007; Smerdon et al., 2005).

Two hydrological land units co-exist within this landscape: (1) wetlands: considered to be water sources in this landscape as their limited storage capacities often lead to over-saturation, and (2) upland forests: considered to be moisture sinks due to their deeper, drained soils where PET can be significant because of root penetration to considerable depths (Devito et al., 2005). These Hydrological Units overlay Hydrological Response Areas, which are categorized as areas possessing sediments of similar grain sizes and permeability (i.e. Fine textures, Coarse textured, Veneer-type). Antecedent moisture conditions of both the hydrological units and hydrological response areas directly influence runoff following precipitation events. Under conditions of moisture deficit, precipitation will generally go into soil storage. However, during relatively short episodes, every 10-15 years, P>PET, the forestlands soil storage capacity is exceeded and precipitation inputs contribute substantially to near-surface flushing and hydrological re-distribution throughout the landscape (Kreutzweiser et al., 2008; Pelster et al., 2008b; Putz et al., 2003). The WBP is hydrologically connected by runoff and groundwater. During dry periods of the wet-dry cycle, the landscape goes through periods of disconnection, where the land units are poorly linked through surface runoff. However, when rare precipitation events do occurs, wetlands can contribute considerable sources of water to upland forests via hydraulic lift from the roots of forested upland species (Norton and Hart, 1998; Dawson et al., 1993; Hutline et al., 2006).

2.3.2 Solute and nutrient re-distribution

The transport of nutrient within the WBP landscape is governed by soil texture as the permeability and infiltration capacities determine the proportion of water that can move as subsurface or overland flow, as well as the reduction-oxidation conditions (Kreutzweiser et al., 2008; Prepas et al., 2008). The forestlands of the WBP tend to be nitrogen limited systems. Most forestland nitrogen is cycled from trees to litter layers and surface soils, where microbial activity will mineralize the organic nitrogen into inorganic nitrogen, in the form of ammonium (NH₄⁺) and nitrate (NO₃⁻) (Quideau et al., 2013; Westbrook et al., 2006). Both these forms of inorganic

nitrogen are readily available as plant nutrients, however NO_3^- is the more mobile of the two forms, and therefore, the most often lost through leaching (Kreutzweiser et al., 2008). The infrequent flushing of near-surface soils, may result in a buildup of inorganic nitrogen at the surface, where significant losses from surface runoff occur once every 10-15 years when the upland water table reaches approaches the surface (Devito et al., 2005; Macrae et al., 2006). The export of inorganic nitrogen in surface runoff (mostly NO_3^-), peaks in the spring and decreases in the summer as nutrient demand from the growing vegetation increases (Pelster et al., 2008b). During the typical dry periods, groundwater flow and root distribution may be an important pathway by which inorganic nitrogen is exported between land units (Hinton et al., 1994; Kreutzweiser et al., 2008; Macrae et al., 2006).

Inorganic phosphorus is primarily weathered from minerals near soil surface horizons, where it becomes highly labile and mobile (Kreutzweiser et al., 2008). The inorganic phosphorus is then assimilated and converted to organic phosphorus by plants and microbes. As organic soil matter decomposes, organic phosphorus is mineralized into plant/ microbe available inorganic forms. The labile nature of phosphorus makes adsorption to particulates an important sink for bio-available P, therefore any hydrological events or processes that move suspended particles through surface or subsurface flow paths can increase phosphorus leaching out of the ecosystem (Kreutzweiser et al., 2008; Putz et al., 2003). Although the WBP is generally considered to be low-relief ($<4^{\circ}$), the impeded infiltration of fine-textured soils renders erosion as being the strongest influence on phosphorus export rates, where rapid increases in overland flow during storm events can flush particulates that have accumulated on the organic and till layers of the forest floor (Kreutzweiser et al., 2008; Prepas et al., 2001; Putz et al., 2003). Loses of phosphorus from forest soils can also be accelerated by co-leaching with organic solutes. Dissolved Organic Carbon (DOC) can decrease the sorption of phosphorus to particulates by occupying the binding sites on metallic oxides (Fe, Al, Ca), mobilizing the phosphorus (Kreutzweiser et al., 2008). As with nitrogen, phosphorus rich surface soils are vulnerable to flushing in wet years. However, the mineral subsurface soils of the WBP possess a high adsorption affinity for phosphorus, and therefore limit it's mobility within groundwater (Kreutzweiser et al., 2008; Macrae et al., 2005). Phosphorus re-distribution is more likely to occur between units where surface hydrological re-distribution is prevalent and where organic

soils limit the buffering capacity of phosphorus, such as ephemeral draws and wetlands (Buttle et al., 2000; Macrae et al., 2005).

2.4 Upland reclamation procedures in the AOSR

Open-pit mining removes mine overburden up to 70 meters deep in order to extract bituminous oil sands (Rowland et al., 2009). The first mining procedure extracts the reclamation material, which is generally a Pleistocene or Holocene (e.g. peat, LFH) deposit, ideal for plant colonization. The second extraction process removes the non-saline-sodic overburden from the Pleistocene deposits, deemed not acceptable for surface reclamation, due physical limitations such as: high clay content, pH, sodium absorption ratio and hydrocarbon content. Finally, the Clearwater saline-sodic shale overburden is removed and kept separate from the other materials as they also contain similar elements detrimental to reclamation (Lanoue, 2003; Mackenzie, 2011; Mossop, 1980). Tailing sands, another by-product of open-pit mining, is obtained when the bitumen is isolated from the remaining natural deposits. Once all the bitumen has been mined, the tailing sands and potentially harmful overburden are used to fill the hollowed pits. Suitable Pleistocene epoch overburden and subsoil is then used to create a barrier between roots and the potentially harmful underlying substrates. Finally, the reclamation material is used as a medium for the eventual development of a boreal forest vegetation community (Hemstock et al., 2010; Jacobs et al., 2015; Jamro et al., 2014).

Energy companies must reclaim all disturbed land to *equivalent land capability*, meaning the area must develop towards a self-sustaining system where biotic and abiotic components interact with one another ensuring resilience during periodic stresses (Welham, 2013). Two main types of cover soil are used for reclamation cover in the AOSR: Peat-Mineral Mix (PMM) and Forest-Floor Material (FFM). As peatlands dominate much of the pre-disturbance landscape, peat is a readily available source of organic matter, which is favorable for plant growth (Archibald, 2014; Howell, 2015; Jung et al., 2014). The peat is often mixed (~40-60%) with a mineral substrate (generally suitable overburden material) as this improves the tilth and reduces the risk of losing organic matter due to rapid decomposition (Mackenzie, 2011). Forest Floor Material is the most valuable of the two cover soils as it is a much better representation of predisturbed forested uplands. It provides a source of organic matter, plant nutrients, seed propagules and woody debris un-matched by PMM. Although current reclamation strategies are

transforming the AOSR towards an upland dominated landscape (Johnson and Miyanishi, 2008; Rooney et al., 2012; Rooney and Bayley, 2011), pre-disturbed AOSR is primarily dominated by wetlands, which makes FFM a limited resource and must therefore be carefully managed and utilized (Macdonald et al., 2015b; Mackenzie, 2011; MacKenzie and Quideau, 2012).

Following placement, the cover soil will then be assessed based on the guidelines of the Land Capability Classification System for Forested Ecosystems (LCCS), used to evaluate preand post-disturbed soil capabilities (Alberta Environment, 2006). This system evaluates key soil parameters including soil moisture regimes and soil nutrient regimes, as well as other potentially limiting physical and chemical properties. During the extraction and placement processes, the organic LFH and mineral layers (A, B, C) are no longer structurally intact, therefore reclaimed soils require unique soil profile nomenclature. The organic layer consists of all reclamation material greater than 17% Total Organic Carbon (TOC) and is used to predict the soil nutrient regimes. The mineral profile (<17% TOC) is classified into three distinct groups: topsoil (~0-20cm); upper subsoil (~20-50cm); and lower subsoil (~50-100cm) and is used to assess the Available Water Holding Capacity (AWHC), which is a predictive measure of the soil moisture regime, obtained by measuring the difference between field capacity and soils wilting point. The information gained from the soil moisture and soil nutrient regimes are then used to assess potential ecosites, where adaptive site specific management strategies will aid with accelerating the successional stages of reclamation (Alberta Environment, 2009, 2006; Mackenzie, 2011; Straker and Donald, 2010).

Once potential ecosites are established, afforestation is conducted relative to the vegetation composition of the targeted ecosites following the Guidelines For Reclamation to Forest Vegetation in the AOSR (Alberta Environment, 2009). Management strategies integrated within reclamation procedures include the addition of woody debris, the establishment of non-competitive stabilising species (*i.e.* use of vegetation for erosion control) and the application of fertilizer to supplement the soil nutrient regimes of the targeted ecosites (Johnson and Miyanishi, 2008; Kwak et al., 2016; Rowland et al., 2009; Sloan et al., 2016). Newly placed cover soils differ substantially from natural forested uplands in nutrient availability and biogeochemical cycling (Quideau et al., 2013; Rowland et al., 2009). Fertilizer application to reclaimed areas is often needed to aid with the establishment of both planted species and native communities (Haase et al., 2007; Pinno et al., 2012; Sloan et al., 2016). Although often beneficial,

inappropriate use of fertilizer can be detrimental to desirable plant species and often promotes the establishment of weeds and herbaceous species outcompeting planted tree and shrub species and preventing the development of locally common boreal species, compelling the need for better fertilizer practices (Landhäusser and Lieffers, 1994; Mackenzie, 2011; Wilson and Pinno, 2013).

Immediately available fertilizers (IAF), traditionally used in agriculture, reclamation and forestry release nutrients instantly upon application with generally low fertilizer use efficiency rates (FUE) as a result of exceeding the soils total exchange capacity and plant uptake rates, leading to off-site movement (Jacobs et al., 2005; Sloan et al., 2016). Therefore, the application of Controlled Release Fertilizers (CRF) has been emphasized as these fertilizers release nutrients gradually over longer time periods, minimizing nutrient leaching while improving overall FUE. CRFs consist of polymer coated granules gradually releasing nutrients to the soil in relation to increased osmotic pressure and are therefore affected by factors such as: temperature; soil moisture and time (Azeem et al., 2014; Kochba et al., 1990). Although CRF's exhibit numerous benefits, a proper understanding of how these fertilizers function within the altered hydrology of constructed soils is still lacking and studies have only been performed on PMM (Chang et., 1996; Sloan et al., 2016).

2.5 Soil development and vegetation colonization in a post-mined landscape

2.5.1 Pedogenic processes

Land reclamation in the AOSR involves accelerating pedogenic processes by building artificial soils (anthrosols) (MacKenzie and Quideau, 2010; Naeth et al., 2012). Contrary to secondary succession (e.g., fire, infestation, harvesting), open pit mining unsettles all previous soil structure and propagule banks, requiring the land to evolve through complete initial ecosystem development (Raab et al., 2012). The primary successional stages are mostly abiotically dominated and development is almost exclusively dependant on the substrate properties, emphasizing the importance of salvaging proper reclamation material as this stage determines further pathways of ecosystem evolution, such as plant succession (Hunter, 2011; Macdonald et al., 2015a; Mackenzie, 2011). In the cooler climate of the AOSR, initial succession is generally characterized by slow colonization, with nutrient and water availability being limiting factors (Raab et al., 2012). During this abiotic stage, soil properties and structure change

rapidly because of the initial disequilibrium of the parent material. For instance, particle size and mineral content, related to pore space and hydraulic properties of the soil, influence ecosystem processes such as: infiltration, surface runoff, water retention, ground water recharge and soil-atmosphere interactions (Freeze and Cherry, 1979; Raab et al., 2012). The poor soil structure of these initial landscapes make them incredibly susceptible to erosion, which in constructed landforms, is among the most common impediment to successful reclamation (McKenna, 2002). For these reasons, topographic positioning strongly affects early soil development and spatial patterns in initial ecosystems (Raab et al., 2012).

As the abiotically driven system begins to stabilize, pioneer vegetation gradually starts colonizing the soils, contributing the first deposits of Soil Organic Matter (SOM), one of the most significant pedogenic processes. For instance, SOM possesses multiple cation binding sites, ameliorating the soil's cation exchange capacity, which includes plant essential nutrients, ultimately limiting the potential for nutrient flushing. SOM also possesses strong water retaining capabilities, generated from strong adhesion forces, crucial in a post-mined landscape, which contains greater topographic ranges than pre-disturbance (Leatherdale et al., 2012). Increases in SOM also increases nutrient availability and the pH buffering capacity of the soil, while decreasing bulk density (Anderson, 2014). Furthermore, annual freeze and thaw processes enables the formation of cracks within the soil, which develops preferential flow paths, channeling the transport of dissolved SOM and nutrients into the subsoil, resulting in increased bioactivity and root growth (Anderson, 2014; Raab et al., 2012; Turcotte et al., 2009).

As the soil continues to stabilize, the biotic components, specifically microorganisms become increasingly important, as their decomposition of SOM is the first step towards the development of soil horizons. The decomposition of SOM leads to the mobilization of trace elements and base cations within the uppermost layers of the soil, favouring the leaching of these reactive cations into the lower sublayers (i.e. podsolization) (Carrillo-Gonzalez et al., 2006; Raab et al., 2012). As SOM increases, organic matter is bound to the metal elements of the clay particles, creating chelated compounds, which are translocated into the mineral layer, decreasing the mineral content of the soil (Carrillo-Gonzalez et al., 2006). Recreating the functional microorganism communities of the pre-disturbed AOSR is crucial for the proper reclamation of these soils as they regulate the natural biogeochemical cycles of undisturbed forested uplands. Research has shown that there is potential for assessing ecosystem recovery on reclaimed sites

by examining shifts in microbial community structure and by profiling the resulting nutrient regimes (Dimitriu et al., 2010; Hahn, 2012; Quideau et al., 2013). This further contributes to the need for proper material salvaging and optimal usage of FFM, as the majority of organic matter used is predominantly peat, with the microbial communities being representative of the acidic and anaerobic conditions of peatlands and not that of typical forest upland soils (MacKenzie and Quideau, 2010).

2.5.2 Vegetation colonization

Plant colonization in a post-mined system is often limited by a combination of extreme pH values, high salinity, low organic matter, increased bulk density and the poor soil-structure of the excavated substrate (Mackenzie, 2011; Raab et al., 2012). Poor soil structure can result in unstable slopes by reducing the supporting capacity of soils for plant growth (Welham, 2013). Once soil structure begins to stabilize, autogenic (i.e. biotic) processes drive the presence of spatial structure in vegetation through positive feedback mechanisms, where plants modify the physical environment to their advantage, favoring their reproduction and continued environmental modification (Clements, 1916; Raab et al., 2012). Newly disturbed areas tend to be dominated by forbs and graminoids, with shrubs becoming a larger component of the ecosystem as the stand develops (Wilson and Pinno, 2013). The degree of disturbance inflicts a major role on how species are colonizing the system. For example, research conducted by Errington and Pinno, (2015) compared successional communities post-fire and post-mined disturbances, concluding that disturbance severity was relative to the regeneration mechanisms in the recovering plant community. Post-fire locations only had native perennial species, as these species quickly regenerated from existing root system. However, this existing root system is absent in post mined forest soils, allowing for the occupation of weedy-herbaceous, often nonnative early successional annuals and biennial species. This was made increasingly evident as non-native invaders had greater cover in PMM comparatively to the FFM as the PMM propagules are not accustomed to the same environmental conditions as that of FFM (Errington and Pinno, 2015a).

The autogenic processes initiated by the pioneer species colonizing heavily disturbed soils can be problematic if these species are non-native to the landscape as they have the potential to alter ecosystem development over time, suppressing diversity for the long term (Wilson and Pinno, 2013). Furthermore, these species out-compete planted sapling for requirements such as nutrients and moisture (Landhausser et al., 2007; Sloan et al., 2016; Smreciu et al., 2013). Nitrogen is thought to be one of the main limiting resources for tree growth in reclaimed ecosystems, which is considered to be one of the final successional stages before land can become self-sustaining and certified reclaimed (Jung et al., 2014; Pinno and Hawkes, 2015).

2.6 2.6 Summary

This literature review demonstrates the strong potential for nutrient flushing into topographically lower land units in reclaimed landscapes. Pre-disturbed forestlands of the AOSR rarely witness surface runoff during the growing season, therefore, the transport of nutrients is not necessarily influenced by topography. Conversely, the lack of soil structure within newly placed cover soils implies that runoff and erosion occur rather frequently. This means that areas heavily impacted by erosion will likely lead to phosphorus loading, and areas receiving runoff contributions will likely be rich in both phosphorus and nitrogen. Although nutrients are often a limiting factor, the addition of fertilizer to novel ecosystems is likely to leach out of the system by exceeding the soils exchange capacity. This could potentially lead to the colonization of undesirable species within constructed peatlands. As many invasive species initiate autogenic processes, this can alter ecosystem dynamics over time, jeopardizing the successful colonization of fen peatland species. Therefore, there is a need to understand the influence topography has on hydrophysical properties in reclaimed systems, as these properties influence spatial and temporal physicochemical components of the soil, which, in turn influence vegetative dynamics.

Chapter 3: Materials and Methods

3.1 Site Descriptions

3.1.1 Upland-fen system

The study was conducted on a constructed watershed (56°55.944'N, 111°25.035'W), herein referred to as the Nikanotee Fen Watershed, consisting of an upland-fen system surrounded by three previously reclaimed hillslopes and one natural hillslope. The Nikanotee Fen Watershed was constructed on an overburden dump within the Suncor Energy Inc. Millennium mine lease, ~40 km north of Fort McMurray, Alberta (Figure 3.1). The upland is underlain by a 3 m thick tailing sand aquifer, situated above an impermeable engineered geotextile clay liner, designed to support sufficient lateral groundwater flow from upland to fen to maintain suitable hydrological conditions under periods of water stress (Price et al., 2010). The upland system comprises two units, the first being a 2.2 ha transition area (hereafter named "transition zone"), designed to intercept runoff from both the constructed upland and surrounding hillslopes. The second unit (5.5 ha) comprises the remaining upland area (hereafter named "upland"), consists of strategically placed hummocks and hollows aimed to detain runoff from the constructed watershed while simultaneously promoting infiltration and groundwater recharge (Daly et al., 2012). The hummocks (topographically elevated areas ~ 1 m), enclose exposed tailing sands (i.e. recharge basins) where the capping layer was removed to increase infiltration following surface runoff contributions (Ketcheson and Price, 2016c; Kessel, 2016). The upland was constructed with a topographic slope of ~2-3%, hydrologically modeled to provide a sufficient hydraulic gradient to support the movement of water downslope without excessive seepage or ET loss. The cover soil used for the upland was salvaged from a "Moist-rich" (ecosite d) forested upland soil (FFM), characterized by a mesic moisture regime and medium nutrient levels (Beckingham and Archibald, 1996). The FFM (~0.3-0.5m) was transferred directly to the resurfaced upland and never stockpiled. Particle size distribution was variable, with a mean soil classification of sandy-loam (52% sand, 42% silt and 6% clay) (Ketcheson and Price, 2016c). The surface of the FFM was tilled in the autumn of 2013 in an effort to increase the recharge to the upland aquifer through increased retention of surface water and improved surface infiltration capacity (Ketcheson and Price, 2016c).

An experimental peat-lined basin (~0.2 ha; 0.5 m) occupies the southern portion of the upland. The lower lying fen (2.9 ha), situated at the toe of the upland, is 2 m of moderately decomposed peat, overlying a 0.5 m thick petroleum coke layer (mine waste), that extends ~100 m into the transition zone portion of the upland. Peat substrate was also located at the interface between the transition zone and fen peatland.

Planting was initiated over the summer of 2013 and guided by the Cumulative Environmental Management Association (CEMA) Revegetation Manual (Alberta Environment, 2009). Approximately 10,188 tree saplings were planted on June 21st 2015 with the addition of 10 g continuemTM RT (18:9:9:9(S)) controlled release fertilizer (CRF) biodegradable paper packets, applied to each individual sapling, at an approximate rate of 17,56 kg/ha. Revegetation strategies were developed according to soil characteristics derived from the watershed design, which predicted soil moisture and nutrient regimes (Daly et al., 2012). Following the guidelines from the Land Capability Classification System (LCCS) (Alberta Environment, 2006), revegetation strategies segregated the upland into two distinctive vegetation communities (i.e. ecosites). The transition zone (community 1) was planted as a "rich treed fen" (ecosite k1.1), characterized by flowing water and alkaline nutrient rich conditions (Alberta Environment, 2009). Planting here consisted of: black spruce (Picea mariana), labrador tea (Rhododendron groenlandicum), willow (Salix sp.), dwarf birch (Betula pumila), bog cranberry (Vaccinium oxycoccos) and various graminoid species. The remaining upland (community 2), was classified as a "moist-poor site" (ecosite c1.1). This ecosite is generally found at the mid and upper-slope of undisturbed boreal uplands. Revegetation consisted of: jack pine (Pinus bankstana), black spruce, Labrador tea, and an assortment of other shrubs and forbs such as prickly rose (Rosa acicularis), blueberry (Vaccinium myrtiloides) and bunchberry (Cornus Canadensis) (Daly et al. 2012).



Figure 3-1 Map of the upland-fen system, including soil moisture transects (20 access tubes) and incubation plots (9 in total).

Vegetation surveys conducted found that the upland and transition zone combined for 44 species following the removal of rare individuals. Forbs possessed the greatest diversity (28 species) and dominated much of the canopy cover (50%) (e.g., *Sonchus arvensis (non-native, 15%), Bassia scorparia (non-native 9%), Taraxacum officiale (non-native, 5%) and Aster constipuus (native, 5%.)*. Grasses had the second greatest diversity (8 species), also dominating much of the canopy (29%) (e.g., *Agropyron tracycaulum (native, 9%)* and *Hordeum jubatum (native, 6%)*). This was followed by 3 species of shrubs (8%) and 5 species of tree saplings (7%). Native species dominated most the canopy cover (66%) and possessed the highest diversity (28 species). Non-native species composed a large portion of

the canopy cover (26%), however possessed little diversity (8 species) and finally planted species covered little of the canopy cover (8%) and were composed of 7 viable species.

3.1.2 The reclaimed hillslopes

The entire upland - fen system (10.6 ha; Figures 3.1; 3.2) is situated in a much larger reclaimed watershed (32.1 ha) composed of four primary hillslopes. The west (2.4 ha) and southeast (8.4 ha) hillslopes were constructed in 2011 and re-vegetated in 2012. The west slope was planted as a moist rich site (ecosite d/e), where dominant species typically consist of white spruce, aspen and an assortment of shrubs (e.g., Saskatoon berry (Amelanchier alnifolia), pin cherry (Prunus pensylvanica) and chokecherry (Prunus virginiana)). The southeast slope was categorized as a dry site (ecosite b), where dominant species typically consist of jack pine, white spruce, blueberry and a similar composition of assorted shrubs. The east (8.1 ha) hillslope was reclaimed in 2007, and re-vegetation consisted of mostly of white spruce, aspen, white birch, green alder (Alnus crispa), and similar shrubs. The south hillslope is an undisturbed hillslope and was excluded from this study. All three slopes were composed of overburden substrate from the Cretaceous Clearwater formation, dominated by shale and siltstone (Hackbarth and Nastasa, 1979). The Clearwater overburden material is overlain by a ~ 100 cm secondary capping layer of suitable overburden material, which is then capped with \sim 40-50 cm of Peat-Mineral Mix (PMM) cover soil. Both the secondary capping material and PMM soil layers were each directly placed (i.e., not from stockpile) during the construction of the reclaimed slopes in this study.

Vegetation cover on the East slope (87.5%) was considerably more mature than both the southeast (72.5%) and west slope (82.5). On the east slope, tree (27%), shrub (30%) and grass species (25%) dominated, however forbs (18%) were still prevalent. Native (50%) and planted species (39%) composed most the canopy, while non-native species only constituted a portion (11%). On the southeast slope, forbs (50%) and graminoids (34%) dominated, meanwhile shrub (13%) and tree (3%) species were minimal. Native (53%) and non-native species (43%) composed the clear majority, and planted species (4%) were minimal. Finally, on the west slope, forbs (41%) and graminoids dominated (51%), meanwhile shrubs (4%) and trees (5%) only constituted a small portion. Native species (67%) completed much of the canopy, with non-natives (28%) still a considerable constituent, and planted species minimal (5%).



Figure 3-2 Map showing the constructed watershed, including the reclaimed hillslopes. Transects were in relation to a downslope gradient.

3.2 Sampling and field instrumentation

3.2.1 Hydrophysical properties

Transects of soil moisture profile access tubes (PR2 Delta-T Devices[©]) were installed throughout the constructed watershed. These access tubes measure volumetric water content (VWC) at selected depth intervals within the soil profile (10, 20, 30, 40, 60, 100 cm below ground surface (bgs)). Standard calibrations for mineral soils were used when measuring VWC. At each access tube location, additional measurements of surface soil moisture (i.e. 0-7 cm), surface temperature, and pore water conductivity (ECp) were recorded using a Delta-T WET Sensor (type WET-2). Temperature probes (2, 5, 10, 20, 30 cm bgs) built from Thermocouple Wire T Type, Duplex Insulated, (Omega[©]) were installed within the immediate vicinity of soil moisture profile tubes. Transect measurements were conducted twice a week. Total Soil Water (TSW) was converted from the VWC of the soil profile measurements as a estimation of total water content at two depths (15, 35cm), by multiplying the VWC by the thickness of the depth increment (Burk et al., 2000; Leatherdale, 2008),

$$\mathbf{TSW}_{15} = \left(\frac{VMC10}{100} \mathbf{x150}\right) \tag{1}$$

$$TSW_{35} = \left(\frac{VMC10}{100} \times 150\right) + VMC20 + VMC30$$
(2)

where VMC10 is the volumetric moisture content (%) at the 10 cm depth and is assumed to represent the top 15 cm of soil, which represents the approximate depth of average root development, and 35cm the approximate thickness of the FFM (Burk et al., 2000; Leatherdale, 2008).

Soil moisture stations (Figures 3.1; 3.2) were located throughout the constructed watershed and were used to indicate precipitation events which lead to considerable infiltration through the cover soils. These stations contain soil moisture probes (Stevens Hydra II Probe) connected to Campbell Scientific CR1000 data loggers, measuring VMC at various increments within the FFM (5cm, 15cm, 25cm and 40cm) and ~5cm below the FFM/ tailing sand interface. The data loggers recorded 30-minute average values of measurements taken every minute. Precipitation was recorded at the upland meteorological station with a tipping bucket rain gauge (Texas instruments TR-525M).

3.2.2 Physical properties

Incubation plots were placed throughout the constructed watershed and quantification of hydrophysical properties were assessed from the nearest immediate soil moisture profile access tube. If plots were not located near transect points, access tubes were installed within proximity of the incubation plots (Figures 3.1; 3.2). Physical soil characteristics were measured near each of the incubation plots, where measurements of bulk density (g cm⁻³), organic matter content (%), pH, average plant root to shoot ratio (g g⁻¹) and unsaturated surface soil moisture absorption (mm hr⁻¹) were completed near each of the incubation plots. Organic matter and bulk density were measured using soil core replicates (top 10 cm). Organic matter content was estimated using loss on ignition (LOI) (Heiri et al., 2001). Bulk density was measured using subsamples (100 g)

placed in a drying oven for a minimum 48 hours at 70 °C, where they were re-weighed following the drying process (McMillan et al., 2007). In field unsaturated surface soil moisture absorption (SSMA) measurements were conducted using the single ring infiltration tests (Negley and Eshleman, 2006; USDA, 1999). Excess from the core samples were used to assess the pH by forming a 1:2 slurry solution of soil to deionized water. Measurements were recorded with HI-98139 Pocket EC/TDS and pH Tester (HANNA instruments ©) in triplicates.

Root to Shoot ratio (R/S ratio) was assessed using triplicated rectangular (20x50 cm) quadrats, randomly placed around the incubation plots. Live above ground biomass was removed at the surface of the cover soil and placed within a paper bag, where samples would later be dried in an oven. Prior to removing below ground biomass, surveys were conducted to identify the average root depth (~20 cm). Two cores (860 cm³ per core) were selected per quadrat and average below ground biomass was then applied to the entirety of the quadrats volume (20000 cm³). Below ground biomass was also placed in paper bags. Methodology from Ravindranath and Ostwald (2008) was used to determine proper drying and weighing of the samples. It is important to note that plots did not include planted tree and shrub species and only forbs and grasses were selected for destructive sampling.

3.2.3 Soil chemical properties

Soil chemical composition was assessed using Plant Root Simulator (PRSTM) probes (Western Ag innovations Inc., Saskatoon, Saskatchewan) incubated in triplicates. Plant Root Simulators are ion membranes embedded in a plastic frame, which can be used to quantify soil nutrient availability under various field conditions and have been regularly used in characterizing ion availability within reclaimed forested uplands (Dimitriu et al., 2010; Jamro et al., 2014; MacKenzie and Quideau, 2010). Nutrient supply estimates (μ g 10cm⁻² incubation periods⁻¹) obtained by the probes combine the effect of soil temperature and moisture on nutrient fluxes during the incubation period (Huang and Schoenau, 1997; Nwaishi, 2016). The resin membranes are used in pairs (anion/cation), where the positively charged membrane simultaneously attracts and adsorbs all negatively charged anions such as NO₃⁻, phosphate (H₂PO₄⁻, HPO₄²⁻), and sulphate (SO₄²⁻), while the negatively charged membrane attracts and adsorbs all positively charged cations such as ammonium (NH₄⁺), potassium (K⁺), calcium (Ca²⁺), and magnesium (Mg²⁺). Once the burial periods were complete, the probes were removed from the soil and washed with deionized water to remove soil particles, placed in a Ziploc bag, and stored in the refrigerator at 4 °C until they were analyzed at Western Ag Innovations Inc., for elution with 0.5M HCl and subsequent nutrient analysis. Ammonium (NH₄⁺), nitrate (NO₃⁻) and phosphate (PO4³⁻) were analysed colourimetrically using an [Autoanalyzer III] (Brand and Lubbe Inc., Buffalo, N.Y., USA), while potassium (K⁺), SO₄²⁻, Ca²⁺, Mg²⁺, aluminum (Al³⁺), Iron (Fe³⁺), manganese (Mn²⁺), copper (Cu²⁺), Zinc (Zn²⁺) and Boron (B³⁺) analysis was conducted using [ICP spectroscopy] (PerkinElmer Optima 3000-DV, PerkinElmer Inc., Shelton, CT).

3.2.4 Surface flushing of major nutrients

The mobility of major nutrients such as NO₃⁻, NH₄⁺ and SRP following storm events (\geq 5mm of rain) was conducted using V-notched flumes constructed near the topographic lows of the individual reclaimed unit. The flumes were 2 m wide built from aluminum rain gutters, which guided surface runoff into sample collectors covered with polyethylene tarps to prevent direct rain water from entering the samples. Screening was placed directly in front of the sample collectors to limit the accumulation of sediments. Samples were collected immediately following storm events. Samples analyzed for NO₃⁻, NH₄⁺ and SRP were refrigerated at 4 °C and filtered within 24 hours using [SC0601C FlipMate 0.45 um PES] (Delta Scientific ©) and frozen until ready for laboratory analysis. Nutrient concentrations within the runoff samples were determined using colorimetric analyses in the Biogeochemistry Lab at the University of Waterloo [Bran Luebbe AA3, Seal Analytical, Seattle, USA, Methods G-102-93 (NH₄⁺), G-109-94 (NO₃⁻), G-103-93 (SRP)].

3.2.5 Vegetation surveys and analysis

Vegetation surveys were conducted from late-July to early-August within each of the incubation plots (2m x 2m) as well as near each of the soil moisture profile access tubes. Species were individually identified (Johnson et al., 1995) and organized into classes of 5% (0-5; 5-10; 10-15...). For statistical purposes, the classes were later converted to exact values (i.e. 0-5%=2.5%; 5-10%=7.5 %...). The proportions were transformed using an arcsine square root transformation (McCune and Grace, 2002). Species located in less than 10% of the 19 plots were removed from the data set to limit noise, as this often enhances the detection of relationships between community composition and environmental factors (McCune and Grace, 2002).
Unknown species were removed from the data collection, most of which were a single specimen. Measurements of alpha (α), beta (β_w) and gamma (Υ) diversity followed where: α represents average plot species diversity, β_w the amount of compositional variation between all the sampling units and Υ the landscape scaled diversity (McCune and Grace, 2002). When $\beta_w=0$, all the sampling units are identical in terms of species presence. In terms of field data, $\beta_w<1$ is rather low (homogeneous vegetation distribution) and $\beta_w>5$ is considered "high" (multiple distinctive communities) (McCune and Grace, 2002). Shannon-Wiener's measurement of diversity (H') was calculated to measure evenness (J), which calculates how equal a community is numerically. When all species are evenly distributed, J=1 (McCune and Grace, 2002).

Species were then classified relative to specific lifecycle characteristics. Vegetation was first organized by origin (i.e. planted, native, non-native). *Planted* species refer specifically to saplings composing an undisturbed mature boreal forest canopy (e.g. black spruce, white spruce, jack pine, tamarack, willow, dwarf birch, river alder, poplar and aspen) and not species planted for erosion control (e.g., barley (*Hordeum jubateum*)). Flora was then categorised relative to major functional community groups (i.e. forbs, graminoids, shrubs and trees). Classification was achieved using the Guidelines for Reclamation to Forest Vegetation in the Athabasca Oil Sands Region (Alberta Environment, 2009), site specific re-vegetation information (Daly et al., 2012; Unpublished data, 2013) and the Oil Sands Research and Information Network (OSRIN) Boreal Plant Species for Reclamation of Athabasca Oil Sands Disturbances (Smreciu et al., 2013).

3.2.6 Statistical analysis

All statistical analyses were performed with R \bigcirc (R Development Core Team, 2015). Prior to analyses, the raw field data was assessed for normality using the Shapiro-Wilks test (p>0.05) ("Shapiro.test" stats package R.3.2.3). Data that did not meet the criteria of normality were transformed using general relativization followed by arcsin squareroot transformation (Rooney and Bayley, 2011; Rowland et al., 2009; Turcotte et al., 2009). Transformation was not effective on all variables; therefore, non-parametric tests were used for the analyses. Specific details of the usage of statistical tests are available within the following Chapters (4 & 5), this section is for stating redundant information applicable to both research questions. Chapter 4: Assessing spatial and temporal hydrogeochemical characteristics during the growing season of a forest upland undergoing reclamation on a post-mining landscape near Fort McMurray, Alberta.

4.1 Introduction

Forested uplands are a crucial component of the Western Boreal Forest (WBF), as they convey multiple hydrogeological functions (Devito et al., 2005; Ketcheson et al., 2016). These forested soils are categorized by a low relief (~7-12%) with deep glaciated substrates varying in storage and transmission properties (Devito et al., 2005; Rowland et al., 2009). These hydrophysical properties, and the sub-humid climate of the region, influence how moisture and solutes are re-distributed within the landscape, where wetlands and open-water ponds interact with the uplands, ensuring resilience during periodic stresses (*i.e.* drought) and re-distributing moisture during wet years (Johnson and Miyanishi, 2008). Differences in topographic positioning and soil substrate generally form a gradient favoring the accumulation of moisture and solutes towards the lower portions of the land units, influencing biogeochemical cycling and vegetation establishment (Dimitriu and Grayston, 2010). Despite this gradient, surface hydrological re-distribution is rare during the growing season, as deep unsaturated zone storage, high vegetation water demands and vertical flow dominate over lateral flow (Devito et al., 2005; Johnson and Miyanishi, 2008).

Currently, the Athabasca Oil Sands Region (AOSR) of the WBF is being heavily disturbed by industrial development for bituminious oil sand through *in-situ* recovery and open pit mining (Rooney & Bayley 2012). The energy companies are required by the Alberta government regulations to reclaim all disturbed land back to equivalent capability (Alberta Government, 2000). In order to fulfill this obligation, entire watersheds consisting of forested uplands, open bodies of water and peatlands are being engineered (Daly et al., 2012; Pollard et al., 2012; Price et al., 2010). The concept is developed around the belief that re-initiating pre-disturbed hydrogeochemical connectivity amongst individual land units will allow for the formation of self-sustaining fen peatlands, something which has yet to have been successfully accomplished in the AOSR (Ketcheson et al., 2016a). Although forested upland reclamation has been heavily studied and somewhat successful in the past, these land units only represent a small portion (~23%) of the pre-disturbed landscape, where fen peatlands dominate (~45%) (Audet et

al., 2015; Price et al., 2010; Rooney et al., 2012). Furthermore, the majority of studies have been conducted on isolated land units where vertical flow and aquifer recharge are discouraged (Daly et al., 2012; Pollard et al., 2012).

With the new incentives implemented for landscape reclamation, it is important that upland management and monitoring strategies are modified. Upland reclamation consists of filling the excavated pits with mine waste such as unsuitable overburden and/ or tailing sands, capped with salvaged organic amendments such as Forest Floor Material (FFM) or Peat-Mineral Mix (PMM) (Hemstock et al., 2010; MacKenzie and Quideau, 2010; Naeth et al., 2012). The capping layer serves as a substrate for plant colonization, while also limiting alkaline-sodic mine residues from penetrating to the rooting zone, discouraging the vertical flow of moisture and nutrients through the formation of a capillary barrier (Carey, 2008; Jung et al., 2014). Although favorable for isolated upland systems, the concept of hydrogeochemical connectivity amongst land units means reclaimed uplands are likely to include the addition of atmospherically exposed recharge basins to promote groundwater flow (Kessel, 2016; Ketcheson and Price, 2016c). Constructed uplands differ from natural systems not only in terms of hydrogeological storage capacities, but their surface soils vary considerably in terms of vegetation, biogeochemical cycling and hydrophysical properties (Dimitriu et al., 2010; Macdonald et al., 2015b; MacKenzie and Quideau, 2012). Studies have found that similarities between natural uplands were dependant on the salvaging and placement of the material used in reclamation. For instance, when comparing land reclaimed with FFM to that of PMM, FFM was shown to be more similar to that of pristine boreal forest soils. Meanwhile PMM, was shown to have greater moisture absorption capabilities, which is imperative to sapling survival within the first few years (Mackenzie et al., 2014; Pinno and Errington, 2015; Schott et al., 2015). PMM is also much more readily available for reclamation as peatlands dominate the pre-disturbed landscape (Mackenzie et al., 2014). Regardless of amendment type, the mixing and degradation of the salvaged material during placement disturbs pre-excavated soil structure, presenting greater heterogeneity within the reclaimed system (Ketcheson and Price, 2016c; Macdonald et al., 2015b; Mackenzie, 2011). Additionally, newly reclaimed soils possess hydrophobic properties and limited water storage capacities, leading to runoff and the erosion of unconsolidated particles downslope (Keshta et al., 2010; Ketcheson and Price, 2016a).

Because runoff is rare during the growing season within the undistrurbed forests of the WBF, the infrequent near-surface flushing of soils leads to the accumlation of major nutrients such as Soluble Reactive Phosphorus (SRP) and inorganic nitrogen (i.e. NO_3^-, NH_4^+) (Macrae et al., 2006; 2005). Phosphorus mobility is primarily governed through erosion due to it's strong affinity for the mineral forest soils (Kreutzweiser et al., 2008). Meanwhile, inorganic nitrogen mobility is strongly influenced by biogeochemical processes (i.e. nitrification), and groundwater flow, given the dominance of vertical flow and unsaturated storage (Macrae et al., 2006; 2005). As upland reclamation aims to return equivalent capability to the disturbed land, it is important to understand how: the heterogeneity of the soils, erosional processes and incorporation of groundwater recharge promoting structures will influence the hydrophysical properties responsible for the formation of a moisture-nutrient gradient influencing the establishment of boreal forest communities.

This study explores the spatial and temporal properties during the growing season of an upland undergoing reclamation to determine how topographic position and heterogeneity of the soil influences nutrient mobility. The specific objectives are: 1) Characterise spatial hydrophysical properties of the reclaimed upland relative to topographic positioning and substrate cover; 2) determine how the hydrophysical properties affect spatial and temporal physicochemical soil properties; and 3) identify how functional vegetation communities respond to the developing soils' moisture-nutrient gradient. It is hypothesized that: 1) hydrophysical properties will not be influenced by a topographic position as the reclaimed soil is still too undeveloped; and 2) mobile ions will accumulate in the lower topographic positions despite the lack of moisture content, due to downhill transport and repeated near surface flushing events.

4.2 Materials and methods

4.2.1 Study site

This study was conducted on an upland-fen system surrounded by three previously reclaimed hillslopes and one natural hillslope. The 7.7 ha forested upland consists mostly of FFM, with strategically placed areas of peat substrate (Figure 3.1). A 2.2 ha transition zone is incorporated at the toe of the low-relief (2-3%) upland, where pockets of peat substrate are located. The remainder of the upland (5.5 ha) is designed as a combination of hummocks and hollows aimed to reduced excessive runoff while promoting infiltration and groundwater

recharge. The surface of the FFM was tilled in 2013 to increase recharge to the upland aquifer. An experimental peat-lined basin (~0.2 ha) occupies the southern portion of the upland. Planting was initiated over the summer of 2013 and continued throughout the study period. Approximately 10,188 tree saplings were planted on June 21st 2015 with the addition of 10 g continuum TM RT (18:9:9:9(S)) controlled release fertilizer (CRF) biodegradable paper packets, applied to each individual sapling at an approximate rate of 17,56 kg/ha. Vegetation surveys were conducted throughout the upland during peak growing season. Currently, the vegetation canopy is dominated by forbs and grasses (e.g., *Sonchus arvensis, Agropyron tracycaulum*).

4.2.2 Hydrophysical properties

Transects running north- south (A-A'; B-B') and east-west (C-C'; D-D') were installed throughout the upland (Figure 3.1). In total, 20 soil moisture access tubes were used to measure volumetric water content (VWC) at various intervals. These values were used within the transect to determine total volume of water (TSW) at 15 and 35 cm. At each access tube, additional measurements of surface soil moisture, surface temperature and pore water connectivity (ECp) were recorded. Transect measurements were conducted approximately twice a week. Additionally, measurements of unsaturated surface soil moisture absorption (SSMA) (mm⁻¹ hr⁻¹) were conducted near each of the soil moisture access tubes. Three soil pit moisture stations were located along topographic positions (. i.e. High, Mid, Low). These stations were used to determine the infiltration capacity of the soil, displaying storm events where precipitation and antecedent moisture conditions led to infiltration into the tailing sand aquifer.

4.2.3 Physicochemical soil properties

Nine incubation plots were located throughout the constructed upland along three northsouth transects, following a topographic gradient (Figure 3.1). Quantification of hydrophysical properties were assessed from the nearest immediate soil moisture profile access tube. If plots were not located near the transect points, access tubes were installed within proximity. Physical soil characteristics were determined near each of the incubation plots, where measurements of bulk density (g^{-1} cm⁻³), organic matter content (%), pH, average plant root to shoot ratio ($g^{-1} g^{-1}$) were recorded. Soil chemical composition was assessed using Plant Root Simulator (PRSTM) probes in triplicates within each of the nine incubation plots. Three 21-day incubation periods (i.e. Early, Middle, Late growing season) determined the temporal variability during the growing season. The PRS probes adsorb anions such as: NO_3^- , phosphate (H₂PO₄⁻, HPO₄²⁻), and sulphate (SO₄²⁻), and cations such as ammonium (NH₄⁺), potassium (K⁺), calcium (Ca²⁺), magnesium (Mg²⁺), aluminum (Al³⁺), iron (Fe³⁺), manganese (Mn²⁺), copper (Cu²⁺), Zinc (Zn²⁺) and Boron (B³⁺).

4.2.4 Surface flushing and vertical flow of major nutrients

Three V-notched flumes located at the topographic lows of the upland collected runoff samples following precipitation events measuring concentrations of SRP and dissolved inorganic nitrogen (i.e. NO_3^- , NH_4^+). The samples were used to compare nutrient concentrations in runoff with temporal fluxes of ion availability detected with the PRS probes. A network of wells and piezometers was previously installed throughout the upland to measure groundwater fluxes within the constructed aquifer (see Kessel 2016 for additional information). Incubation plots were located relatively near these wells to detect if surface ion availability was linked to groundwater composition. Samples were collected four times throughout the duration for of the study period for SRP, NO_3^- and NH_4^+ . Samples were also collected from the recharge basins to determine if these structures encouraged leaching. All samples were stored at 4 °C until being passed through a 0.45 µm cellulose nitrate filter within 24 hours of being retrieved. Samples were frozen then sent to the Biotron Experimental Climate Change Research Facility at Western University, London, ON. Cation (NH_4^+) and anion (PO_4^{2-} , NO_3^-) concentrations were determined by a Dionex ICS-1600 Method EPA 300.0 with AS-DV auto-sampler to an analytical precision of $\pm 1.0 \text{ mg L}^{-1}$.

4.2.5 Statistical methods

To assess spatial (i.e. high, mid, low-slope) and temporal (i.e. early, middle, late) variability in hydrophysical properties, a Scheirer-Ray Hare extension of the Kruskal-Wallis test was used as a nonparametric equivalent to a two-way analysis of variance (ANOVA) (Dytham, 2011), followed by a post-hoc analysis (function "kruskal", package "agricolae). A Mann-Whitney U test was used to measure the degree of variability in hydrophysical properties between both cover substrates (i.e. FFM, Peat). Permutation multivariate analysis of variance

(perMANOVA) (function "Adonis", package "Vegan") was used to determine significant spatial and temporal variability in ion availability. This was followed by a multi-response permutation procedure (MRPP) (function "mrpp", package "Vegan"), which indicated when and where these significant variations occurred. Indicator species analysis (function "indicators", package "indicspecies") was used to complete the analysis by identifying the incubation plots that were significantly different from one another, and the ion concentration that caused the variation. A linear mixed effect model (function "lme", package "nlme") with repeated measures was used to determine if SRP and TIN (NH₄⁺, NO₃⁻) were influenced by random (i.e. Surface soil moisture, TSW_{15} , TSW_{35}) and fixed factors (i.e. cover substrate, time of season) as a prediction of their susceptibility to surface flushing. A similar model was also applied to determine if groundwater samples collected were influenced by both random (i.e. pH, Watertable level, recent precipitation, surface ion availability) and fixed factors (location, time of season, overlying substrate). Spearman's rank correlation coefficient was used to determine which ions and edaphic characteristics correlated to one another to predict potential models. The models with the highest coefficient of determination (R^2) and the lowest Akaike Information Criterion (AIC) were selected. The accepted significance level for all statistical tests was $p \le 0.05$. Ordination (NMDS) was used to display correlations between hydrophysical properties and chemical distribution, as well as the combination of both nutrients and moisture on vegetation community establishment.

4.3 Results

4.3.1 Hydrophysical properties

No spatial topographic gradient was detected throughout the upland for either surface moisture (p=0.5), TSW₁₅ (p=0.42) or TSW₃₅ (p=0.02; however mid-slope had the greatest moisture) (Figure 4.1) Cover substrate did significantly influence surface soil moisture. Peat was found to contain greater VWC than that of the FFM (p=0.03). However, this was not noticed for total volume of water within the soil column (Figure 4.1b-c). Unsaturated surface soil moisture absorption (SSMA) was found to be heterogeneous throughout the upland and significantly greater in peat (p<0.001) (Table 4.1).



Figure 4-1 Topographic north-south moisture transects (A-A'; B-B'), (a) Seasonal averages of surface soil moisture content (%) (0-7cm), collected using a Wet-2 wet sensor, (b) Total Soil Water (TSW₁₅) within the first 15cm of the soil profile, (mm), converted from VWC measured from PR2 soil profile access tubes, (c) TSW₃₅ **The greater the size of the soil moisture plot, the greater the moisture content*.

Infiltration was variable throughout the upland. Major infiltration and groundwater recharge rarely occurred for both the transition and mid-slope sections of the upland during the 2015 research season (see Kessel (2016)), indicating that surface and sub-surface runoff were considerable factors in these areas following storm events, especially near the transition zone (Figure 4.2b). However, infiltration did occur rather frequently within the higher sloped area of the upland (Figure 4.2d). Surface FFM layers (5 and 15 cm) responded often to precipitation events, however the base (25 cm) and tailing sands top (~30 cm) solely responded to precipitation events when antecedent moisture conditions and the magnitude of the events were optimal.



Figure 4-2 May-August 2015 precipitation (a) and Volumetric Water Content (VWC) of the FFM cover substrate (grey) and tailing sands top layer (black) of the soil profile recorded at three different soil moisture stations, transition zone (low-slope) (b), mid-slope (c) and high-slope (d). The base FFM layer for both the transition and high-slope was located at~25cm. The base of the FFM layer at the mid-slope was located \sim 40cm.

4.3.2 Physicochemical properties

The soil displayed spatial variability in physical properties throughout the constructed site (Table 4.1). Bulk density (ρ_b) was found to be significantly higher in the mid-slope (p=0.02) than the other positions, and higher within the FFM than the peat substrate (p<0.001). These findings coincide with R/S ratio being significantly lower (p=0.008) at the mid-slope position

and within the FFM (p<0.001). Organic matter did not demonstrate any significant difference between slope positions. Organic matter content (p<0.001) was higher within the peat substrate than FFM. pH was found to be more neutral at the low slope (p=0.025) and within peat (p=0.03), while more acidic at the mid-slope as well as the FFM.

Table 4-1 Soil characteristics (mean \pm se) relative to topographic position and substrate showing root to shoot ratio (R/S), bulk density (ρ_b), unsaturated surface soil moisture absorption (SSMA), organic matter (%) and pH. Characters: a, ab, b are used to indicate significant differences (Kruskal test) among treatments (comparing slope position and substrate separately).

| Parameters | | Slope position | Substrate | | |
|--|----------------------------|------------------------------|----------------------------|------------------------------|----------------------------|
| | High-Slope | Mid-Slope | Low-Slope | FFM | Peat |
| $R/S(\sigma/\sigma)$ | $\frac{n=3}{1.95(0.29)^a}$ | $\frac{n=3}{0.32(0.01)^{b}}$ | $\frac{n=3}{0.95(0.02)^a}$ | $\frac{n=0}{0.86(0.01)^{b}}$ | $\frac{n=3}{1.18(0.17)^a}$ |
| | 1.95(0.29) | 0.52(0.01) | 0.95(0.02) | 0.00(0.01) | 1.10(0.17) |
| $\boldsymbol{\rho}_b (\mathrm{g}\mathrm{cm}^{-3})$ | $1.26(0.03)^{b}$ | $1.51(0.01)^{a}$ | $1.26(0.4)^{b}$ | $1.42(0.02)^{a}$ | $1.18(0.04)^{b}$ |
| SMMA (mm ^{-hr}) | 48.00(19.17) ^a | 37.65(9.81) ^a | 39.19(14.72) ^a | 37.33(9.81) ^b | 49.49(13.75) ^a |
| Organic matter (%) | 18.83(0.87) ^a | 19.5(0.59) ^a | 18.33(0.97) ^a | 17.94(0.54) ^b | 22.04(0.68) ^a |
| pH | 6.43(0.18) ^{ab} | 6.13(0.04) ^b | 6.5(0.06) ^a | $6.22(0.65)^{b}$ | 6.66(0.13) ^a |

Soil chemical properties varied spatially and temporally. Topographic position (p=0.003) contributed to this spatial variability, however the low coefficient of determination (R^2 =0.11) implies that factors outside of topography were likely associated with the observed spatial heterogeneity, as was the case with the hydrophysical properties. Results from the MRPP (Table 4.2) demonstrated that over the course of the research season, the greatest degree of separation occurred between the mid and low-slope positions (T=-12.77). No significant difference was observed between high and low-slope incubated areas (p=0.301), where within group replicates had very minimal group homogeneity (A=0.014). Subsequent cluster and indicator species analysis found three significantly different groups of incubation plots, where Ca^{2+} , Mg^{2+} , B^{3+} and SO_4^{-2} corresponded strongly with group 1, while K⁺ and SRP corresponded strongly to group 2. A third group was significantly different from the other two but did not possess specific indicators. Group 1 corresponded to all plots incubated near peat, meanwhile Group 2 and 3 correspond to plots located with FFM.

Table 4-2 Multi-Response Permutation Procedure (MRPP) results for ion availability relative to slope position and time of season. Results demonstrate the separation (T) between slope positions and separation (A) amongst same-slope replicates. *Bold numbers represent significant differences (p > 0.05).

| | Time of Season | | | | | | | |
|-----------------------|-------------------|-------|--------|-------|--------|-------|--------|-------|
| Position | Throughout season | | Early | | Middle | | Late | |
| | Т | А | Т | А | Т | А | Т | А |
| High vs Mid vs Low | -3.75* | 0.083 | -4.06* | 0.098 | -4.73* | 0.091 | -2.47* | 0.065 |
| High vs Mid | -3.72* | 0.036 | -3.47* | 0.079 | -3.06 | 0.043 | -0.87 | 0.039 |
| High vs Low | -0.47 | 0.014 | -0.74 | 0.007 | -2.92* | 0.077 | -0.82 | 0.016 |
| Mid vs Low | -12.77* | 0.111 | -5.35* | 0.164 | -4.63* | 0.096 | -4.27* | 0.11 |

Temporal variability did not increase throughout the growing season (Table 4.2). Among the slope positions, the greatest degree of separation occurred during the middle of the growing season (T=-4.73, A=0.098)), where mid and low-slope demonstrated the greatest separation (T=-4.63, A=0.096). The lowest amount of spatial variability occurred during the late-season (T=-2.47, A=0.065), where neither mid-(T=-0.87, A=0.039, p=0.15) or low-slope (T=-0.82, A=0.016, p=0.23) demonstrated significant differences with the high-slope, attributed the very low within group homogeneity.

Ordination results demonstrate that, while topographic position did not influence hydrophysical properties, the hydrophysical properties influenced nutrient availability within the cover soil (Figure 4.3). Throughout the entirety of the growing season (Figure 4.3a), substrate cover influenced ion availability (p<0.001, R^2 = 0.25), where ions such as Ca²⁺, Mg²⁺, and SO₄-²⁻ were strongly correlated with peat substrates, as confirmed by the indicator species analysis. FFM was found to show greater availability in SRP, K⁺, NO₃⁻, Mn and NH₄⁺. NO₃⁻ was strongly influenced by areas of increased temperature (p<0.001, R²=0.2), mostly in the mid-slope region. pH (p<0.001, R²=0.34) and soil moisture (p<0.001, R²=0.30) were correlated with incubation plots located near peat substrates. Early in the season, hydrophysical and physicochemical properties of the soil had the greatest influence on ion availability (Figure 4.3b). Plant root development is strongly correlated with plots incubated near elevated available SRP and K⁺ located near the high slope. Similar trends were noticed in the mid-season (Figure 4.3c), however the influence of soil properties diminished as the growing season continued (Figure 4.3d).







Figure 4-3 NMDS ordination plots demonstrating the degree of separation amongst plots in relation to ion availability. Vectors represent strong correlations (p<0.05) between environmental variables and ion availability. (T)-values represent the degree of separation amongst slope positions. (A)-value represents same-slope replicate homogeneity and (P)-value demonstrates the significance of the separation. (a) Throughout the research season (DOY 141-220), (b) Early-season (DOY 141-163), (c) Mid-season (DOY 170-191) and (d) Late season (DOY 198-220). K represents the number of dimensions for best fit and the stress value describes the fit of the ordination model.

4.3.3 Surface flushing and vertical flow of major nutrients

Soluble Reactive Phosphorus (SRP) demonstrated a strong potential for near-surface soil flushing out of the constructed upland. The linear mixed effect model (R²=0.58) demonstrated that, though minimal, SRP was positively influenced by TSW_{15} (F= 4.35; p=0.04), as well as time of season (F=41.09; p<0.001) and FFM substrate (F=13.53; p=0.007). These results coincide with the samples gathered from the runoff collectors (Figure 4.4a), where samples demonstrated elevated concentrations regardless of surface ion availability (Figure 4.4b). Contrarily, Total Inorganic Nitrogen demonstrated much lesser susceptibility towards surface runoff. No linear mixed effect model was proven to be significantly influenced by soil moisture, neither for NH_4^+ or NO_3^- . Furthermore, DIN runoff samples responded strongly to TIN availability. Following the application of fertilizer (DOY 172), both SRP and TIN availability increased considerably (114 and 82% respectively), which was further represented by the runoff samples (Figure 4.4a). Late-season however, TIN availability decreased throughout the system to values similar to that of earlier in the season (10.99 and 7.49 μ g/10cm²). This decrease was also detected in the runoff samples (Figure 4.4b). Following the application of fertilizer, N: P ratios decreased for the remainder of the research season (early-season, 10.43; mid-season, 7.81; end of the growing season, 2.62).



Figure 4-4 (a) Dissolved Inorganic Nitrogen (DIN) and Soluble Reactive Phosphorus (SRP) (mean \pm se) concentrations collected from runoff samples following major storm events (>5mm). *Fertilizer was applied to the site on June 21st 2015 (DOY 172). (b) Inorganic Nitrogen (TIN) and Soluble Reactive Phosphorus (SRP) (mean \pm se) seasonal supply rates within the reclaimed upland soils. Characters: a,b, are used to indicate significant differences (Kruskal) throughout the season.

Groundwater samples demonstrated that groundwater chemistry was not influenced by the availability of ions located within the near surface proximity of the sampling well. NO_3^{-1} groundwater concentration ($R^2=0.38$) demonstrated that overall upland surface NO₃⁻ availability (F=4.77, p=0.029), time of season (F=2.9, p=0.05), overlying substrate (F=22.21, p<0.001), precipitation (F=5.04, p=0.028) and the interaction between precipitation and overlying substrate (F=13.12, p<0.001) best explained its concentration (Figure 4.5). The best fit for NH_4^+ (R²=0.35) was found to influenced by pH (F=7.57, p=0.007), electrical conductivity (F=13.19, p=0.006), time of season (F=11.68, p=0.001) and the interaction between precipitation and overlying substrate (F=10.11, p=0.035) (Figure 4.5). Contrarily, no model could quantify groundwater SRP as samples often fell below detection limits. Spearman's rank correlation (APPENDIX) demonstrated that Soluble Reactive Phosphorus availability was negatively correlated with elements such as Ca^{2+} (p=0.044, R²=-0.36), Fe³⁺ (p=0.007, R²=-0.4) and SO₄²⁻ (p<0.001, R²= -0.47). These elements are highly concentrated in post-mined overburden materials and can form insoluble precipitates, immobilizing phosphates. NO₃⁻ concentration was greatest following large precipitation events and this was only witnessed within the recharge basins, particularly the east basin (Figure 3.1). Conversely, NH₄⁺ concentrations decreased following precipitation events

within these recharge basins and increase during the drier sampling periods. Nitrate concentrations were consistently minimal within the groundwater samples taken where a capping substrate was located. Dissolved Inorganic Nitrogen content within the groundwater remained relatively uniform throughout all four of the sampling periods (5.5, 5.8, 6.4, and 5.9 mg ml⁻¹, respectively).



Figure 4-5 (a) 2015 precipitation, (b) Dissolved Inorganic Nitrogen (DIN) concentrations collected from four separate sampling periods. Recharge Basins (RB) are displaying elevated NO₃ concentrations when sampling followed considerable (>10mm) precipitation events. Upland (UPL) groundwater is primarily dominated by NH_4^+ . Surface NO₃ was positively related to groundwater concentrations (*Surface NO₃ availability for DOY 197 was assumed to be mean between middle and late PRS incubation periods). Characters: a,ab,b, represent significant differences amongst sampling periods and locations. Nitrate represents the first character, followed by ammonium (NO₃, NH_4^+).

4.3.4 Vegetation functional group colonization

The β diversity of the constructed system was considerably low (β_w = 1.54). Although hydrophysical and physicochemical properties were not influenced by a topographic control,

vegetation colonization was influenced by the distinctive soil properties (Figure 4.6). Figure 4.6a demonstrates that forb colonization was correlated with areas of higher temperatures (p=0.029, R^2 = 0.70), while grasses demonstrated a relationship with SRP (p=0.023, R^2 =0.73). Tree sapling colonization and survival was influenced by areas of elevated unsaturated surface soil moisture absorption (SSMA). Although ordination did not find it to be significant (p=0.2), the soil properties demonstrating relationships with tree sapling presence (Ca⁺², B⁺, Mg²⁺) are all indicative of peat substrate. Figure 4.7b demonstrates that non-native species were strongly correlated with areas of higher temperature p=0.007, R²=0.78) and bulk density (p=0.05, R²=064). Native species were correlated with areas of elevated SRP (p<0.00,1 R²=0.76) and K⁺ (p=0.05, R²=0.58) availability. Planted species were significantly greater within peat substrate (p=0.024, R²=0.35), and in areas of higher unsaturated surface soil moisture adsorption (p=0.036, R²=0.68).





Figure 4-6 NMDS ordination plots demonstrating relationships between environmental vectors and functional vegetation groups. Vectors represent strong correlations (p<0.05). (a) forbs, graminoids, shrubs and trees. (b) native, non-native and planted species. K represents the number of dimensions for best fit and the stress value describes the fit of the ordination model.

4.4 Discussion

4.4.1 Hydrophysical properties

Results shown demonstrate a lack of topographic influence on moisture content and nutrient availability throughout the upland 3-years following placement. These findings are similar to the results from other upland reclamation studies (Leatherdale, 2008; MacKenzie and Quideau, 2010). The handling and placement of the FFM by large operational equipment blends the original soil structure of the pre-disturbed LFH layers and mixes it as it is re-distributed throughout the constructed system (Mackenzie, 2011). Homogenizing pre-disturbed LFH layers suggests that reclaimed soils will need time and pedogenic processes such as freeze thaw, organic matter accumulation and plant root development before macropores creating preferential

pathways allow for a moisture gradient to develop (Guebert and Gardner, 2001; Ketcheson et Price, 2016c). Furthermore, the tillage performed perpendicular to the topographic gradient in the summer of 2013, formed various microtopographical features, which are potential areas of water accumulation following storm events. Additionally, areas of hummocks and hollows incorporated within the design of the constructed watershed influenced areas of preferential flow directing water to groundwater recharge basins (Ketcheson, 2016c; Kessel, 2016).

Textural interfaces within the constructed system led to infrequent water percolation into the tailing sands aquifer. The placement of a fine-grained material (FFM) over a coarser-grained material (tailing sands), lead to the formation of a capillary barrier discouraging losses from precipitation events below the FFM. These textural discontinuities are purposely implemented on post-mined landscapes to limit any vertical seepage of soil moisture and nutrients below the cover soil while impeding highly sodic water from rising below the tailing sands (Huang et al., 2013; Jung et al., 2014; Naeth et al., 2011). However, these interfaces can also inhibit soil development by limiting percolation through the soil profile, complicating the re-establishment of hydrogeochemical connectivity (Vitt and Ghatti, 2012)

4.4.2 Physicochemical properties

The lack of a hydrophysical gradient related to topographic controls impeded the formation of a moisture-nutrient gradient. Although lack of soil structure and the formation of microtopographical features was previously discussed, nutrient sinks such as microorganisms, metal oxides $(A1^{+3}, Fe^{+3}, Ca^{+2})$ and vegetation could be immobilizing available ions, reducing the detection of any topographic gradient (Hangs et al., 2004; Leatherdale, 2008; McMillan et al., 2007). Furthermore, the high intra-group variability of ion availability among slope position (A-value) demonstrates that the heterogeneity of the cover soil could be restricting the detection of a topographical influence on ion availability (Leatherdale, 2008).

Although significant differences occurred even among FFM incubation plots, substrate cover proved to be a considerable influence on ion availability. Peat substrate plots were found to possess higher availability of Ca^{+2} , Mg^{+2} and SO_4^{-2} , while K⁺ and SRP proved to be indicative of FFM plots (Figure 4.3). Elevated levels of sulfate found in the peat substrates are attributed to either atmospheric deposition from the surrounding industrial development, or to the drainage of

the donor fen prior to harvest, which would disrupt the reduction of sulfates from sulfur-reducing bacteria due to a shift from anaerobic to aerobic conditions (Nwaishi et al., 2016).

Previous work has confirmed that FFM has higher K⁺ and SRP availability than peat, due to such substrates having a more similar mycorrhizae fungi community to that of natural forest floors, which produce extracellular enzymes catalyzing phosphate and K⁺ mineralization (Brown and Naeth, 2014; Jamro et al., 2014). Furthermore, the elevated concentration of Ca²⁺ located in the peat substrate could be immobilizing phosphates by forming insoluble precipitates (Gurevitch et al, 2006). Although insignificant, FFM was generally found to contain considerably greater amounts of available TIN, especially within the mid-sloped areas where temperatures were considerably higher (Figure 4.3). Peat typically has higher C: N ratio and is therefore more difficult for microorganism to mineralise the organic matter into available nutrients (Mackenzie et al., 2014; McMillan et al., 2007). Furthermore, the microorganisms typically found in peat are subject to very different environmental conditions (i.e. anaerobic, acidic) than FFM, and therefore community structure takes longer to adapt to forest floor conditions (Jamro et al., 2014; MacKenzie and Quideau, 2012).

Spatial variability was detected even amongst FFM incubated plots, likely related to placement of the soil during the construction phase. Initial construction plans deemed 20 cm of FFM necessary as a cover soil to promote infiltration while maintaining adequate moisture content to promote vegetation growth (Daly et al., 2012; Price et al., 2010). The site-scale hydrological assessment (Ketcheson and Price, 2016c) demonstrated that the FFM was nearly twice that thickness in certain areas of the upland (~40cm). This excess amount of forest floor material implies that the quality of FFM is heterogeneous, which has been confirmed by both textural and physicochemical properties presented. Increased thickness of salvaged FFM reduces organic matter content and lowers initial nutrient availability, reducing the overall quality of the material (MacKenzie and Naeth, 2010; MacKenzie and Quidea, 2012; Rokich et al., 2000). Furthermore, near-surface tillage performed on the upland could also have affected spatial nutrient availability through changes in soil hydraulic, aeration and diffusive properties, increasing the lability of the organic matter, and thereby mobilizing many essential plant nutrients (Lipiec and Stepniewski, 1995; Das Gupta et al., 2015).

Temporal changes in chemical availability within the soil (Figure 4.4b) could have been linked to various biological and abiotic factors (e.g., microbial/ plant/ ion immobilization, soil

moisture, temperature) (Leatherdale, 2008; Nwaishi et al., 2015a). The high degree of variability was more likely caused by the application of CRFs, which are designed to gradually release ions to the soil matrix, as was determined to be influential with both the PRS probes and runoff samples. Nevertheless, determining precise release rates is rather inconsistent between soil types and the highest release rates are typically detected immediately upon application (Haase et al., 2007; Hangs et al., 2003a). A recent study by Sloan et al. (2016) found that although CRFs have the ability of greatly reducing overall fertilizer application in a post-mined landscape, the majority of applied nutrients were unaccounted for within the saplings and competing vegetation, likely attributing these loses to off-site movement (i.e. runoff and leaching).

4.4.3 Surface flushing and vertical flow of major nutrients

It was hypothesized that both SRP and TIN concentrations would be greatest at the lower portions of the upland slope due to surface flushing and erosion. However, results indicate that only SRP is susceptible to near-surface soil flushing following precipitation events. Firstly, SRP is positively correlated with areas of elevated soil moisture and, secondly, the continuously decreasing bio-available N: P ratio throughout the research season suggests that SRP is in surplus in the reclaimed soil, exceeding the soils exchange capacity. The responses from the runoff samples insinuate that, while TIN likely witnessed major off-site movement immediately following the application of CRF, immobilization from vegetation and microorganisms reduced the potential for off-site movement, as TIN became increasingly limited, as is regularly the case in reclaimed forest soils (Duan and Chang, 2015; Hemstock et al., 2010; McMillan et al., 2007). Furthermore, ordination (Figure 4.3) demonstrated that the main influence on the spatial distribution of NO_3^{-1} was elevated temperature, suggesting that mineralization is likely influencing NO₃⁻ availability. Additionally, ordination also demonstrates that SRP was positively related to areas with elevated R/S ratios, which is commonly a sign of nitrogen limitation, as the vegetation needs to invest its resources below ground to find additional sources of TIN (Gurevtich et al., 2006). Although TIN does not seem susceptible to near-surface soil flushing, results demonstrated that it might be vulnerable to vertical flow leaching. NO₃⁻ is the more mobile form of inorganic nitrogen and often a concern for groundwater pollution (Huang and Schoenau, 1997; Kreutzweiser et al., 2008). At the surface, NO₃⁻ was shown to be the dominant form of TIN, while NH₄⁺ dominated in the groundwater aquifer. Research has demonstrated that

the capillary barrier at the FFM/ Tailing sand interface can induce locally reduced (or anaerobic) conditions caused by slow water movement, favoring the accumulation of NH₄⁺ (Brady and Weil, 2008). Although NH₄⁺ is not very mobile, the hydrophobic properties of the tailing sands suggest that once moisture penetrates below the cover soil, water and ions leach into groundwater aquifers (Huang et al., 2011; Jung et al., 2014). This was observed in the groundwater data, where wells located within either FFM or peat substrates had minimal NO_3^{-1} concentrations and NH₄⁺ was the dominant inorganic form of nitrogen. Groundwater samples from wells where such a capillary barrier did not exist (i.e. recharge basins) had increased NO_3^{-1} concentrations when sampling followed recent precipitation events. Although NO₃ inputs could be related to the chemical composition of precipitation, only one recharge basin (. i.e. east basin) demonstrated considerable increases in NO_3^{-} . This recharge basin has often promoted enhanced groundwater recharge, attributed to the elevated runoff contributions for the south-east portion of the upland (Kessel, 2016; Ketcheson, 2015). This was further confirmed by the positive relationship between groundwater NO₃⁻ concentrations and overall surface availability. As with natural forested uplands, SRP did not seem susceptible to groundwater leaching, potentially due to the formation of insoluble precipitates within the upper subsoils (~20 to 50cm).

4.4.4 Vegetation functional group colonization

Vegetation communities demonstrated poor spatial heterogeneity, suggesting that environmental factors did not function as a major influence on community establishment, instead, the use of cover soil substrate seemed to have the strongest influence. Aggressive pioneer forbs and grasses are currently the dominant vegetation throughout the constructed upland. Pinno and Hawkes (2015) demonstrated that forbs and grasses, along with non-native species dominate reclaimed uplands for the first five years post-soil placement, gradually declining with developing canopy cover. Forb (e.g., *Sonchus arvensis*) establishment appeared to be related to areas of elevated soil temperature and bulk density, colonizing portions of the soil that are currently inhabitable for native shrub/ tree establishment or sapling survival. Graminoids (e.g., *Agropyron tracycaulum*) were more influenced by ion availability and have been known to out compete planted saplings for nutrients within reclaimed soils (Errington and Pinno, 2015a; Hangs et al., 2003b; Landhäusser and Lieffers, 1994).

Planted tree and shrub species are currently the least abundant groups throughout the upland. However, areas with peat substrate had higher sapling cover than FFM. Sapling survival rates studies on aspen have demonstrated that peat substrates outperformed FFM during the first few years following soil placement, likely attributed to the peat substrates higher moisture retaining abilities, nutrient adsorption capacities and decreased competition from a lesser viable seed bank (Pinno et al., 2012; Pinno and Errington, 2015; Schott et al., 2015). Within this constructed soil, FFM generally had higher N-P-K availability, implying that sapling survival is influenced by a moisture deficit.

Native species were dominant within the FFM comparatively to the peat substrates. Salvaged FFM is advantageous comparatively to peat as it possesses a more natural seedbank, typical to that of forested uplands and also contains greater densities of viable propagules (Archibald, 2014; Errington and Pinno, 2015b; Mackenzie and Naeth, 2010). Native species colonize reclaimed systems either through dispersal (. i.e. wind, animal) or viable propagule banks from the cover soil. Results demonstrated that natural species were influenced by ion availability (SRP, K^+), suggesting that colonization was induced by areas of the FFM exhibiting better quality.

4.5 Conclusion

Topographic controls did not contribute to a hydrophysical moisture gradient downslope, likely because of the lack of soil structure typical of newly reclaimed soils limiting gravitational influences on moisture re-distribution. Additionally, the incorporation of hummock and hollow land features, and the inclusion of tillage is likely to minimized downslope lateral surface redistribution and erosion in low-relief constructed uplands. When the formation of multiple ecosites ranging in moisture and nutrient regimes is desired, as was anticipated for the transition zone, the placement of peat substrate can develop such ecosites as it demonstrated stronger moisture retaining abilities. Early vegetation assessment from a similar constructed fen within the AOSR found that transitional riparian zones are likely to form over areas of peat substrate with continuously shifting aerobic conditions (Vitt et al., 2016). This would allow for the optimization of quality FFM.

Physicochemical soil properties were not influenced by topographic controls either, with the high and low-slope often demonstrating insignificant differences. Inorganic nitrogen availability was highly influenced by temperature, as mineralization likely had the greatest influence on spatial variation, and not soil moisture or mobility. Nitrogen has often proved to be a limiting macronutrient in reclaimed uplands and therefore available nitrogen was likely immobilized immediately by biotic components. However, the highly mobile nature of NO_3^- would speculate its susceptibility to vertical leaching following precipitation events, specifically where recharge promoting structures are present. Contrarily, although SRP detection was minimal in the groundwater, excess in availability and a positive relationship with soil moisture demonstrates that phosphates are susceptible to leaching during near surface soil flushing events. Reclamation strategies should monitor physicochemical soil characteristics prior to the application of amendments such as fertilizer to reduce potential for leaching.

Vegetation communities were rather homogeneous and predictable. Nevertheless, functional groups were influenced by both hydrophysical and physicochemical soil properties. However, this was not along a moisture-nutrient topographic gradient as observed in undisturbed WBF. Saplings cover was shown to be limited by moisture availability, and less likely limited by nutrient availability. Non-native undesired species cover on the other hand, was influenced by areas of poor soil quality, with elevated bulk density low moisture availability. Although non-native species can help accelerate pedogenic development, the autogenic processes initiated by the pioneer species cover was related to areas of the cover soil with more elevated SRP availability, colonizing areas of the cover soil consisting of better quality. The moisture limitation witnessed by planted species cover further demonstrates the potential for using multiple organic amendments during the reclamation of upland forest soils. Although FFM appears to be favorable in the long-term for colonizing native species, as shown by multiple studies, peat substrates have often demonstrated favorable conditions for sapling survival, therefore reducing the need for fertilizers early-on in reclaimed soils.

Chapter 5: The influence of topographically elevated hillslopes in a post-mined landscape on solute transport towards low-relief reclaimed systems near Fort McMurray, Alberta.

5.1 Introduction

Topography does not always control watershed hydrology in the Western Boreal Forest (WBF), a landscape categorized by a sub-humid climate and deep glaciated substrates resulting in complex surface-groundwater interactions, where soil water storage and evapotranspiration dominate the water budget (Petrone et al., 2007; Smerdon et al., 2005). In this landscape, low-relief (7~12%) mineral upland soils interact with wetlands situated in topographic lows, where runoff contributions to the wetlands are rare and water table gradients are often against topography (i.e. from wetland to adjacent mineral uplands), as moisture from the wetlands supply upland aquifers (Petrone et al., 2015; Devito et al., 2005; Ferone and Devito, 2004; Prepas et al., 2008). Nevertheless, the surface and subsurface upland soils generally demonstrate a moisture gradient influenced by slope position, due to differences in water-contributing area, slope angle, and substrate transmissivity (Johnson and Miyanishi, 2008; Macrae et al., 2005). This gradient is important as it influences the physicochemical properties of the soil, creating a moisture-nutrient gradient that influences vegetation communities (Dimitriu and Grayston, 2010; Johnson and Miyanishi, 2008; Leatherdale et al., 2012).

Currently, disturbances occurring in the Athabasca Oil Sands Region (AOSR) are converting a generally flat landscape into one dominated by topographically elevated forested hillslopes as a result of overburden mine waste expending following extraction (Leatherdale et al., 2012; Rowland et al., 2009). The incorporation of these new land units influences hydrogeochemical re-distribution in the post-mined landscape, primarily by the incorporation of an additional topographic gradients, but also through hydrophysical and physicochemical properties of the cover substrates (Keshta et al., 2010; Ketcheson and Price, 2016a). Organic amendments such as Peat Mineral Mix (PMM) and Forest Floor Material (FFM) are generally placed above sodic-alkaline mine overburden as a substrate for re-vegetation while minimizing contamination, however these amendments generally demonstrate hydrophobic properties and poor transmissivity at a young age, generating considerable volumes of runoff following precipitation events (Barbour, 2015; Huang et al., 2015). The lack of soil structure in reclaimed

hillslopes is generally associated with low infiltration rate, leading to infiltration-excess overland flow when rainfall exceeds the rate of infiltration of the soil (Guebert and Gardner, 2001). However, the hydrophysical properties of reclaimed hillslopes can undergo substantial changes in the first few years following their construction by processes such as root development and freeze thaw cycles, enhancing soils infiltration capacity (Ketcheson et al., 2016b; Raab et al., 2012). As soils infiltration capacity increases, rainfall may lead to deep groundwater recharge or be diverted downslope as shallow subsurface flow, forming a moisture gradient similar to predisturbed conditions (Guebert and Gardner, 2001; Huang et al., 2015). This evolution of the soil properties not only influences hydrological re-distribution, but also nutrient transport as frequent near-surface flushing events lead to the re-distribution of both inorganic nitrogen (NO₃, NH₄⁺) and Soluble Reactive Phosphorus (SRP) (Devito et al., 2000, 1999). Inorganic nitrogen is produced by the decomposition of organic canopy litter into NH₄⁺, followed by nitrification, forming NO_3^{-} , a highly mobile ion with a great potential for leaching in both groundwater and surface runoff, which is undesirable in post-mined landscapes as reclaimed forest soils have often demonstrated nitrogen-limitation (Jung et al., 2014; Kreutzweiser et al., 2008; Macrae et al., 2006). Phosphorus on the other hand, is highly labile and quickly adsorbs to particles and forms insoluble precipitates with mineral elements (e.g. Fe³⁺), making it vulnerable to hydrological processes that move suspended particles, such as erosion following important runoff events (Devito et al., 2000; Kreutzweiser et al., 2008).

Reclamation projects in the AOSR are moving from the construction of individual land units, towards the development of entire watersheds, consisting of forested hillslopes, low-relief uplands and wetlands, where re-establishing natural hydrogeochemical connectivity is essential for the re-development of self-sustaining landscapes (Daly et al., 2012; Pollard et al., 2012). As reclamation practices will accentuate the proportion of topographically elevated hillslopes in the WBF landscape, there requires an understanding as to how these land units will influence nutrient availability within lower lying systems. Therefore, this study explores the spatial and temporal characteristics of three reclaimed hillslopes, varying in age since reclamation, and their hydrogeochemical connectivity during the growing season with a low-relief reclaimed upland situated at the toe of the hillslopes. The specific objectives are to: 1) Characterise the spatial and temporal hydrophysical and physicochemical properties of three reclaimed hillslopes; 2) determine if those properties influence the mobility of SRP and Total Inorganic Nitrogen (TIN) downslope; and 3) make inferences on how vegetation establishment in the low-relief upland may be influenced by the contributions. It is hypothesized that: 1) the older hillslope will demonstrate a downslope hydrophysical and physicochemical gradient; 2) near-surface flushing of SRP and TIN will occur more frequently in the younger slopes, leading to greater contributions to the low-relief upland; and 3) those contributions will be reflected within the near-by vegetation species.

5.2 Materials and methods

5.2.1 Study site

This study was conducted in a constructed watershed, located near Fort McMurray Alberta (Figure 3.2). The individual landforms within this constructed watershed (total watershed area = 32.1 ha), include a low-relief (~3% slope) upland aquifer (7.7 ha) constructed with tailings sand materials, underlying FFM; a fen peatland (2.9 ha) built using fen peat; a sloping natural remnant of the pre-mining landscape (2.8 ha; the "natural slope"); and three reclaimed hillslopes of varying age and characteristics (combined area = 18.7 ha). The east slope (8.1 ha) was reclaimed in 2007 (soils placed) and revegetated in 2008. Canopy cover (Table 5.1) was dominated by tree and shrub species such as trembling aspen (Populus tremuloides) and green alder (Alnus viridis). Both the west (2.4 ha) and southeast hillslopes (8.2 ha) were reclaimed in 2011 and revegetated in 2012. Canopy cover on both slopes consisted primarily of forbs (i.e. Sonchus arvensis) and graminoids (i.e. Hordeum jubatum). All three hillslopes are composed of overburden substrate from the Cretaceuous Clearwater formation, which is dominated by shale and siltstone. The Clearwater overburden material is overlain by a ~100 cm secondary capping layer of suitable overburden material, which is than capped with ~40-50 cm of Peat-Mineral Mix (PMM) cover soil. Both the secondary capping material and PMM soil layers were each directly placed (i.e., not from stockpile) during the construction of the reclaimed hillslopes. For more details on the placement of the soils during the reclamation process see Ketcheson and Price (2016a).

Table 5-1 Description of reclaimed slopes including: size, grade (slope inclination), aspect, vegetation cover and soil texture. Soil texture data was collected in 2014 and might be slightly different from 2015 study. (*) represents year soil was reclaimed. Table modified from (Ketcheson and Price, 2016a). SE=Southeast.

| Hillslope (*) | Size | Grade | Aspect | Veg. cover | Sand | Silt | Clay |
|---------------|------|-------|-----------|------------|------|------|------|
| | (ha) | (%) | | (%) | % | % | % |
| East (*2007) | 8.1 | 13 % | West | 88 % | 38 | 56 | 6 |
| SE (*2011) | 8.2 | 13 % | Northwest | 73 % | 45 | 48 | 7 |
| West (*2011) | 2.4 | 19 % | East | 83 % | 45 | 48 | 7 |

5.2.2 Hydrophysical properties

Transects going along a topographic gradient (upper-slope, mid-slope, downslope) were installed throughout each of the three hillslopes (Figure 3.2). In total, nine soil moisture access tubes were used to assess volumetric water content (VWC) at various intervals (10, 20,30,40,60,100 cm). The access tubes were used to predict soil moisture storage capacity and downslope shallow subsurface flow. VWC measurements were used to determine total volume of water at 15 and 35 cm (TSW₁₅; TSW₃₅) (see chapter 3). At each of the access tubes, additional measurements of surface soil moisture, surface temperature and pore water conductivity (ECp) were recorded. Transect measurements were conducted approximately twice a week. Two soil pit moisture stations were installed, one on the east slope, and one on the west slope. No soil moisture station was installed on the southeast hillslope as it was assumed to respond in a similar manner to that of the West hillslope as they were reclaimed simultaneously. Soil moisture probes (CS-650) were installed horizontally into the PMM reclamation surface at 2.5, 10, and 32.5 cm depths, as well as within the underlying secondary capping material at depths of ~70 cm. Independent VWC calibration curve functions were derived for soils from each of the hillslopes in the laboratory using methodology from Ketcheson and Price (2016a).

Furthermore, data from the soil moisture stations, combined with precipitation data recorded from the watershed's meteorological station were used to determine the frequency and intensity of runoff events on the west slope (although the determination of exact volumes was not possible due to the development of rill erosion). This was not conducted on the east slope as research conducted by Ketcheson and Price (2016a) demonstrated that minimal runoff occurred here. Ketcheson and Price (2016a) used flumes in 2014 to determine the minimum precipitation intensity required to produce a surface runoff response (i.e. precipitation intensity threshold).

The combination of precipitation event data, precipitation intensity threshold, the soil water storage capacity of the near surface (0-2 cm) soil layer (S₀₋₂), and the percolation of water deeper into the soil profile (f_{10}) determined the frequency and intensity of runoff events where:

$$RO_{surf} = S_{0-2} - (i - f_{10}) \tag{1}$$

The soil water storage capacity (i.e., S_{0-2}) represents the amount of additional water that the upper 2 cm soil layer can hold before becoming saturated. This was calculated by subtracting the amount of water stored within the upper 0-2 cm soil layer at the start of precipitation event (VWC from soil pit moisture stations) from the total soil porosity ($S_{0-2} = ~ 5$ mm). The average infiltration capacity measured at 10 cm depth was used to represent precipitation events that percolated into deeper soil layers (i.e., f_{10}), and *i*, represents precipitation intensity that exceeds the threshold (>3 mm). Surface runoff generation was presumed to occur once the storage capacity of the near-surface layer was exceeded and the precipitation intensity exceeded the observed threshold from the runoff flume observation. Although Ketcheson and Price (2016a) and this study occurred only 1 year apart, the hydrophysical properties of the reclaimed soils (i.e. soil porosity, infiltration capacity) changed rapidly. Therefore, results should be regarded with some caution.

5.2.3 Physicochemical properties

Two incubation plots were located within each of the three hillslopes (upper-slope, midslope), with an additional plot located at the toe of the hillslope, within the low-relief upland (Figure 3.2). Quantification of hydrophysical properties were assessed from the nearest immediate soil moisture profile tube. If plots were not located near the transect points, access tubes were installed within the immediate proximity. Physical soil characteristics were determined near each of the incubation plots, where measurements of bulk density (g cm⁻³), organic matter content (%), pH and average plant root to shoot ratio (g g⁻¹) were recorded.

Soil chemical availability was assessed using Plant Root Simulator (PRSTM) probes in triplicates within each of the nine incubation plots. Three 21-day incubation periods (i.e. Early, Middle, Late) determined the temporal variability in ion availability during the growing season. The PRS probes adsorb anions such as: NO_3^- , phosphate (H₂PO₄⁻, HPO₄²⁻), and sulphate (SO₄²⁻),

and cations such as ammonium (NH_4^+) , potassium (K^+) , calcium (Ca^{2+}) , magnesium (Mg^{2+}) , aluminum (Al^{3+}) , iron (Fe^{3+}) , manganese (Mn^{2+}) , copper (Cu^{2+}) , Zinc (Zn^{2+}) and Boron (B^{3+}) .

5.2.4 Surface flushing between land units

Two V-notched flumes were constructed near the toe of each hillslope, directly above the upland-hillslope interface. These flumes collected runoff samples following precipitation events measuring concentrations of SRP and dissolved inorganic nitrogen (. i.e. NO_3^- , NH_4^+). The samples were used to compare ion concentration in runoff with temporal fluxes of ion availability between the hillslopes and the reclaimed upland located downslope.

5.2.5 Statistical analysis

Scheirer-Ray Hare extension of the Kruskal-Wallis test (Dytham, 2011), followed by a post-hoc analysis (function "kruskal", package "agricolae") were used to determine if hydrophysical (soil moisture, VWC, TSW) and physicochemical properties (PRS probes, organic matter, bulk density, pH and plant root to shoot ratio) varied significantly with hillslope (east, southeast, west), slope position (upslope, mid-slope) and/ or time of season (early, middle, late). Permutation multivariate analysis of variance (perMANOVA) (function "adonis", package "vegan) was used to determine spatial and temporal variability in ion availability. This was followed by a multi-response permutation procedure (MRPP) (function "mrpp", package "vegan"), which indicated when and where these significant variations occurred. Indicator species analysis (function "multipatt", package "indicspecies") was used to complete the analysis by informing which incubation plots were significantly different from one another and due to which ions. A linear mixed effect model (function "lme", package "nlme") with repeated measures was used to determine if SRP and TIN (NH_4^+, NO_3^-) were influenced by random (i.e. Surface soil moisture, TSW₁₅, TSW₃₅) and fixed factors (i.e. hillslope, time of season) as a prediction of their susceptibility to surface flushing. Ordination (NMDS) (function "metaMDS", package "vegan") was used to display correlations between hydrophysical properties and chemical distribution, as well as the combination of both nutrients and moisture on vegetation community establishment.

5.3 Results

5.3.1 Hydrophysical properties

Hydrophysical responses between individual hillslopes and topographic positions throughout the growing season were highly variable (Table 5.2). Surface soil moisture demonstrated neither significant differences between hillslopes (p=0.14) nor topographic position (p=0.86). Total volume of water at 15 cm (TSW₁₅) also did not vary between hillslopes (p=0.40), but did demonstrate significant differences among topographic positions (p<0.001), where both the southeast and west hillslope demonstrated greater volumes of water at the upperslope position (20.73 ± 1.38 , 19.26 ± 1.23 mm) compared to both the mid (12.62 ± 2.06 , 18.55 ± 2.41 mm) and low-slope (11.95 ± 0.92 , 7.82 ± 1.01 mm). However, the east-hillslope did not vary spatially (p=0.3). The total volume of water at 35 cm (TSW₃₅) also demonstrated significant differences between hillslope (p<0.001) and topographic position (p<0.001) (all sites pooled). Of the different slopes studied, the southeast hillslope was considerably drier than the east and west hillslope (Table 5.2), meanwhile the east, southeast and west hillslopes all demonstrated greater volumes of water at the upper-slope position (25.93 ± 1.65 , 28.93 ± 1.83 , 10.58 ± 1.32 mm).

Table 5-2 Hydrophysical and physicochemical characteristics ((mean \pm se) relative to each individual hillslope, showing Surface soil moisture (%), Total Soil Volume at 15, 35 cm (TSW₁₅, TSW₃₅), Root to Shoot ratio (R/S), bulk density (ρ_b), organic matter content (%) and pH. Characters: a,ab,b,c are used to indicate significant differences (Kruskal test) between hillslopes.

| Hillslope | Hydrophysical | | | Physicochemical | | | | | |
|-----------|------------------------|------------------------|------------------------|-----------------|------------------------|-------------------------|-------------------------|--|--|
| | Surface | TSW ₁₅ | TSW ₃₅ | R/S | ρ_b | ОМ | pН | | |
| | (%) | (mm) | (mm) | (g/g) | (g/cm^3) | (%) | | | |
| East | $17.1(0.9)^{a}$ | $11.9(0.7)^{a}$ | 27.8(1.9) ^a | $3.1(0.2)^{a}$ | $1.1(0.06)^{b}$ | 20.8(0.48) ^a | $6.9(0.03)^{a}$ | | |
| SE | $17.4(0.9)^{a}$ | 14.9(0.9) ^a | 23.5(1.5) ^b | $0.8(0.1)^{b}$ | $1.2(0.03)^{a}$ | 11.9(0.65) ^c | 6.7(0.11) ^{ab} | | |
| West | 16.0(0.8) ^a | 13.7(0.8) ^a | 33.3(2.5) ^a | $4.7(0.1)^{a}$ | 1.3(0.04) ^a | 14.2(0.08) ^b | $6.4(0.05)^{b}$ | | |

The near surface (0-2 cm) of the east hillslope responded more frequently to precipitation events comparatively to the west hillslope (Figure 5.1). The east hillslope demonstrated increases in VWC, dependent on the intensity of the precipitation events. However, the deeper layers of the PMM substrate (10 and 32.5 cm) responded very little to the precipitation events, and both

appeared to gradually decrease throughout the research season, eventually reaching a plateau (0.21, 0.24 respectively). Volumetric water content from the secondary overburden capping layer remained stable throughout the season (Figure 5.1). Comparatively, the west slope did not respond to precipitation events in the surface layers (e.g., low variability in VWC profile), indicating that net percolation was low and lateral surface flow was greater than on the east hillslope. Between June 11th (DOY 162) and August 10th (DOY 222), 8 rainfall and subsequent runoff events occurred on the west slope. Although no moisture pit was located on the southeast hillslope, it can be assumed that runoff events were either equal or greater than on the west hillslope. Following an eighteen-day period, (DOY 185-203), approximately 40 % (60 mm) of the growing season's precipitation occurred. Soil moisture profile access tubes on the southeast hillslope (APPENDIX) demonstrated minimal variations in VWC, indicating that this hillslope frequently conveyed lateral surface flow towards the low-lying constructed upland, as confirmed by in-situ observations.



Figure 5-1 In situ VWC measurements on the (a) East hillslope and (b) West hillslope for the periods of June-August 2015. (c) Precipitation (black bars) and runoff (gray bars), for west Slope only.

5.3.2 Physicochemical properties

Physical properties varied between hillslopes as expected (Table 5.2). However these variations were not influenced by topographic position. Similarly, ion availability also varied between hillslopes but was not influenced by topographic position (Table 5.3). Hillslope variability in ion composition (p<0.001; F=10.59) demonstrated that, although significantly different, very little separation was detected between the southeast and west hillslope (p<0.001; T=-4.31; A=0.1). The greatest degree of separation occurred between the east and southeast hillslope, (p<0.001; T=-22.16; A=0.381), followed by the east and west hillslopes which demonstrated a similar degree of separation (p<0.001; T=-21.84; A=0.344). Although, time of season was influenced (p=0.015; F=3.52) it demonstrated little influence on separation among hillslopes. Cluster analysis confirmed results from the MRPP, indicating that the two groups of incubation plots varied significantly between one another, with one group representing both the west and southeast hillslope, and the second group representing the east hillslope. Ions and compounds indicative of the southeast and west hillslope were; SO₄²⁻, Fe³⁺, Mn²⁺, Cu²⁺, Zn²⁺, Ca²⁺ and TIN. The east hillslope was indicative of SRP only (Figure 5.2).

Ordination demonstrated little to no significant correlations between environmental variables and ion availability (Figure 5.2). It appears that the salvaged PMM and maturity of the hillslope had a greater influence on ion availability. Furthermore, the east slope appeared to demonstrate greater temporal and spatial heterogeneity in ion availability throughout the research season compared to both other hillslopes.

Table 5-3 Multi-Response Permutation Procedure (MRPP) results for ion availability relative to Hillslopes and time of season. Result demonstrate separation (T) between hillslopes and separation (A) amongst same-slope replicates. SE= Southeast. *Bold numbers represent significant differences (p>0.05).

| | Time of Season | | | | | | | | |
|-----------------------|-------------------|-------|---------|------|---------|-------|---------|-------|--|
| Hillslope | Throughout season | | Early | | Middle | | Late | | |
| | Т | А | Т | А | Т | А | Т | А | |
| East vs SE vs West | -24.49* | 0.355 | -14.36* | 0.37 | -14.55* | 0.371 | -15.73* | 0.379 | |
| East vs SE | -22.16* | 0.381 | -13.09* | 0.39 | -13.23* | 0.393 | -14.25* | 0.403 | |
| SE vs West | -4.82* | 0.049 | -4.31* | 0.08 | -5.63* | 0.093 | -0.365 | 0.006 | |
| West vs East | -21.84* | 0.344 | -12.16* | 0.34 | -13.79* | 0.347 | -14.39* | 0.392 | |



Figure 5-2. NMDS ordination plots demonstrating the degree of separation amongst hillslopes in relation to ion availability. Vectors represent strong correlations (p<0.05) between environmental variables and incubation plots. (T)-value represents the degree of separation amongst slope positions. (A)-value represent same-hillslope replicate homogeneity.

5.3.3 Surface flushing between land units

Runoff samples demonstrated different responses relative to individual hillslopes (Figure 5.3). The east and southeast hillslope demonstrated similar concentrations of DIN, as both showed maximum DIN runoff early in the growing season (0.59 ± 0.25 ; 0.63 ± 0.31 mg ml⁻¹), gradually decreasing late in the season (0.13 ± 0.05 ; 0.24 ± 0.14 mg ml⁻¹). The west slope showed a similar trend in maximum DIN runoff early-season (1.84 ± 0.45 mg ml⁻¹) gradually decreasing late in the season (0.32 ± 0.16 mg ml⁻¹), however concentrations were much greater. Conversely, the east and west slope showed similar trends for SRP concentrations, where early-season had the greatest concentration (0.94 ± 0.34 ; 0.83 ± 0.28 mg ml⁻¹), while mid (0.37 ± 0.04 ; 0.38 ± 0.13 mg ml⁻¹) and late-season (0.53 ± 0.12 ; 0.47 ± 0.22 mg ml⁻¹) had similar values. Alternatively, the southeast hillslope demonstrated entirely opposite trends, where SRP concentrations in runoff increased throughout the season (Figure 5.3)



Figure 5-3 Dissolves Inorganic Nitrogen (DIN) and Soluble Reactive Phosphorus (SRP) (mean \pm se) concentrations collected from runoff samples following major storm events on each individual hillslope. E=Early, M=Middle, L=Late

Plant Root Simulators demonstrated differences in TIN and SRP concentrations among each individual land unit, and, although topographic position had little influence on the hillslope, ion availability within the low-lying constructed upland demonstrated strong spatial variability (Figure 5.4). Throughout the growing season, TIN availability on the east hillslope did not vary significantly (p=0.15). However, the incubation plot located at the toe of the hillslope (i.e. upland), demonstrated a large increase in availability mid-season (160%), but then decreased to its lowest availability (10.53±0.39 μ g/10cm²). Conversely, the southeast and west hillslope demonstrated significant differences in TIN availability throughout the growing season (p=<0.001, F=22.22; p<0.001; F=21.51). Both slopes demonstrated increases in the mid-season (261; 248 %). However, the southeast slope decreased to its lowest availability late into the season (3.12±0.85 μ g/10 cm²), meanwhile the west slope did not decrease as dramatically and had slightly greater availability compared to earlier in the season (5.63±0.78; 10.05±1.7885 μ g/10 cm²). At the toe of the west hillslope, TIN availability responded similarly to the plots located on the hillslope. Additionally, the west hillslope was shown to contribute the greatest DIN concentrations in runoff. Very little difference in availability was noticed at the toe of the southeast slope.

SRP availability was highest within the east hillslope compared to the southeast and west hillslopes throughout the growing season (Figure 5.4b). East hillslope SRP availability increased in the mid-season (3.40 ± 0.81 to $8.65\pm1.62 \ \mu g/10 \ cm^2$), as was found within the respective upland plot located immediately downslope (0.73 ± 0.07 to $3.50\pm0.50 \ \mu g/10 \ cm^2$). However, this trend was also noticed within the upland plots located downslope of both the southeast (2.0 ± 0.32 to $5.13\pm0.35 \ \mu g/10 \ cm^2$) and west hillslope (1.03 ± 0.24 to $4.23\pm1.04 \ \mu g/10 \ cm^2$), where the incubation plots located on the hillslopes themselves varied very little throughout the growing season. The upland incubation plot located downslope of the southeast hillslope consistently had the greatest availability of SRP (Figure 5.4). Furthermore, the linear mixed-effect model determined that SRP was solely influenced by soil moisture content. A strong coefficient of determination (R^2 =0.69) dictates that SRP availability was influenced by surface soil moisture (p<0.001; F=7.2), time of season (p<0.001; F=23.94), hillslope (p=0.0017, F=29.92), TSW₁₅ (p<0.001; F=23.94), and the interaction with substrate: TSW₁₅ (p<0.001; F=31.10).


Figure 5-4 PRS data demonstrating (a) TIN and (b) SRP availability across topographic transects located at the upper and middle-slope location of each individual hillslope, and the lowest plot located at the immediate vicinity of the hillslope-upland interface (C.Upland). Characters: a, ab, abc, b, bc, c are used to indicate significant differences (Kruskal test) among treatments. Post-hoc analysis included the PRS data from each hillslope (grey bars). The constructed upland incubation plots were isolated from the hillslope data (black bars). SE=SouthEast

5.3.4 Vegetation responses to land unit connectivity

Vegetation colonization appeared to have a stronger relationship with maturity of the reclaimed land units compared to the soils edaphic characteristics (Figure 5.5). Figure 5.5a shows that the east hillslope, reclaimed in 2007, was strongly correlated with tree and shrub species, while the only physicochemical property showing a positive relationship was SRP (R^2 =0.8; p=0.009). On the other two hillslopes, certain plots displayed similar edaphic characteristics to the lower lying constructed upland, despite having different organic amendments (i.e., PMM, FFM) and planting prescriptions. Forb colonization was strongest within a plot located on the west slope and the upland plot located at the toe of the east hillslope, where bulk density (R^2 =0.62; p=0.05), NO₃⁻ (R^2 =0.83; p=0.008), temperature (R^2 =0.73; p=0.013) and Mn⁺² (R^2 =0.54; p=0.06) strongly influenced forb colonization.





Figure 5-5 NMDS ordination plots demonstrating relationships between environmental vectors and functional vegetation groups. Vectors represent strong correlations (p<0.05). (a) forbs, graminoids, shrubs and trees. (b) native, non-native and planted species. K represents the number of dimensions for best fit and the stress value describes the fit of the ordination model.

Figure 5.5b further demonstrates the influence of maturity (or time since reclamation), as planted species dominate the east hillslope, while functional groups are similar between the southeast and west hillslope, as well as the upland. Canopy cover (% Cover) (R^2 =0.64; p=0.05) and SRP (R^2 =0.63; p=0.038) demonstrated positive relationships with the vegetation communities on the east hillslope, native and non-native species were equally distributed amongst the upland and the remaining two hillslopes. Plots located near areas of elevated NO₃⁻ (R^2 =0.73; p=0.022), Fe⁺² (R^2 =0.81, p=0.007), temperature (R^2 =0.68, p=0.038) and bulk density (R^2 =0.53, p=0.01) showed positive correlations with non-native species colonization, while native species seemed to have little environmental vectors.

5.4 Discussion

5.4.1 Hydrophysical properties

Neither the younger nor the more mature hillslopes demonstrated a topographic gradient in relation to VWC. This contradicts previous research by Ketcheson and Price (2016a) within the same research site, who demonstrated that VWC on the east hillslope (i.e. 2007) increased downslope, while the southeast and west hillslope demonstrated increased VWC with increased elevation. They attributed this topographic control on moisture distribution as influenced by increased hydraulic conductivity and increased interflow, as hydrophysical properties of cover materials quickly evolve with time (Barbour et al., 2004; Guebert and Gardner, 2001). Although other studies have also found a lack of topographic influence on moisture content in slopes ranging from 3-13 years (Leatherdale et al., 2012), it is possible that our study required greater replication to capture the effect of topographic gradient on soil moisture content. Ketcheson and Price (2016a) conducted moisture surveys over the entirety of the hillslopes, recording measurements every 20 meters, while this study focused solely on transects consisting of three moisture plots for the individual hillslopes.

Ketcheson and Price (2016a) had determined that the east hillslope served as a moisture storage unit, while the southeast and west hillslopes functioned as water conveyers to the lower land units. However, this 2015 study was conducted during a relatively dry year in comparison to the previous studies (i.e. 2013, 2014) at this same site. VWC below the PMM surface layers (i.e. 10, 32.5 cm) was never influenced by precipitation events, suggesting that the surface layer (2.5 cm) demonstrated high water retaining properties, as the PMM was never saturated enough to promote considerable infiltration and/or interflow during the growing season (Figure 5.1). High soil water capacity and elevated evapotranspiration demands could explain the lack of topographic controls on the east slope in 2015.

5.4.2 Physicochemical properties

As initially hypothesized, physicochemical properties demonstrated considerable differences between the east (mature) and the southeast and west (younger) hillslopes (Figure 5.2; Table 5.3). Organic matter content was much higher on the east hillslope, as expected due to greater canopy cover and more mature vegetation, producing greater volumes of litter. Soil organic matter accumulation is one of the most significant processes of initial pedogenic

development and is often used as a criterion for assessing reclamation progress (Raab et al., 2012; Turcotte et al., 2009). Using this criterion, it can be hypothesized that the west hillslope is maturing quicker than the southeast hillslope, as it demonstrated greater organic matter content. *In-situ* observations demonstrated that considerable erosion events occurred regularly on the southeast hillslope, while these were less evident on the west hillslope in 2015. The lack of stabilized soil structure can also explain the lack of below ground biomass development on the southeast hillslope. The stabilization of soil structure allows for the leaching of organic matter deeper into the soil profile, increasing bioactivity and root growth, initiating autogenic processes where root development will continue to stabilize the soil (Landhäusser et al., 2015; Raab et al., 2012).

It was initially hypothesized that the east hillslope would demonstrate topographic controls on ion availability. However, this was not the case, as topographic position was not shown to influence ion availability on either of the three hillslopes. This could have been the result of the relatively dry conditions during the growing season, which limited water from moving downslope, therefore having a limited influence on the spatial distribution of mobile ions. Furthermore, as interconnectivity amongst land units with respect to lateral surface redistribution was the focus of this research, only two plots (i.e. upper and mid-slope) were located on each hillslope. The placement of a third incubation plot at the "lower" portion of the hillslope prior to the upland-hillslope interface could have augmented the detection of topographic controls.

With agreement on the initial hypothesis, the inter-hillslope degree of variation in term of ion availability was likely a result of age since reclamation and the ecosite of the salvaged organic amendment as the east hillslope was substantially different from both the southeast and west slope. Although all three hillslopes were similarly designed with PMM (~50 cm) overlying a secondary (~100 cm) and primary overburden capping layer, the salvaged peat mineral mix was likely from a different ecosite (Figure 2.1). A study by Dimitriu et al. (2010) found that microbial composition and function, which largely controls nutrient availability, was more strongly influenced by amendment type (i.e. salvaged ecosite) comparad to age since reclamation, suggesting that younger reclaimed soils were more similar to natural forested areas depending on reclamation material. However, the inclusion of a forest canopy dominated by native tree and shrub species on the more mature east hillslope, means that inputs for

mineralization from the canopy are different than those on the southeast and west hillslopes, where canopy cover is still dominated by forbs and grasses, and therefore, the east hillslope is progressively leading towards more natural ecosystem function (Norris et al., 2013; Quideau et al., 2013; Rowland et al., 2009). Furthermore, the lack of direct correlation between environmental vectors and ion availability (Figure 5.2), demonstrates the degree to which these land units are different from one another. Additionally, the east hillslope, which should have tighter biogeochemical cycling due to its maturity and proximity to that of natural boreal forest (MacKenzie and Quideau, 2012; McMillan et al., 2007), demonstrates the greatest heterogeneity on the ordination plot, while the two other slopes were grouped rather closely together. As previous years demonstrated topographic controls on soil moisture on the east hillslope, the heterogeneity could be linked to the formation of a moisture-nutrient gradient developing along topographic controls as was initially hypothesized. Even though both plots demonstrated similar community composition (Figure 5.5a), the upper-slope demonstrated a strong relationship with tree canopy. However, the mid-slope demonstrated a strong affinity towards shrubs, potentially leading towards the development of two different ecosites, ranging in soil moisture and nutrient regimes (Figure 5.5a).

5.4.3 Surface flushing between land units

Dissolved Inorganic Nitrogen concentrations decreased within the runoff samples at each of the hillslopes throughout the research period. This is to be expected as leaching in the WBF typically decreases throughout the growing season due to an increasing demand from the vegetation (Ferone and Devito, 2004; Pelster et al., 2008b). While DIN runoff concentrations were similar for both the east and southeast hillslope, the west hillslope contributed much higher concentrations. The west hillslope was expected to contribute higher flushing of DIN than the east slope due to the soils moisture retaining abilities; however it was unexpected that the southeast and east slope demonstrated similar responses, especially considering that the southeast and west hillslope had similar TIN availability. Furthermore, the heterogeneity of the low-lying upland, the change in cover substrate material and the addition of fertilizer made it difficult to determine specific contributions from the hillslopes, as the incubation plot located at the toe of the east hillslope demonstrated the highest TIN availability, while the southeast hillslope showed the lowest. NO₃⁻ availability within the upland (see chapter 4, Figure 4.3) was shown to be

strongly influenced by temperature, signifying that hillslope contributions, and therefore landscape connectivity had a minimal influence on TIN availability, and nitrification was likely the primary source of TIN, as typically seen in reclaimed uplands (Hemstock et al., 2010; McMillan et al., 2007). Additionally, linear mixed effect model demonstrated absolutely no influence of soil moisture on TIN (i.e. NH_4^+ , NO_3^-), and was thereby likely immobilized prior to any flushing event.

Contrarily, SRP concentrations in runoff demonstrated similar concentrations and trends between the east and west hillslope, decreasing throughout the growing season, typical within the WBF. However, the opposite trend was noticed on the southeast hillslope. Phosphorus mobility within the WBF is generally governed by erosional processes due to its strong affinity to soil particles (Kreutzweiser et al., 2008; Pelster et al., 2008a, 2008b). Although SRP availability varied minimally on the southeast hillslope throughout the growing season, erosion was often witnessed following precipitation events. SRP availability was shown to be influenced by moisture, this could imply that desorption of SRP following erosional processes within the upland, either by the influence of DOC within soil water, or extracellular microorganism enzymes within the FFM, capable of extracting phosphates from soil particles (Brown and Naeth, 2014; Jamro et al., 2014). The minimal precipitation events which occurred late-season (Figure 5.1), might have led to the accumulation of SRP until the last major flushing event (DOY \sim 220), which was depicted from the runoff samples. The southeast hillslope experienced erosion frequently, even rendering sampling difficult as the runoff collectors would often fill with soil particles despite preventive measures (i.e. mesh screening). Phosphorus inputs from the southeast hillslope is feasible as the upland incubation plot located at the toe of the hillslope demonstrated the greatest SRP availability for most of the growing season. Furthermore, unlike TIN, SRP availability did not demonstrate any strong correlation with edaphic soil parameters within the low-lying constructed upland (Figure 5.5), suggesting allogenic sources, such as the surrounding hillslopes, could be contributing SRP to the upland. Additionally, the linear mixed effect model demonstrated that SRP was positively influenced by moisture.

5.4.4 Vegetation responses to land unit connectivity

Vegetation colonization seemed to have a stronger relationship with the maturity of the land units relative to cover substrate. The more mature hillslope demonstrated a stronger relationship towards shrub and tree species, which are imperative for reclaiming the land to selfsustaining conditions. Research has shown that within the first five years following soil placement, regardless of amendment type, forbs, graminoids and non-native species will dominate the soil due to their competitive abilities (Errington and Pinno, 2015a; Pinno and Hawkes, 2015). This was made evident as plots located in both PMM and FFM (i.e. upland) demonstrated very similar vegetation communities, where forb colonization was influenced by elevated NO₃⁻ availability, temperature and bulk density. These results further confirm that NO₃⁻ contributions from the hillslopes are likely minimal as soil temperature is the primary influence on NO₃⁻ availability, regardless of proximity to hillslopes. Soluble reactive phosphorus was correlated with the more mature hillslope, and certain studies have shown that SRP availability could be used as an indication of successional stage in reclaimed forested uplands (Pinno and Hawkes, 2015). Furthermore, elevated canopy cover and SRP (Figure 5.5b) also demonstrated strong correlations with the planted species (e.g. trees and shrubs), suggesting that once proper vegetation cover is established, autogenic processes will limit the colonization of invasive forb and graminoid species, allowing for the development of desired vegetation communities.

5.5 Conclusion

Neither of the three reclaimed hillslopes demonstrated any topographic controls on soil moisture or soil nutrients during the 2015 growing season. However, the east hillslope (i.e. mature) demonstrated topographically driven influences on soil moisture in both 2013 and 2014 (Ketcheson and Price., 2016b), and could thereby explain why physicochemical soil properties exhibited greater heterogeneity than both the southeast and west hillslope (i.e. younger) and differences in tree and shrub canopy composition, potentially developing towards two different ecosites. Furthermore, the difference in maturity and salvaged organic amendments made it difficult to attribute spatial and temporal differences in ion availability to that of hydrophysical properties, as physicochemical properties were found to be more similar between the southeast and west hillslopes, which were reclaimed at the same time and constructed from the same salvaged PMM material.

Determining surface flushing inputs of TIN and SRP to the low-lying upland from the surrounding hillslopes proved difficult for a variety of reasons, as the heterogeneity of the cover soil in the upland, the addition of fertilizer mid-season and the differences in physicochemical

properties between hillslopes added unexpected challenges to the initial objective. Building upon the results from Ketcheson and Price (2016a), the east hillslope was determined to be a water storage unit during the growing season, releasing minimal amounts of runoff, contrarily to the other two hillslopes, deemed as water conveyers to the low-lying upland. Given these results, it is possible that samples collected on the east hillslope were strongly linked to the chemical composition of precipitation. This would suggest that the west hillslope might be contributing TIN to the low-lying upland as samples collected were much higher than both the east and southeast hillslopes. However, ordination demonstrated that NO₃⁻, the dominant form of TIN in the aerobic components of the watershed, was highly influenced by temperature, and therefore contributions from the surrounding hillslopes might be negligible in relation to overland flow. This could be related to the rapid uptake of available TIN on the hillslopes by the vegetation, which is expected in the development stage of a newly reclaimed site. Conversely, SRP availability was generally strongest at the toe of the southeast hillslope, an area where erosion frequently occurs, and SRP was found to be significantly influenced by elevated moisture.

Vegetation colonization was related to the maturity of the land units and physicochemical soil properties rather than organic amendment type. SRP contributions from the southeast hillslope might have influenced species colonization, however this was not conducive. Research presented in the previous chapter found that SRP was strongly correlated to native species, many of which were graminoids, which can limite the survival of planted species. This research demonstrates that the additional topography incorporated within the post-mined landscape could potentially lead to nutrient flushing, especially phosphorus from erosion prone land units, towards the land units situated on topographic lows.

Chapter 6: Summary of thesis and recommendations for oil sands reclamation

Reclamation strategies will need to be reconsidered when engineering entire watersheds consisting of multiple land units. The lack of topographic control on moisture-nutrient regimes depicted by the results from this study implies that ecosites increasing in moisture content in close proximity of wetlands will necessitate cover soil stabilization and the development of subsurface preferential flow paths prior to the formation of transitional areas. Hence the need for long term monitoring of topographic controls. Riparian zones, which are naturally occurring transition zones, have crucial roles in regulating biogeochemical cycling in the WBF, and should therefore be integrated when creating post-disturbance landscape functionality. Although FFM has proven to be superior in re-initiating biogeochemical forest floor functionality, results from this study demonstrate that PMM might be ideal for the creation of such transitional areas due to their greater moisture and nutrient absorption abilities. This would allow for the optimization of quality FFM, while also benefiting from the advantageous characteristics of PMM as a reclamation material. Furthermore, as sapling survival is often superior in PMM, as was demonstrated in this study, these transitional areas would likely develop faster than the FFM covered upland, reducing undesirable solute and soil particle contributions downslope into integrated land units following surface flushing events.

The implementation of reclamation practices such as tillage and fertilization might also need to be re-visited considering these results. Although tillage was necessary for increasing hydrological connectivity within the landscape, and is not traditionally practiced in reclamation, tilled land has been known to increase labile soil organic matter, leading to increased leaching. This would potentially annul salvaging efforts to minimize soil disturbance such as reduced stockpiling. If tillage is going to become a regular component of landscape reclamation, further research is needed to determine its influence on physicochemical properties. Additionally, although fertilization was not incorporated within the designs of this study, it likely influenced the temporal and spatial distribution of chemical soil properties throughout the constructed upland. The use of controlled release fertilizers has decreased application rates on reclaimed land relative to the usage of immediately available fertilizers. However, results from the runoff collectors and research conducted on multiple other reclaimed sites, have demonstrated that CRFs lead to considerable leaching. Very little research on the fertilizer use efficiency of CRFs has been conducted on reclaimed soils, as interconnectivity between land units was of minimal concern. Further research on FUE and the buffering capacity of the soil should be conducted prior to fertilization in land units vulnerable to frequent surface flushing. Additionally, while N-P-K availability was greater within the FFM, and are generally plant growth limiting factors, the fact that sapling survival was superior in areas of elevated moisture suggest that water stress is causing sapling mortality and the addition of fertilizer might not be beneficial until FFM absorption abilities are increased. The development of a moisture stress protocol for individual sapling species (i.e. drought tolerance), through measurements such as stomatal conductance and transpiration rates could improve afforestation procedures by limiting sapling mortality during drought years. The addition of fertilizer when sapling survival rates are low favors the colonization of stress-tolerant invasive species and can promote leaching.

While runoff samples demonstrated the susceptibility of fertilizers to being leached immediately upon application, this study demonstrated that SRP is more vulnerable to being mobilized following surface flushing events due to its likely excessiveness within the constructed upland. However, the strong affinity of phosphates for mineral subsoils and the lack of detection within groundwater chemistry suggests' that SRP vertical movement is less likely of concern, similarly to natural forested uplands. Contrarily, as nitrogen limitation was probable in our study site, as is generally the case for reclaimed soils, immobilization from the vegetation and soil microorganisms likely reduced its mobility within the surface. However, inorganic nitrogen might be susceptible to vertical leaching, especially with the likely implementation of recharge basins used to promote surface-groundwater interactions. Research focusing on the accumulation of nutrients at the capillary barrier formed by the cover soil/ tailing sand interface following precipitation events would increase our understanding of leaching within reclaimed soils.

The addition of topographically elevated land units in post-mined landscapes will likely translate towards phosphorus loading into land units located at the toe of the hillslopes. Erosion occurs frequently in newly placed cover soils and is often one of the factors impeding successful reclamation. Although excessive available phosphorus did not necessarily demonstrate any undesirable influence on the constructed upland, reclaimed soils differ uniquely and could therefore be of concern in other engineered watersheds. The construction and stabilization of topographically elevated land units prior to incorporating additional land units, could be beneficial towards limiting such contributions. Additionally, the hillslopes demonstrated negligible inputs of inorganic nitrogen towards the constructed upland, as elevated temperature, and thereby mineralization, was likely the primary source of available nitrogen in the upland. While the addition of SRP demonstrated a potential benefit in favoring the colonization of native species, additional TIN inputs could be detrimental as it demonstrated strong correlations to forb and non-native species.

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Appendix

| Land unit | Time of Season | TIN | SRP | \mathbf{K}^+ | SO_4^{-2} | Ca ⁺² | Mg^{+2} |
|-----------|----------------|--------------|-------------|----------------|-----------------|------------------|----------------|
| Upland | Early | 10.96 (1.09) | 1.05 (0.1) | 51.73 (4.63) | 298.26 (43.30) | 1329.86 (122.82) | 193.36 (13.03) |
| | Middle | 19.89 (2.5) | 2.54 (0.3) | 113.16 (13.03) | 452.43 (63.89) | 1571 (97.58) | 225.13 (12.41) |
| | Late | 7.46 (0.69) | 2.86 (0.28) | 92.32 (10.09) | 300.79 (55.99) | 1224.67 (118.56) | 172.57 (14.96) |
| East | Early | 4.67 (1.1) | 3.4 (0.72) | 88.27 (3.41) | 138.52 (23.12) | 1978.78 (200.6) | 238.68 (20.69) |
| Hillslope | Middle | 5.35 (0.71) | 8.65 (1.07) | 130.63 (16.98) | 208.65 (26.79) | 2043.9 (147.47) | 223.88 (14.4) |
| I | Late | 4.55 (1.64) | 3.62 (0.5) | 82.70 (10.62) | 130.17 (29.61) | 1780.78 (172.10) | 227.43 (21.3) |
| SE | Early | 6.2 (1.26) | 0.75 (0.14) | 26.85 (1.82) | 923.83 (29.61) | 2042.83 (94.6) | 259.45 (10.06) |
| Hillslope | Middle | 16.2 (3.06) | 0.95 (0.11) | 39.22 (2.66) | 965.53 (63.17) | 2365.53 (102.58) | 292.87 (12.42) |
| Ĩ | Late | 3.12 (0.54) | 1.28 (0.14) | 36.67 (5.22) | 897.07 (128.44) | 2439.9 (62.52) | 310.03 (21.66) |
| West | Early | 5.63 (0.71) | 0.97 (0.17) | 40.47 (3.82) | 811.30 (70.88) | 2013.02 (78.12) | 244.12 (8.26) |
| Hillslope | Middle | 13.97 (0.93) | 1.2 (0.13) | 72.02 (10.99) | 1135.6 (30.97) | 2302.67 (97.37) | 252.75 (11.07) |
| | Late | 7.0 (1.06) | 1.32 (0.18) | 84.10 (5.87) | 717.73 (51.24) | 2115.98 (122.46) | 254.75 (16.19) |

Table A1. Ion availability (mean \pm se) relative to individual land units and time of season (μ g/ 10cm² / burial period)

| Substrate | | FFM | | | | PMM | | | | | | |
|-------------------|-------|-------|----------------|-------------|-----------|-----------|-------|-------|----------------|-------------|-----------|-----------|
| Factor | TIN | SRP | \mathbf{K}^+ | SO_4^{-2} | Ca^{+2} | Mg^{+2} | TIN | SRP | \mathbf{K}^+ | SO_4^{-2} | Ca^{+2} | Mg^{+2} |
| TIN | | | | 0.37 | 0.42 | 0.51 | | | | 0.28 | 0.29 | |
| SRP | | | 0.75 | | | | -0.19 | | 0.68 | -0.54 | | |
| \mathbf{K}^+ | | 0.75 | | | | | | 0.68 | | -0.49 | | -0.22 |
| SO_4^{-2} | 0.37 | | | | 0.59 | 0.67 | 0.28 | -0.54 | -0.49 | | | 0.45 |
| Ca^{+2} | 0.42 | | | 0.59 | | 0.88 | 0.29 | | | | | 0.30 |
| Mg^{+2} | 0.51 | | | 0.67 | 0.88 | | | | -0.22 | 0.45 | 0.30 | |
| Fe ⁺³ | 0.48 | -0.30 | | 0.85 | 0.67 | 0.70 | | -0.35 | -0.40 | 0.39 | 0.24 | 0.24 |
| Mn^+ | 0.65 | | | 0.62 | 0.61 | 0.62 | | -0.31 | | 0.22 | | |
| Zn^{+2} | | 0.28 | 0.46 | 0.28 | 0.27 | | | | -0.23 | 0.45 | 0.41 | 0.22 |
| \mathbf{B}^{+3} | | -0.49 | -0.36 | | | | | | -0.24 | | | |
| Al^{+3} | | | | | 0.30 | 0.33 | | | -0.21 | | | 0.33 |
| Soil Moisture | 0.30 | -0.28 | | | | | | | -0.23 | | 0.50 | |
| Ecp | | -0.44 | -0.40 | | | | | | | | 0.48 | |
| Temperature | 0.34 | | | 0.31 | 0.44 | 0.36 | | 0.25 | 0.28 | | -0.32 | |
| T2cm | 0.40 | | 0.26 | 0.38 | 0.44 | 0.30 | | 0.36 | | | | |
| T10cm | 0.38 | | | | | 0.28 | | | | | | |
| TSW_{15} | -0.34 | | | | -0.26 | | | | | | | 0.29 |
| TSW ₃₅ | | | | | | | | | 0.24 | | | |
| Organic M | | | | | | | | 0.38 | 0.32 | -0.66 | | -0.44 |
| Bulk D | | | | | | | | -0.50 | -0.49 | 0.73 | | 0.45 |
| R.S | | | | | -0.36 | -0.38 | | | 0.28 | | | |
| PC | | | | | | | -0.31 | 0.40 | 0.47 | | -0.39 | |
| pН | | | | 0.29 | | | | 0.36 | | | 0.25 | -0.31 |

Table A2. Spearman rank correlation matrix of major plant nutrients (TIN, SRP, K^+ , SO_4^{+2} , Ca^{+2} , Mg^{+2}) and environmental variables. Only significant (p<0.05) correlations are shown.



Figure A1. Soil profile access tubes demonstrating differences in interflow between upper and lower hillslope position following an 18-day period ((DOY 185-203), where approximately 40 % (60 mm) of the growing season precipitation fell. Black line represents before event, grey lines represent after.