

Growing Woody Plants in Oil Sands Fine Tailings

by

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## Abstract

Fine fluid tailings (FFT) are a by-product created during the extraction of bitumen from oil sands mining operations. Over 975 million m<sup>3</sup> of FFT are currently being stored in tailings ponds in the Athabasca oil sands region (AOSR) of Alberta, Canada. These tailings cause industrial and environmental concerns due to storage and management issues, and potential hazard to the surrounding environment. A potential solution for managing these tailings ponds is to dewater them through technologies such as centrifugation and use the dewatered FFT cake as a subsoil material in reclamation. The FFT would need to be capped with suitable soil and depth in order to support plant growth and meet reclamation requirements. The optimal minimal capping material and depth are not well studied.

In the first study (Chapter 2), a 16-week greenhouse study was conducted to assess whether FFT cake and caps of various mixes and depths (0, 5, 10 and 20 cm depth) of forest floor mineral mix (FFMM) and peat mineral mix (PMM) would support plant growth of trembling aspen (*Populus tremuloides* – native broadleaf tree) and beaked willow (*Salix bebbiana* – native broadleaf shrub). *S. bebbiana* had a greater survival rate (100%) when grown directly in FFT cake compared to *P. tremuloides* (16.7%). The same *S. bebbiana* seedlings had 10 times higher foliar concentrations of Al, Cr and Ti compared to any other treatments. Plants grown directly in FFT cake were negatively impacted by high water content and low nitrate supply rates. *S. bebbiana* can tolerate and survive in these high metal, saturated soil, and low NO<sub>3</sub><sup>-</sup> conditions while *P. tremuloides* could not. However, adding any soil cap significantly increased aboveground biomass for both species. The capping material that best supported plant growth was a mixture of FFMM and PMM, although differences among soil types were not large. The 5 cm capping depths for PMM and FFMM in *P. tremuloides* had significantly reduced

aboveground biomass, likely caused by the FFT cake's poor draining which resulted in saturated soils. Results from this study show that capping FFT cake at a minimum depth of 10 cm substantially improves woody plant growth, and *S. bebbiana* and *P. tremuloides* are potentially suitable species for tailings reclamation.

In the second study (Chapter 3), biochar was added to one of the capping treatments (1:1 ratio of PMM and FFMM at 10 cm depth over FFT cake) to determine if it had any positive effect on plant growth. The results found that there were no differences between the biochar treatment and the non-biochar treatments.

## Preface

The following thesis is composed of original data generated and analyzed by Ryan Scott Lalonde, with no data having been published at the time of submission. Data from Chapter 2 “Capping Dewatered Oil Sands Fine Fluid Tailings with Salvaged Reclamation Soils at Varying Depths to Grow Woody Plants” was presented in a poster presentation at the 2019 Soil Society of Science of America (SSSA) Annual Meeting in San Diego, California, USA and in an oral presentation at the 2019 Alberta Soil Science Workshop (ASSW) in Calgary, Alberta, Canada. Chapter 2 has been submitted for publication to the Canadian Journal of Soil Science.

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# Table of Contents

<b>Chapter 1 – Introduction</b> .....	<b>1</b>
1.1. Alberta Oil Sands .....	1
1.1.1. <i>Oil Sands Mining</i> .....	1
1.1.2. <i>Environmental Concerns</i> .....	2
1.2. Athabasca Region .....	2
1.2.1. <i>Mining Activities and Disturbance</i> .....	2
1.2.2. <i>Boreal Forests</i> .....	3
1.3. Oil Sands Tailings .....	3
1.3.1. <i>Oil Sands Tailings Defined</i> .....	3
1.3.2. <i>Physical and Chemical Properties</i> .....	4
1.3.3. <i>Tailings Management</i> .....	5
1.4. Reclamation .....	5
1.4.1. <i>Reclamation Practices</i> .....	5
1.4.2. <i>Soil Caps</i> .....	6
1.4.3. <i>Biochar as an Amendment</i> .....	7
1.5. Research Overview .....	8
<b>Chapter 2 – Capping Dewatered Oil Sands Fine Fluid Tailings with Salvaged Reclamation Soils at Varying Depths to Grow Woody Plants</b> .....	<b>9</b>
2.1. Introduction .....	9
2.2. Materials and Methods .....	12
2.2.1. <i>Experimental Design</i> .....	12
2.2.2. <i>Foliar Nutrients</i> .....	14
2.2.3. <i>Soil Properties</i> .....	14
2.2.4. <i>Bioavailable Nutrients</i> .....	15
2.2.5. <i>Statistical Analysis</i> .....	15
2.3. Results .....	16
2.4. Discussion .....	18
2.5. Conclusion .....	21
2.5. Tables and Figures .....	22
<b>Chapter 3 – Adding Biochar to Soil Caps Over FFT Cake</b> .....	<b>28</b>
3.1. Introduction .....	28
3.2. Materials and Methods .....	29
3.3. Results .....	30
3.4. Discussion .....	31
3.5. Conclusion .....	32
3.6. Tables and Figures .....	33
<b>Chapter 4 – Conclusion</b> .....	<b>39</b>
4.1. Study Implications and Recommendations .....	39
4.2. Study Limitations .....	40
4.3. Future Research .....	40
<b>References</b> .....	<b>42</b>
<b>Appendix</b> .....	<b>57</b>

## Table of Figures

<b>Figure 2.1.</b> Diagram of experimental design with different soil materials and capping depths. .	25
<b>Figure 2.2.</b> Aboveground biomass (g) for <i>Salix bebbiana</i> (a) and <i>Populus tremuloides</i> (b). Values represent the means with standard errors (n=6). ANOVA data presented in these figures exclude the zero capping depth controls in its analysis. ....	26
<b>Figure 2.3.</b> Soil volumetric water content for <i>Salix bebbiana</i> (a) and <i>Populus tremuloides</i> (b). Values represent the means with confidence levels (0.95, n=18-24). ....	27
<b>Figure 3.1.</b> Aboveground biomass (g) for <i>Salix bebbiana</i> (a) and <i>Populus tremuloides</i> (b). Values represent the means with standard errors (n=6; p>0.05). ....	36
<b>Figure 3.2.</b> Plant height (cm) for <i>Salix bebbiana</i> (a) and <i>Populus tremuloides</i> (b). Values represent the means with standard errors (n=6; p>0.05). ....	37
<b>Figure 3.3.</b> Average soil volumetric water content for <i>Salix bebbiana</i> (a) and <i>Populus tremuloides</i> (b). Values represent the means with confidence levels (0.95, n=18-24). ....	38
<b>Figure A1.</b> Final plant height (cm) for <i>Salix bebbiana</i> . Values represent the means with standard errors (n=6). ....	58
<b>Figure A2.</b> Final plant height (cm) for <i>Populus tremuloides</i> . Values represent the means with standard errors (n=6). ....	59
<b>Figure A3.</b> Soil water retention curve of different capping materials and tailings cake. ....	60
<b>Figure A4.</b> <i>S. bebbiana</i> growth after 16 weeks grown directly in FFT cake. ....	61
<b>Figure A5.</b> <i>S. bebbiana</i> growth after 16 weeks in 50/50 mix at 5 cm depth. ....	62
<b>Figure A6.</b> <i>P. tremuloides</i> growth after 16 weeks in 50/50 mix at 20 cm depth. ....	63
<b>Figure A7.</b> Dead <i>P. tremuloides</i> grown directly in FFT cake. ....	64

## Table of Tables

<b>Table 2.1.</b> Soil properties of subsoil and capping materials. ....	22
<b>Table 2.2.</b> Soil nutrient data from Plant-Root-Simulator (PRS) probes ( $\mu\text{g } 10 \text{ cm}^{-2} \text{ 28 Days}^{-1}$ ) for top 5 to 10 cm depth. ....	23
<b>Table 2.3.</b> Foliar nutrient concentrations ( $\text{mg g}^{-1}$ ) of <i>Salix bebbiana</i> . ....	24
<b>Table 3.1.</b> Soil properties of subsoil and capping materials. ....	33
<b>Table 3.2.</b> Soil nutrient data from Plant-Root-Simulator (PRS) probes ( $\mu\text{g } 10 \text{ cm}^{-2} \text{ 28 Days}^{-1}$ ). ....	34
<b>Table 3.3.</b> Foliar nutrient concentrations ( $\text{mg g}^{-1}$ ) of <i>Salix bebbiana</i> . ....	35
<b>Table A1.</b> Foliar nutrient concentrations ( $\text{mg g}^{-1}$ ) of <i>Populus tremuloides</i> . ....	57



# Chapter 1 – Introduction

## 1.1. Alberta Oil Sands

### 1.1.1. Oil Sands Mining

Alberta Oil Sands Region (AOSR) - which includes Peace River, Cold Lake and Athabasca regions - represents the third-largest oil reserves in the world (Alberta Energy 2017). In 2018, the AOSR was producing 2.9 million barrels of crude oil per day, of which 20% was recovered by surface mining in the Athabasca region as opposed to in-situ (NRCan 2018). Surface mining is used when the reserves are found within 70 meters of the earth's surface (CAPP 2018). Oil sands occur naturally in the region and are composed of a mixture of bitumen, sand, clay, water and minerals (Alberta Energy 2017). At mining operations, the bitumen is extracted using the Clark hot water extraction process. The oil sands sector is one of the largest employers in the country (NRCan 2018) and is expected to contribute nearly 1.7 trillion dollars to the Canadian economy from 2017 to 2027 (CERI 2017), making it a critical part of Canada's economy. The industry is expected to expand; as the number of barrels produced per day is forecasted to steadily increase in the coming years, with a high case scenario peaking at 5.8 million barrels of crude oil per day by 2039, and a low case scenario still peaking at 4.1 million barrels per day (CERI 2019).

### 1.1.2. Environmental Concerns

There are numerous environmental concerns associated with oil sands development in Alberta. Some of the major environmental concerns associated with oil sands are related to its large carbon footprint, surface water and groundwater consumption, impacts on surface water and groundwater quality, air quality, the feasibility of reclamation, and tailing ponds (Gosselin et al. 2010). Oil sands make up 11% of Canada's greenhouse gas emissions, which represents 0.1% of global emissions (NRCan 2018). The industry uses large volumes of freshwater, where an average of 3 barrels is required to produce every one barrel of oil in surface mining production (CAPP 2018). A large part of this water goes to tailings ponds to allow for suspended fines to settle. Tailings ponds are among the most important environmental concerns regarding the oil sands industry (Azam and Rima 2014) due to their growing volume as well as their physical and chemical properties.

## 1.2. Athabasca Region

### 1.2.1. Mining Activities and Disturbance

Oil sands are underlying 142 200 km<sup>2</sup> of land in the AOSR (Alberta Energy 2017). Only 3% of this area is surface mineable, which represents 4800 km<sup>2</sup> (Alberta Energy 2017) and is found in the Athabasca region of northern Alberta. Over 900 km<sup>2</sup> of land in the Athabasca region has already been disturbed (Alberta Environment and Parks 2017) by surface mining activities. Surface mining creates a destructive disturbance, as it completely removes the top layers of soil (Rowland et al. 2009), which result in irreversible change that leads to different ecosystems (Audet et al. 2015).

### 1.2.2. Boreal Forests

The boreal forest covers massive northern areas in Siberia, Alaska, Europe and Canada (Binkley and Fisher 2013), and extends approximately 5.5 million km<sup>2</sup> (Brandt et al. 2013) in Canada. Within the boreal forest, the Athabasca region lies within the Central Mixwood Subregion (Alberta Parks 2015), a region composed of vast upland forests and wetlands on level to gently undulating plains. This region has annual precipitation of 419 mm and an annual mean temperature of 1°C (Government of Canada 2019), with the majority of the precipitation occurring as rainfall. The region has an average of 97 frost-free days between late May and September (NRC 2006). Common tree species on upland sites are trembling aspen (*Populus tremuloides*), white spruce (*Picea glauca*) and jack pine (*Pinus banksiana*); black spruce (*Picea mariana*) and tamarack (*Larix laricina*) are common trees on peatlands sites (Alberta Parks 2015). Willows can also be found in the region (Kuzovkina and Volk 2009). Fire is a prominent disturbance in these boreal forests and plays an important role in ecosystem processes (Hicke et al. 2003).

## 1.3. Oil Sands Tailings

### 1.3.1. Oil Sands Tailings Defined

Extracting the bitumen through the Clark hot water extraction method produces a fluid slurry stream made up of silts, clays, sand, water and residual bitumen (OSTC 2012). This slurry known as tailings contains suspended solids including sand particles which make up approximately 82 wt%, fine particles such as silts and clays make up approximately 17 wt% and approximately 1 wt% is made up of unrecovered bitumen (Chalaturnyk et al. 2002). The slurry is

discharged into tailings ponds where the larger particles quickly settle, leaving the remaining fine particles to make up what is defined as fine fluid tailings (FFT), containing between 2-15 wt% solids content (Chalaturnyk et al. 2002). After a few years, finer particles settle and the tailings reach 30 wt% solids content and are then known as mature fine tailings (MFT) (Owolagba and Azam 2017). Between 75-80% is considered the ideal solids content to develop long term stability and stiffness (OSTC 2012), however MFT has very slow consolidation rates which result in tailing ponds of MFT remaining in a fluid state for decades (Kasperski 1992).

### 1.3.2. Physical and Chemical Properties

Tailings have a large content of clay minerals, and although the amounts and types can vary significantly, typically MFT is mostly kaolinite and illite (Chalaturnyk et al. 2002). Fine tailings have high water holding capacity and very poor dewaterability (Chalaturnyk et al. 2002) due to naphthenic acids present in the bitumen as well as the clay and silt presence. Studies have found tailings to include heavy metals such as Al, Fe, Mg, Mn, Ti, V and Zr (Mikula et al. 1996). There is also a significant presence of inorganic salts and phytotoxic organic compounds (such as naphthenic acids), which is one of the primary environmental concerns related to MFT (FTFC 1995). Tailings material can also have high salinity which can result in inhibited water uptake and reduction in plant growth (Apostol et al. 2004). Although there is still a lack of understanding on how naphthenic acids impact plants, some studies suggest they can be toxic to plants and also to a vast array of other flora and fauna (Brown and Ulrich 2015). Tailings are also considered to be slightly basic (BGC 2010a), and generally nutrient-poor (Alkio et al. 2005).

### 1.3.3. Tailings Management

MFT is stored in large tailings storage ponds which currently hold more than 975 million m<sup>3</sup> of tailings (Alberta Environment and Parks 2015). The very slow settling rates and growing volume of tailings ponds are among the most important environmental concerns regarding the oil sands industry (Azam and Rima 2014). These storage ponds are susceptible to leakage (Wang et al. 2014) and can cause environmental harm to surrounding water quality, ecology and wildlife (Singh 2004; Timoney and Ronconi 2010). For this reason, there exist several technologies to try and improve current tailings management issues, with one report reviewing 34 existing technologies (BGC 2010b).

Many of the new technologies focus on dewatering and consolidation of tailings to increase solids content, through mechanical, natural or chemical processes (Utting et al. 2017). Some of the dewatering technologies include natural drying, deep in-pit deposition (rim ditching), filtration and centrifugation (Wang et al. 2014). Of these methods, dewatering by centrifugation has shown very promising results (Azam and Rima 2014) and has potential environmental benefits such as saving water for reuse (Mikula et al. 2009). Centrifugation uses flocculation and the addition of coagulant such as gypsum, followed by processing through a solid-bowl scroll centrifuge (OSTC 2012).

## 1.4. Reclamation

### 1.4.1. Reclamation Practices

Oil sands surface mining involves a massive disturbance to entire ecosystems by removing all vegetation, soil and overburden to remove the oil sand (Rowland et al. 2009). According to Government of Alberta regulations, land disturbed by surface mining activity must be reclaimed to “equivalent land capability” (Alberta Government 2005), which requires the re-establishment of a self-sustaining ecosystem, made up of native species (Alberta Environment and Sustainable Resource Development 2013). So far, of the more than 900 km<sup>2</sup> of disturbed land, only 1.0 km<sup>2</sup> of it has been certified reclaimed (Alberta Environment and Parks 2017), although 50.6 km<sup>2</sup> of land is categorized as “permanent terrestrial reclamation”. The majority of this land is being reclaimed to upland forests (Mackenzie and Naeth 2010), however, 64% of the landscape was previously supporting wetlands before oil sands disturbance (Rooney et al. 2012). For this reason, the primary reclamation material used in these restored upland forests is peat soils (Rowland et al. 2009). The severity of disturbance is so large that ecosystems remain different than natural undisturbed forests even 20 years after reclamation (Dhar et al. 2018). The difference is even more pronounced on sites that used peat (Dhar et al. 2018) compared to sites that used soil sourced from upland forests.

#### 1.4.2. Soil Caps

It’s important to establish a minimum capping depth for successful reclamation to avoid mixing of the materials as well as create a buffer (Alberta Environment and Water 2012), while not overusing the scarce supply of salvaged soils. There are several studies that highlight the positive effects of adding a capping soil in oil sands or tailings reclamation (Kelln et al. 2008; Huang et al. 2015; Barbour et al. 2007; Hargreaves et al. 2012; Luna Wolter and Naeth 2014; Zettl et al. 2011) by improving soil conditions to support plant growth and increase water

storage. In the mineable oil sands region, capping soils are salvaged from the mining process. The two primary soil types salvaged for cover soils are upland forest floor mineral mix soils (FFMM) and wetland peat mineral mix soils (PMM) (Pinno and Errington 2015).

#### 1.4.3. Biochar as an Amendment

Fire is a prominent disturbance in most of the world's boreal forests and plays an important role in the carbon cycle and ecosystem processes (Hicke et al. 2003). Fires in boreal regions mostly convert biomass to gaseous forms of carbon (CO<sub>2</sub>), however up to 7 percent is converted to pyrogenic carbon (Preston and Schmidt 2006). Masiello (2004) describes pyrogenic carbon as being part of a continuum, which includes all carbon-rich residues from fire, such as charcoal, soot and polycyclic aromatic hydrocarbons (Lynch et al. 2004). Black carbon (BC), more specifically describes pyrogenic carbon that is chemically resistant to oxidation (Preston and Schmidt 2006). BC is more resistant to decay than other forms of carbon found in soils (Lehmann 2007). BC has positive effects on soil properties, such as decreasing bulk density and increasing water retention (Busscher et al. 2003), increasing cation exchange capacity (Qiu et al. 2008) and increasing adsorption of cations, which can reduce nutrient leaching (Laird et al. 2010) and immobilize contaminants (Qi et al. 2017). BC can also play a major role in carbon cycling, including carbon sequestration in soils and atmospheric emissions (Bélanger and Pinno 2008; Ding et al. 2015). Furthermore, BC has a significant impact on soil microorganisms, changing bacterial community composition which can affect total soil CO<sub>2</sub> fluxes, thus having a potential influence on the carbon cycle (Whitman et al. 2016).

Despite the increasing evidence that supports the importance of BC in boreal soil function and carbon storage, it is often overlooked as a meaningful component of soils (Hart and

Luckai 2013). Black carbon is essentially ubiquitous in boreal forest soils, therefore, adding it to reclamation soils should have management applications, however isn't currently standard practice in the industry (Mackenzie et al. 2014). Biochar is a manufactured form of pyrogenic carbon, and Thomas and Gale (2015) found evidence for its potential benefits in forest restoration. Similar to black carbon, biochar can improve soil properties, and increase early tree growth response, especially in angiosperms in boreal forests. The evidence, however, is highly variable and more studies are required.

## 1.5. Research Overview

The study investigates the potential use of dewatered tailings in dry landscape restoration. The study used FFT cake (FFT dewatered by centrifugation) as a subsoil material with soil caps above it and grew different woody plant species in a greenhouse experiment.

In Chapter 2, the research objective is to determine how different capping soils (FFMM, PMM and 50/50 Mixture) at different capping depths (0, 5, 10 and 20 cm) placed above FFT cake will impact woody plant growth. The findings will contribute to determining a minimum optimal capping depth for each capping soil, as well as determine species ability to grow in FFT.

Chapter 3 provides supplementary data to Chapter 2 and is not meant to be read as a stand-alone study. It is a sub-experiment which complements the principal findings. The objective of this sub-study builds on Chapter 2 by investigating how biochar could potentially benefit plant growth when added as an amendment in the capping soil placed above FFT cake.



# Chapter 2 – Capping Dewatered Oil Sands Fine Fluid Tailings with Salvaged Reclamation Soils at Varying Depths to Grow Woody Plants

## 2.1. Introduction

Mining from the Athabasca oil sands in northern Alberta produces approximately 20% of the country's oil supply (Alberta Energy 2017). Surface mining uses the Clark alkaline hot water extraction process which produces 3 m<sup>3</sup> of tailings for every barrel of bitumen, which are then transported to tailings ponds (Voordouw 2013). Tailings storage ponds are holding more than 975 million m<sup>3</sup> of fine fluid tailings (Alberta Environment and Parks 2015), which are composed of a mixture of water, sand, fine silts and clays, and residual bitumen. Tailings are generally nutrient-poor (Alkio et al. 2005), have a slightly basic pH and take years to settle (BGC 2010a). Fine fluid tailings (FFT) are defined as containing between 2-15% solids content and mature fine tailings (MFT) are defined as having greater than 30% solids content (OSTC 2012). The growing volume of tailings ponds is among the most important environmental concerns regarding the oil sands industry (Azam and Rima 2014). These fluid tailings have negative impacts on surrounding water quality, ecology and wildlife (Singh 2004; Timoney and Ronconi 2010; Wang et al. 2014), due to the presence of phytotoxic organic compounds and inorganic ions, such as naphthenic acids, trace metals and residual bitumen (FTFC 1995).

Tailings slurries can potentially have an important use in land reclamation, however, must be dewatered before it can be used (Badiozamani and Askari-Nasab 2014). There are several dewatering technologies such as natural drying, deep in-pit deposition (rim ditching), filtration and centrifugation (Wang et al. 2014). Of these methods, dewatering by centrifugation

has shown very promising results (Azam and Rima 2014) and has potential environmental benefits such as saving water for reuse (Mikula et al. 2009). Once dewatered, the FFT cake must be capped with suitable soil material before reclamation.

Plants do not grow well directly in oil sands tailings (Renault et al. 2004; Luna Wolter and Naeth 2014). Boldt-Burisch et al. (2018) grew slender wheatgrass (*Elymus trachycaulus*) and bird's-foot trefoil (*Lotus corniculatus*) in dry MFT, coarse tailings sand (CTS), and in sandy soil, and found that both species had reduced growth in both the MFT and CTS compared to the sandy soil. Renault et al. (2000) also found that seedling survival in conifers was reduced to as low as 55% when grown directly in dry MFT. For this reason, adding a capping material plays an important role in reclamation by improving plant growth (Hargreaves et al. 2012; Luna Wolter and Naeth 2014; Huang et al. 2015), increasing water storage (Zettl et al. 2011) and improving soil conditions to support self-sustaining ecosystems.

In the mineable oil sands region, the two primary soil types salvaged for cover soils in reclamation are upland forest floor mineral mix soils (FFMM) and wetland peat mineral mix soils (PMM) (Pinno and Errington 2015). Some studies suggest FFMM provides a more suitable material than PMM for supporting plant growth (Mackenzie and Naeth 2010; Jamro et al. 2014; Luna Wolter and Naeth 2014; Pinno et al. 2017; Dietrich and MacKenzie 2018) since FFMM is typically richer in nutrients. Other studies found PMM to be well suited to support aspen growth as it is higher in organic matter and has a greater water holding capacity (Pinno et al. 2012; Pinno and Errington 2015). Combining the two soils could have benefits for supporting plant growth (Mackenzie and Naeth 2010; Pinno et al. 2017). Overall, both soils are generally good reclamation materials to use as cover soils, however PMM is more commonly used (Rowland et

al. 2009) due to greater availability. It is less understood what influence these reclamation soils would have on tree and shrub growth when used as a capping material over dewatered tailings.

It's important to establish a minimum capping depth for successful reclamation to avoid mixing of the materials and create a buffer (Alberta Environment and Water 2012). When using cover soils above overburden, Kelln et al. (2008) found that a minimum cover thickness of 50 cm was necessary for long-term tree growth, while Huang et al. (2015) found that thickness exceeding 100 cm no longer had significant differences on growth even after 60 years. Barbour et al. (2007) also indicated a minimum capping depth for overburden or tailing sands in a conservative reclamation scenario to be 30 cm of cover soil over 50 to 70 cm of mineral soil. However, current practice is for operators to only place between 10 and 50 cm of cover soil over suitable overburden and between 20 and 50 cm over tailing sands due to limited availability of reclamation material (Alberta Environment and Water 2012). For this reason, it's important to determine the minimum recommended capping depth for other materials such as dewatered fine tailings, which is not well understood.

Re-establishment of native tree species such as trembling aspen is a critical part of rebuilding soil health and ecosystem function for upland reclamation sites (Sorenson et al. 2011; Dhar et al. 2018), due to its rapid growth and contribution to generating and establishing forest understory, as well as above and belowground organic matter. Species such as willow (*Salix* sp.) are also becoming increasingly important in land reclamation as they have significant potential for phytoremediation and ecosystem restoration (Kuzovkina and Quigley 2005), have a high tolerance to stress (Kuzovkina and Volk 2009), and are suitable to both wet and dry conditions (Mosseler et al. 2014). Willows also have a high tolerance for heavy metal uptake (Sawidis et al., 2001; Robinson et al., 2000), but this can vary greatly between species (Boyter et al. 2009).

Mosseler et al. (2014) suggested *Salix bebbiana* should be further selected for use in coal mine reclamation but hasn't been extensively studied for growth tolerance when grown on oil sands tailings.

The research objective is to determine how different capping soils (FFMM, PMM and Mixture) and depths (0, 5, 10, 20 cm) above FFT cake affect woody plant growth. This will allow me to determine a minimum capping depth for each capping soil type, species tolerances to growing directly in FFT, and better understand the impact FFT cake has on woody plants.

## 2.2. Materials and Methods

### 2.2.1. Experimental Design

Beaked willow (*Salix bebbiana* Sarg.) and trembling aspen (*Populus tremuloides* Michx.) were grown for 16 weeks in pots layered with centrifuged fine fluid tailings cake (FFT Cake) as a subsoil material on the bottom and covered with different reclamation materials as capping soils. The study had a completely randomized design with 2 plant species (aspen and willow), 3 capping soils (FFMM, PMM and Mixture), 4 cap depths (5, 10, 20 cm + no cap treatment), 6 replicates, for a total of 120 pots (Figure 2.1). The 0 cm capping depth treatment (no cap) was not repeated 3 times for each soil type. The three capping soils used were forest floor mineral mix (FFMM), a typically upland forest soil that is predominantly mineral soil mixed with surface organic matter; peat mineral mix (PMM), a roughly even mix of organic-based wetland or lowland soil and mineral soil ; and a 50/50 mixture, containing 50% FFMM and 50% PMM by volume (50/50 Mix). The 50/50 Mix was blended in a concrete mixer for homogeneity. Soil cap depths were 0 cm, 5 cm, 10 cm and 20 cm. The pots were 10.1 x 10.1 cm at the top, 35.6 cm tall, with an actual volume of 2.83 L. The plants grew from May to September. Pots were rotated

twice per week. The greenhouse was set to 25°C during the day and 18°C at night with a daily photoperiod of 16 h. Plants were watered daily with municipal water through an automated sprinkler irrigation system. The irrigation was set to two 1-minute pulses of water, 4 times per day (every 6 hours). Excess water from flooded pots was removed daily using a plastic baster. Fertilization was not used during the experiment other than before transplanting.

*S. bebbiana* and *P. tremuloides* were germinated and grown from seed for eight weeks in styrofoam propagation trays filled with peat potting soil. *S. bebbiana* seeds were collected in spring 2017 from natural occurring shrubs located near Cremona, Alberta (51°30'N, 114°28'W, Eastern Slopes Range Seeds Ltd.). *P. tremuloides* seeds were sourced from Sheffield's Seed Company (Locke, New York) and were collected in Utah in 2014 (Seed Lot: 1820359-FH-4-A10). These were the only aspen seeds available in the inventory onsite as local seeds failed to germinate. After sowing, the trays were watered then covered in plastic for 2 weeks. After germination, the seedlings were fertilized to saturation every 14 days with 1 g L<sup>-1</sup> of 30-10-10 NPK fertilizer (Plant-Prod Ultimate). Sixty seedlings of each species were selected to be transplanted based on average and uniform size, excluding the smallest and largest ones. The seedlings were roughly 5 cm in height when they were transplanted.

Centrifuged tailings cake (FFT Cake) is fluid fine tailings (FFT) that has been dewatered using a centrifuge at CanmetENERGY in Devon, Alberta. This original material contained 27% solids and came from an oil sands mine in northern Alberta. The fine fluid tailings were dewatered to produce a substrate similar to what is produced by oil sands operators. The fine fluid tailings were treated with 1.1 g L<sup>-1</sup> of A3338 polymer and 1.1 g L<sup>-1</sup> of gypsum before being centrifuged, which increased the percent solids to 55.7%. The capping soil materials were

stockpiled reclamation soils sourced from an oil sands mining site and stored in a warehouse for a year before use in this study.

*S. bebbiana* and *P. tremuloides* seedlings were transplanted into layered treatment pots and grown for 16 weeks. The plant stems were then cut at the soil surface, aboveground plant material was placed in an oven at 60°C for 7 days, and then weighed to obtain aboveground biomass. Seedling deaths were also recorded and the survival rate was calculated at the end of the study.

### 2.2.2. Foliar Nutrients

To determine foliar nutrients, dried *S. bebbiana* leaf tissues were sent for microwave digestion and metal quantification. Prior to digestion, the samples were cryo-milled and sieved. The samples of plants grown directly in FFT Cake were manually sized instead of cryo-milled and sieved, due to their small sample size. Dried, cryo-milled samples (0.25 g) were weighed and pre-digested with 9 mL of concentrated, trace-metals grade nitric acid and 1 mL of concentrated, trace-metal grade hydrochloric acid. Microwave digestion was then performed (CEM Mars 6 digestion unit), followed by metal analysis using ICP-QQQ (Agilent 800).

### 2.2.3. Soil Properties

Initial properties of each soil type are given in Table 2.1. Electrical conductivity and pH of FFMM and FFT cake were measured in a soil water ratio of 1:2 (Hendershot et al., 2007), using a VWP pH/EC meter (sympHony H30PCO), while the PMM and 50/50 mixture were measured in a soil water ratio of 1:5. Field capacity was determined for each soil type using the pressure plate method (Campbell 1974). Two soil samples for each soil type were placed in soil rings, soaked to saturation over 24 hours, and then pressurized at 10 kPa for 48 hours. Afterward,

the soil rings were weighed then dried in an oven at 105°C for 48 hours and weighed again to determine gravitational water content at field capacity. Bulk density was calculated and used to calculate volumetric water content at field capacity. Volumetric water content (VWC%) in each pot was measured every four weeks using a handheld time-domain reflectometer (Field Scout TDR100, Spectrum Technologies Inc.) with 7.6 cm probes.

#### 2.2.4. Bioavailable Nutrients

Supply rates of bioavailable nutrients were measured using Plant-Root-Simulator (PRS) probes (Western Ag Innovations, Saskatoon, Saskatchewan), which have ion-exchange resin membranes. Two pairs of anion and cation probes were installed in control pots with no plants for each treatment (3 capping soil types, 4 cap depths, 3 replicates, no plant). The probes were installed in the top 5 to 10 cm of soil, ensuring contact between the resin membrane and the capping soil. After 28 days the probes were removed, rinsed with deionized water, and sent to Western Ag Innovations for analysis. An extraction of the absorbed ions was done using a 0.5 M HCl solution, then NO<sub>3</sub><sup>-</sup> and NH<sub>4</sub> was analyzed by colorimetry using an automated flow injection analysis system (FIALab 2600), and analyzed P, K, S, Ca, Mg, Fe, Zn, B and Al by inductively coupled plasma optical emission spectrometry (Perkin Elmer ICP-OES 8300).

#### 2.2.5. Statistical Analysis

All data analysis was done using R software (version R.3.1.1, R Core Team 2019). First, a permutational ANOVA (permANOVA) using *lmPerm* package (version 2.1.0; Wheeler and Torchiano 2016) was conducted to determine the overall capping soil type and depth effect on aboveground biomass of *S. bebbiana* and *P. tremuloides*. To better understand the effect that different capping soils and their depths had on plant biomass, we also ran a multi-factor analysis of variance ANOVA (*anova* command) using all the treatments with soil caps (i.e. excluding the

0 cm capping depth). Tukey's HSD Post Hoc test ( $\alpha < 0.05$ ) was then applied to determine significant differences in aboveground biomass among treatments. Second, permANOVA was also performed with Tukey's HSD Post Hoc test to determine if significant biomass differences among treatments were affected by nutrient supply rates, VWC% or foliar nutrients.

### 2.3. Results

The aboveground biomass of *S. bebbiana* (Figure 2.2a) ranged from 0.03 g to 12.82 g among all treatments while *P. tremuloides* (Figure 2.2b) ranged from 0.04 g to 6.35 g. Adding a soil cap had a positive effect on growth, as plants grown directly in FFT Cake did not exceed an aboveground biomass of 0.33 g, which was lower than any other treatment ( $p < 0.001$ ; Figure 2.2). The average aboveground biomass of *S. bebbiana* grown directly in FFT Cake was 0.16 g and for *P. tremuloides* was 0.07 g. After 16 weeks of growth, the survival rate of *S. bebbiana* was 100% for all treatments while *P. tremuloides* survival was 16.7% in FFT Cake, 66.7% in PMM for 5 cm depth, and 100% for all other treatments.

Among capping soil types, *S. bebbiana* biomass was overall lower in PMM ( $p < 0.03$ ), but there were no significant differences between FFMM and 50/50 Mix treatments ( $p = 0.658$ ) (Figure 2.2a). *P. tremuloides* biomass was greater in the 50/50 Mix treatment ( $p < 0.03$ ), but there were no significant differences between FFMM and PMM treatments ( $p = 0.747$ ) (Figure 2.2b).

Among soil cap depths of 5, 10 and 20 cm, there were no significant differences in biomass for *S. bebbiana* ( $p = 0.176$ ; Figure 2.2a). However, there was a soil  $\times$  depth interaction for *P. tremuloides* ( $p = 0.013$ ; Figure 2.2b) with 5 cm depths in PMM and FMM having lower biomass than the other treatments.



Volumetric water content in the top 7.6 cm depth (VWC; Figure 2.3) for *S. bebbiana* soils ranged from 12.1% to 66.1% among all treatments and for *P. tremuloides* soils ranged from 5.4% to 71.4%. Soil moisture had a negative correlation with soil cap depths, with VWC increasing as cap depth decreased (Figure 2.3;  $p < 0.001$ ). The average VWC in the FFT Cake treatment for *S. bebbiana* was 61.8% and for *P. tremuloides* was 63.2%, exceeding field capacity (FC=46.2%) for that substrate. Average VWC also exceeded their respective field capacities in PMM soils (FC=37.9%) for both species at the 5 cm capping depth suggesting that these treatments were regularly saturated.

In the top 5 to 10 cm, nutrient supply rates (Table 2.2) in FFT Cake were below detectable amounts for nitrate, while every other soil type had nitrate supply rates greater or equal to  $22.8 \mu\text{g } 10 \text{ cm}^{-2} 28 \text{ days}^{-1}$ . Total nitrogen supply rates in the capping soils were mostly made up by nitrate (Table 2.2). Among soil cap depths, nutrient supply rates for nitrate increased with depth ( $p \leq 0.018$ ).

FFT cake was also lower in calcium ( $p < 0.001$ ) than any other treatment, higher in potassium ( $p < 0.001$ ) and boron ( $p \leq 0.018$ ) than any other treatment, and below the detectable limits for zinc). When comparing the soil types, FFMM had higher nutrient supply rates than PMM for nitrate, magnesium and iron ( $p \leq 0.004$ ), lower for sulfur ( $p < 0.001$ ), and no differences between nutrient supply rates for phosphorus, boron, calcium, aluminum and zinc ( $p > 0.307$ ). Potassium supply rates were not different among soil types ( $p \geq 0.088$ ). Nutrient supply rates for 50/50 Mix soils fell in the range between PMM and FFMM (Table 2.2). Capping depth had no effect on nutrient supply rates for B, Ca, Mg, Al and S ( $p \geq 0.120$ ).

Leaf tissues of *S. bebbiana* grown in the FFT Cake controls had higher concentrations of aluminum, boron, chromium and titanium (Table 2.3) ( $p < 0.001$ ) than in any other treatment. For Al, Cr and Ti, the concentration differences between FFT Cake and all other treatments were about ten-fold, with Al concentrations of  $3044 \text{ mg g}^{-1}$ , Cr concentrations of  $48 \text{ mg g}^{-1}$ , and Ti concentrations of  $63 \text{ mg g}^{-1}$ . These trace metals did not have significant differences among the other soil types or capping depths. Foliar concentrations for other nutrients and metals did not have meaningful differences among treatments.

## 2.4. Discussion

For plants grown directly in FFT cake, growth was significantly less than compared to plants grown in a capping soil. *S. bebbiana* seedlings did survive throughout the 16-week study when grown directly in FFT cake, but there was very little increase in biomass. *P. tremuloides* seedlings had much lower survival rates (16.7%) when grown directly in the FFT cake, which indicates *P. tremuloides* are more sensitive to soil substrate compared to willow. The FFT cake is therefore not a suitable medium for survival or growth of *P. tremuloides* seedlings. These findings align with previous studies (Renault et al. 2000; Renault et al. 2004; Luna Wolter and Naeth 2014; Boldt-Burisch et al. 2018) which also found reduced plant biomass when grown directly in dewatered tailings.

Adding a capping soil had a positive effect on aboveground biomass among all soil types and capping thicknesses. Luna Wolter and Naeth (2014) also found an increase in plant biomass when growing grasses in dry mature fine tailings with soil caps. For *S. bebbiana* there were no significant differences in biomass between the 5, 10 and 20 cm capping depths, and for *P*

*tremuloides* there were no significant differences in biomass between 10 and 20 cm. Crop yields have been observed to increase with greater capping depths over mine waste materials over time (Sydnor and Redente 2000; Bowen et al. 2005), however increasing capping depth may not have a large impact on short term growth. Bockstette (2018) found that capping depth had little effect on early seedling growth, which may explain why no significant differences were found among most capping depths.

In terms of capping soil type, 50/50 Mix produced slightly higher aboveground biomass in both species, while PMM had the lowest aboveground biomass in both species. FFMM had similar results to 50/50 mix for *S.bebbiana*, and similar results to PMM for *P.tremuloides*. This aligns with several studies that favor mineral-based FFMM over organic matter based PMM as a soil cover in oil sands reclamation (Mackenzie and Naeth 2010; Jamro et al. 2014; Dietrich and MacKenzie 2018), as well as other studies suggesting mixing the two reclamation soil types could be beneficial for reclamation (Mackenzie and Naeth 2010; Pinno et al. 2017). Luna Wolter and Naeth (2014) found that forest floor mineral mix caps placed above dry MFT resulted in a 30% increase in aboveground biomass in native grasses, nearly double than the increase associated with PMM caps. Pinno and Errington (2015) found that initial aspen growth response was greater in PMM cover soils, however once established there were no significant seedling height differences between PMM and FFMM. These studies support our findings that FFMM and a mixture of FFMM and PMM are as good or a better capping soil as PMM alone.

Important factors influencing poor plant growth in FFT cake were nitrogen deficiencies, high water content, and high salt and metal concentrations. It is unclear from this study as to the role of dissolved organics on plant growth. Nitrate levels weren't at detectable levels in the tailings, which likely limited plant growth, so capping the FFT provided nutrients for the plants.

Volumetric water content generally increased among all soils as capping thickness decreased with the average volumetric water content in FFT Cake regularly greater than field capacity. Studies have found very low hydraulic conductivity associated with dewatered tailings materials (Jeeravipoolvarn 2010; Owolagba and Azam 2015, 2017), typically classed as a clay with plasticity which has very poor drainage. For the other treatments, the saturated FFT cake prevented the soils above from draining properly with the smaller capping depths more impacted by this effect. For example, the 5 cm depths for PMM and FFMM had high average volumetric water contents for *P. tremuloides*, and were associated with reduced aboveground biomass. This likely impacted *P. tremuloides* growth more than *S. bebbiana* due to aspen's susceptibility to high soil moisture (Kay 1993) and willows ability to grow in high water content soils (Kuzovkina and Quigley 2005). Although there wasn't a significant effect on seedling growth for any capping depth greater than 5 cm, capping depth may have a greater effect over long term plant growth. Huang et al. (2015) found that over a period of 60 years, capping depth incrementally impacted tree growth up to 100 cm in depth.

*S. bebbiana* had a much higher tolerance for growing in oil sands tailings than *P. tremuloides*. The seedling survival rate of willow was 100% after 16 weeks of growth. Foliar concentrations of aluminum, chromium and titanium were nearly ten times higher than any other treatments and would be considered higher than normal concentrations and even toxic in other land plant species (Macnicol and Beckett 1985; Nagajyoti et al. 2010). This supports the literature saying willows are good heavy metal accumulators (Robinson et al. 2000; Sawidis et al. 2001). However, metal accumulation in woody species are more concentrated in the roots (Kelly et al. 1990; Riddell-Black 1994), so it is likely that concentrations would be even higher in the *S. bebbiana* roots than in the leaves. Even with such high metal concentrations, the plants

were able to survive throughout the study, which indicates *S. bebbiana*'s tolerance to growing in tailings and its potential use for oil sands reclamation. Mosseler et al. (2014) also found *S. bebbiana* to be a potentially promising species for metal uptake in coal mine reclamation. Treatments with a soil cap did not have observably high concentrations of any trace metals. Adding any capping soil regardless of depth reduced foliar concentrations of Al, Cr and Ti. Hargreaves et al. (2012) had similar findings in a study that added a cap of organic residuals over mine tailings containing nickel and copper and showed no evidence of metal movement over the course of the 2-year study. Evaluating *S. bebbiana*'s trace metal accumulation in the roots should be further investigated in future studies.

## 2.5. Conclusion

Understanding these findings gives us insight into future management practices in oil-sands reclamation. Our study found that even after centrifuge dewatering, high volumetric water content in FFT was still an issue affecting plant growth. Adding a capping soil is essential to seedling establishment. Waterlogging can be an issue in shallower capping depths, especially for water sensitive species such as aspen. While this research provides insights into capping depth thicknesses, this needs to be studied further as the required cap thickness likely increases as roots establish. This study suggests capping soil type isn't a critical factor for early plant growth, however a mixture of FFMM and PMM did produce slightly greater yields. Both species show promising results as a reclamation species in dry tailings reclamation and should be further investigated.

## 2.5. Tables and Figures

**Table 2.1.** Soil properties of subsoil and capping materials.

<b>Soil Description</b>	<b>pH</b>	<b>Electrical Conductivity (dS m<sup>-1</sup>)</b>	<b>Bulk Density (g cm<sup>3</sup>)</b>	<b>Field Capacity (% water content)</b>
FFT Cake	8.13 (0.19)	1.12 (0.05)	0.95 (0.06)	46.2
PMM	3.99 (0.06)	0.72 (0.04)	0.60 (0.10)	37.9
FFMM	5.59 (0.03)	0.30 (0.02)	1.16 (0.14)	42.2
50/50 Mix	3.98 (0.01)	0.61 (0.01)	0.84 (0.12)	39.1

N=2-8

Values represent the means with standard deviation in brackets.

**Table 2.1.** Soil nutrient data from Plant-Root-Simulator (PRS) probes ( $\mu\text{g } 10 \text{ cm}^{-2} \text{ 28 Days}^{-1}$ ) for top 5 to 10 cm depth.

	<b>NO<sub>3</sub><sup>-</sup></b>	<b>NH<sub>4</sub></b>	<b>P</b>	<b>K</b>	<b>Ca</b>	<b>Mg</b>	<b>Al</b>	<b>B</b>	<b>Fe</b>	<b>S</b>	<b>Zn</b>
Method											
Detection Limits	2	2	0.2	4	2	4	0.04	0.2	0.4	2	0.2
<b>FFT cake</b>											
0 cm	<	9.45 (2.7)	2.10 (0.5)	57.1 (4.2)	1364 (54)	442 (22)	23.3 (4.3)	2.27 (0.3)	94.2 (18)	2642 (52)	<
<b>PMM</b>											
5 cm	22.8 (11)	2.30 (0.9)	2.52 (0.8)	29.8 (2.2)	1850 (30)	333 (6)	22.0 (1.6)	1.23 (0.1)	41.1 (14)	2599 (23)	0.61 (0.1)
10 cm	75.9 (21)	6.70 (1.9)	1.33 (0.1)	26.6 (3.1)	2175 (99)	363 (17)	29.8 (0.4)	1.20 (0.2)	32.3 (10)	2963 (85)	1.54 (0.1)
20 cm	44.4 (21)	6.18 (1.4)	1.37 (0.1)	13.0 (2.8)	1949 (25)	323 (15)	22.7 (3.7)	0.78 (0.4)	26.7 (5)	2958 (99)	1.31 (0.1)
<b>FFMM</b>											
5 cm	133.0 (8)	<	3.45 (1.2)	37.3 (4.7)	1705 (149)	424 (40)	19.2 (6.1)	1.68 (0.5)	121.1 (13)	2295 (34)	0.38 (0.2)
10 cm	380.3 (76)	<	1.52 (0.0)	27.0 (2.9)	1883 (82)	480 (24)	19.6 (2.0)	0.94 (0.4)	40.1 (7)	1544 (97)	1.56 (0.3)
20 cm	586.3 (75)	<	1.64 (0.1)	24.8 (1.4)	2026 (80)	516 (25)	19.5 (0.4)	0.79 (0.3)	72.0 (19)	1406 (12)	2.20 (0.6)
<b>50/50 Mix</b>											
5 cm	112.9 (11)	<	3.05 (0.6)	42.2 (3.7)	2034 (41)	454 (17)	18.1 (7.4)	1.13 (0.1)	82.3 (21)	2587 (85)	0.36 (0.0)
10 cm	118.4 (26)	<	2.05 (0.8)	29.6 (1.5)	1917 (120)	415 (32)	31.2 (1.3)	1.32 (0.3)	36.9 (13)	2386 (130)	0.55 (0.1)
20 cm	378.5 (36)	<	1.44 (0.0)	21.5 (4.0)	1985 (89)	434 (42)	27.0 (1.8)	0.90 (0.2)	32.5 (4)	1998 (79)	0.89 (0.1)
<b>P values</b>											
Soil	< 0.01	< 0.01	0.730	< 0.01	< 0.01	< 0.01	0.135	< 0.01	< 0.01	< 0.01	< 0.01
Cap Depth	< 0.01	0.252	< 0.01	< 0.01	0.141	0.592	0.079	0.130	< 0.01	0.194	< 0.01
Soil x Cap	< 0.01	0.253	0.983	0.394	0.034	0.172	0.398	0.686	0.123	0.091	0.076

N=3

Values represent the means with standard errors in brackets. Values below the detection limit are represented by '<'.

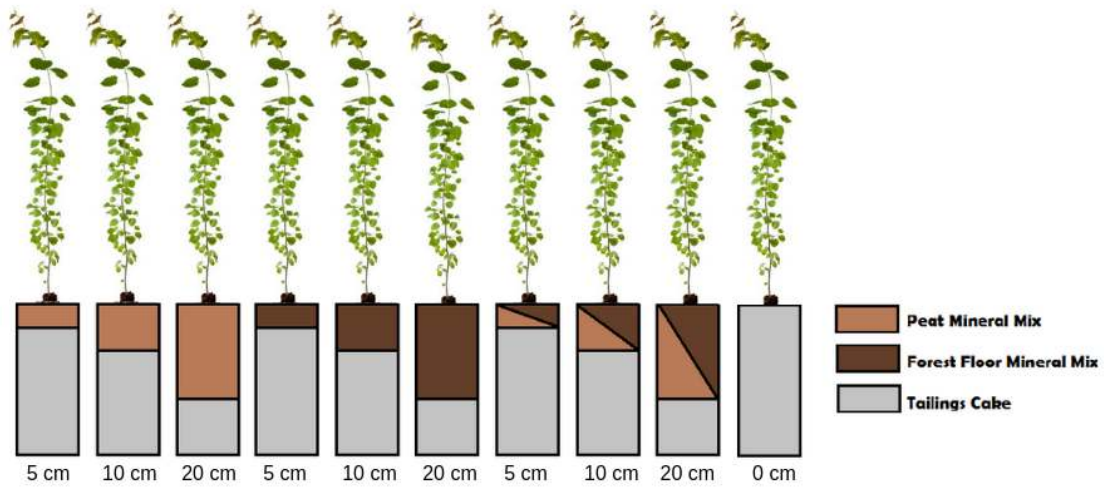
**Table 2.3.** Foliar nutrient concentrations ( $\text{mg g}^{-1}$ ) of *Salix bebbiana*.

	<b>Al</b>	<b>B</b>	<b>Ca</b>	<b>Cr</b>	<b>Fe</b>	<b>K</b>	<b>Mg</b>	<b>P</b>	<b>S</b>	<b>Ti</b>	<b>Zn</b>
<b>FFT cake</b>											
0 cm	<b>3044</b> (1179)	<b>441</b> (48)	$1.23 \times 10^4$ (1077)	<b>47.8</b> (30.9)	989 (392)	$1.41 \times 10^4$ (1531)	3880 (333)	824 (77)	3135 (134)	<b>63.1</b> (26.6)	135 (29)
<b>PMM</b>											
5 cm	471 (210)	321 (40)	$1.57 \times 10^4$ (1343)	4.4 (0.7)	587 (118)	$1.57 \times 10^4$ (2508)	4707 (360)	1995 (319)	2964 (341)	10.0 (4.0)	247 (71)
10 cm	392 (256)	300 (49)	$1.59 \times 10^4$ (889)	5.6 (3.4)	591 (167)	$1.51 \times 10^4$ (2144)	4352 (219)	1531 (156)	2606 (165)	8.1 (4.7)	176 (30)
20 cm	261 (125)	240 (25)	$1.89 \times 10^4$ (992)	2.8 (1.0)	671 (242)	$1.68 \times 10^4$ (776)	5157 (296)	2337 (239)	3135 (206)	7.0 (3.3)	377 (80)
<b>FFMM</b>											
5 cm	293 (124)	327 (18)	$1.48 \times 10^4$ (1194)	2.8 (0.7)	185 (35)	$1.81 \times 10^4$ (643)	4407 (396)	1731 (248)	2635 (385)	7.1 (2.4)	73 (9.9)
10 cm	152 (65)	189 (20)	$1.20 \times 10^4$ (510)	1.7 (0.3)	121 (27)	$1.27 \times 10^4$ (1305)	3352 (136)	954 (86)	2015 (115)	2.1 (0.6)	84 (7.3)
20 cm	241 (74)	149 (11)	$1.51 \times 10^4$ (1050)	1.9 (0.4)	161 (40)	$1.54 \times 10^4$ (1010)	4933 (323)	1349 (107)	2404 (307)	6.6 (1.9)	147 (16)
<b>50/50 Mix</b>											
5 cm	214 (71)	367 (54)	$1.55 \times 10^4$ (955)	2.2 (0.3)	226 (58)	$1.58 \times 10^4$ (1282)	4159 (301)	1302 (62)	2286 (165)	5.3 (1.5)	120 (28)
10 cm	283 (76)	320 (25)	$1.50 \times 10^4$ (1056)	2.1 (0.5)	226 (51)	$1.85 \times 10^4$ (1523)	3860 (354)	1317 (148)	2655 (253)	6.7 (2.1)	135 (20)
20 cm	166 (31)	215 (22)	$1.75 \times 10^4$ (403)	2.3 (0.5)	275 (63)	$1.58 \times 10^4$ (751)	4748 (127)	1847 (156)	2904 (156)	4.5 (0.8)	220 (16)
<b>P values</b>											
Soil	<0.01	<0.01	<0.01	<0.01	<0.01	0.410	0.085	<0.01	0.038	<0.01	<0.01
Cap Depth	0.611	<0.01	<0.01	1.00	1.00	0.679	<0.01	<0.01	0.101	1.00	<0.01
Soil x Cap	1.00	0.250	0.602	1.00	1.00	0.025	0.485	0.071	0.311	1.00	0.394

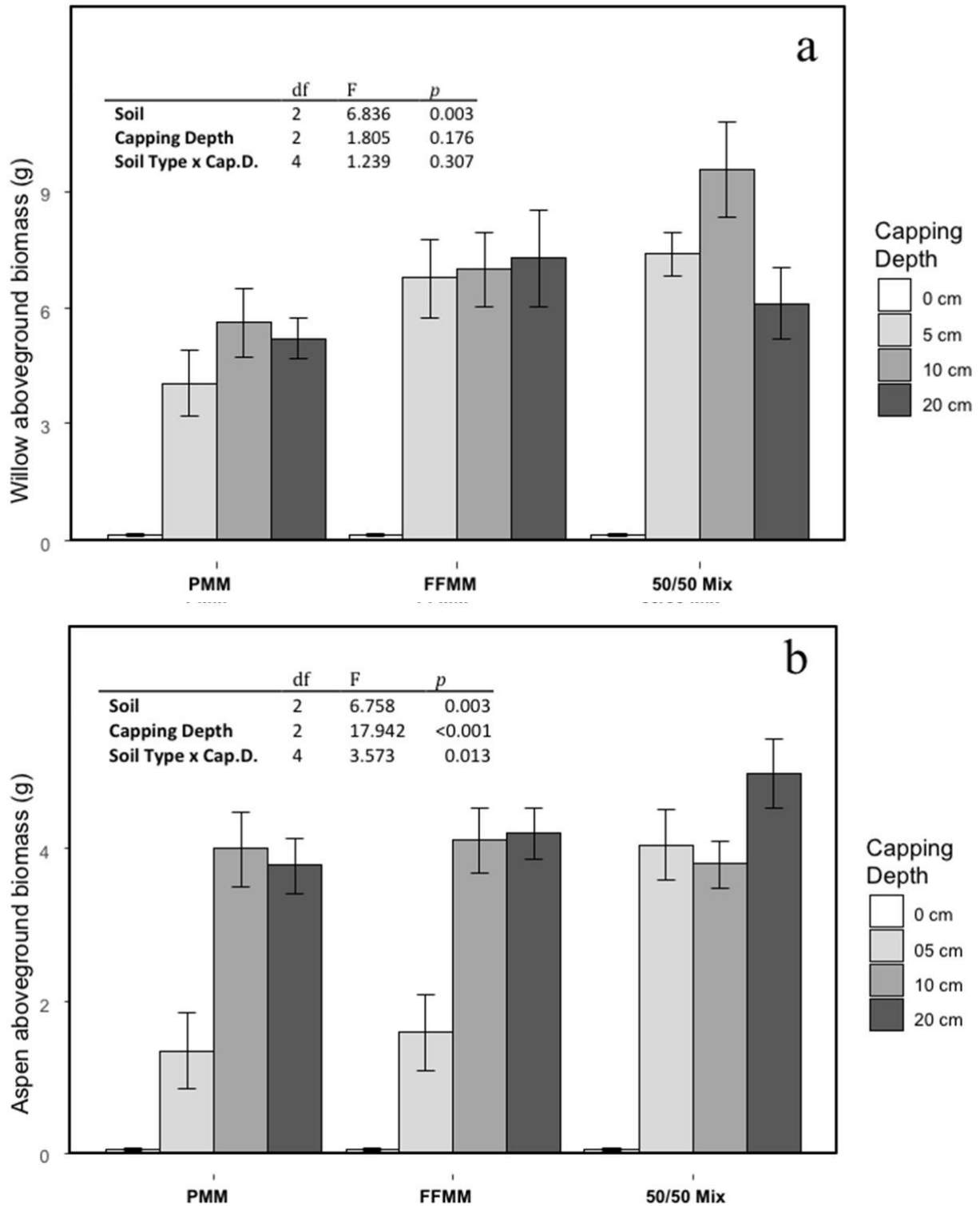
N=6-10

Values represent means with standard errors in brackets. Values that are in bold represent significantly different values than all other treatments ( $p < 0.01$ ).

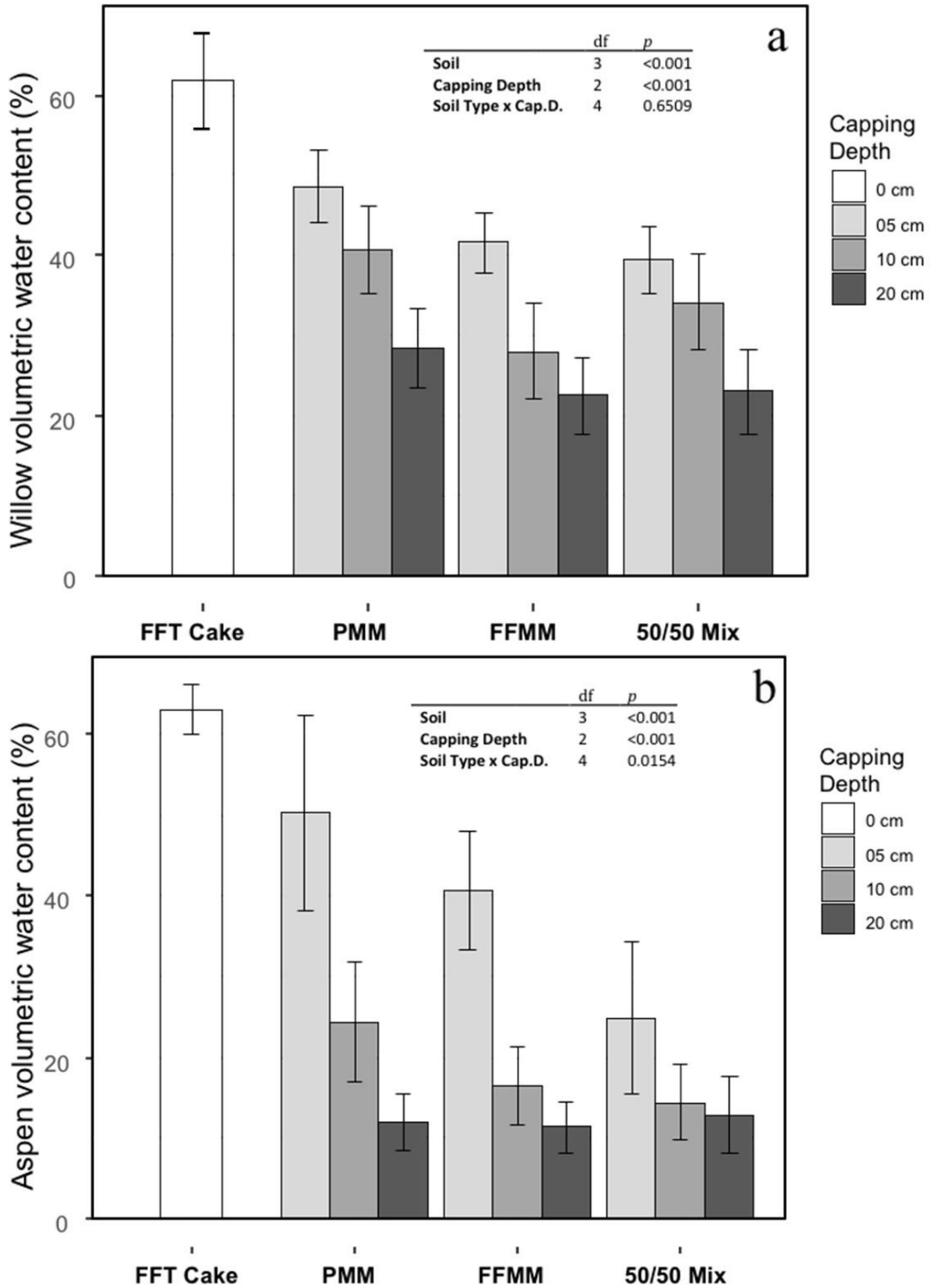




**Figure 2.1.** Diagram of experimental design with different soil materials and capping depths



**Figure 2.2.** Aboveground biomass (g) for *S. bebbiana* (a) and *P. tremuloides* (b). Values represent the means with standard errors (n=6). ANOVA data presented in these figures exclude the zero capping depth controls in its analysis.



**Figure 2.3.** Average soil volumetric water content for *S. bebbiana* (a) and *P. tremuloides* (b). Values represent the means with confidence levels (0.95, n=18-24).

## Chapter 3 – Adding Biochar to Soil Caps Over FFT Cake

### 3.1. Introduction

Fire is a prominent natural disturbance present in most boreal forests (Hicke et al. 2003). Fires produce black carbon (BC), a type of pyrogenic carbon that is chemically resistant to oxidation (Preston and Schmidt 2006). BC has positive effects on soil properties, such as decreasing bulk density and increasing water retention (Busscher et al. 2003), increasing cation exchange capacity (Qiu et al. 2008) and increasing adsorption of cations, which can reduce nutrient leaching (Laird et al. 2010) and immobilize contaminants (Qi et al. 2017). Biochar is a charcoal product created from the pyrolysis of biomass for use as a soil amendment (Lehmann and Rondon 2006), designed to emulate BC and natural disturbance processes (Thomas 2013).

There have been numerous agricultural studies that show the benefits of biochar (Liu et al. 2013; Laghari et al. 2016). Biochar improves agricultural productivity, increasing plant growth and yield (Lehmann and Rondon 2006; Steiner et al. 2008; Major et al. 2010), improving water retention and plant available water content (Atkinson et al. 2010; Downie 2011; Tammeorg et al. 2014), reducing soil acidity (Lehmann et al. 2003; Major et al. 2010) and improving overall biophysical and chemical properties in the soil (Sohi et al. 2010; Jeffery et al. 2015).

There are fewer studies that investigate the benefits of biochar in natural ecosystems or in restoration ecology; however, this area of study is expanding. Thomas and Gale (2015) conducted a meta-analysis of the studies where biochar was applied as an amendment for forest restoration and found a mean increase of 41% in total woody plant biomass. Studies using biochar as an amendment in oil sands reclamation are even less common, however some studies

suggest biochar can improve tree growth in peat-mineral mixes (Dietrich et al. 2017), and can also have potential benefits in forest floor mineral mixes admixed with peat-mineral mix (Dietrich and MacKenzie 2018). Biochar hasn't been studied as a soil amendment in oil sands tailings reclamation but could have promising benefits due to its positive soil benefits found in previous studies. Biochar is also known to have the capacity to absorb and immobilize metal pollutants (Wang et al. 2018) and has been found to reduce the phytotoxicity of soils near bitumen processing plants (Koltowski and Olezczuk 2016), and therefore could have benefits when used as an amendment in oil sands tailings reclamation.

The main study (see Chapter 2) investigated how different capping soils (FFMM, PMM and Mixture) and capping depths (0, 5, 10, 20 cm) above FFT cake impact woody plant growth. The second part of the study aims at understanding how biochar as a soil amendment effects woody plant growth in a 50/50 mix soil cap (10 cm depth) over FFT cake.

### 3.2. Materials and Methods

Many of the methods are a repeat of the main experiment (see Chapter 2), however, the experimental design is different for this study. Beaked willow (*Salix bebbiana* Sarg.) and trembling aspen (*Populus tremuloides* Michx.) were grown again for 16 weeks in pots layered with centrifuged fine fluid tailings cake (FFT Cake) as a subsoil material on the bottom and covered with 10 cm of capping soil. The capping soils were a 1:1 mixture of PMM and FFMM, one treatment without biochar and the second treatment mixed with biochar at a rate of 10 MT ha<sup>-1</sup> (10.2 g per pot). The study was a completely randomized design with 2 plant species (aspen and willow), 2 capping soils (with and without biochar) and 6 replicates for a total of 24 pots.

Soil descriptions, seed sources, greenhouse information and watering schedules are described in Chapter 2.

*S. bebbiana* and *P. tremuloides* seedlings were grown for 16 weeks after transplanting into the layered treatment pots. Aboveground biomass, plant height, foliar nutrients, soil water content and nutrient supply rates were determined in the laboratory. For detailed methods, see Chapter 2. For soil properties, see Table 3.1.

All data analysis was done using R software (version R.3.1.1, R Core Team 2019). To determine the statistical differences between the two treatments, T-tests were run using the *t.test* command for aboveground biomass, plant height, volumetric water content, nutrient supply rates and foliar nutrient concentrations.

### 3.3. Results

The average aboveground biomass of *S. bebbiana* was not different between treatments ( $p=0.311$ ) at 8.12 g for the biochar treatment and 9.57 g for the treatment without biochar (Figure 3.1a). The average aboveground biomass of *P. tremuloides* was also not different between treatments ( $p=0.539$ ) at 3.41 g for the biochar treatment and 3.80 g for the treatment without biochar (Figure 3.1b). The average final plant height of *S. bebbiana* was not different between treatments ( $p=0.603$ ) at 67.6 cm for the biochar treatment and 72.2 cm for the treatment without biochar (Figure 3.2a). The final plant height of *P. tremuloides* was also not different between treatments ( $p=0.241$ ) at 38.1 cm for the biochar treatment and 45.5 cm for the treatment without biochar (Figure 3.2b).

The average VWC in the biochar treatment for *S. bebbiana* was higher (43.1%;  $p=0.028$ ) than for the treatment without biochar (34.2%; Figure 3.3a). The average VWC in the biochar treatment for *P. tremuloides* was also higher (28.4%;  $p=0.004$ ) than for the treatment without biochar (14.5%; Figure 3.3b). Nutrient supply rates (Table 3.2) of Al in the soil for the biochar treatment was  $19.7 \mu\text{g } 10 \text{ cm}^{-2} \text{ 28 Days}^{-1}$ , which was lower ( $p=0.037$ ) than in the treatment without biochar ( $31.2 \mu\text{g } 10 \text{ cm}^{-2} \text{ 28 Days}^{-1}$ ). There were no significant differences between the two treatments for all other nutrients ( $p>0.05$ ). Foliar nutrient concentrations (Table 3.3) of Ti for the biochar treatment was  $2.5 \text{ mg Kg}^{-1}$ , which was lower ( $p=0.043$ ) than in the treatment without biochar ( $6.7 \text{ mg Kg}^{-1}$ ). There were no significant differences between the two treatments for all other nutrients ( $p>0.05$ ).

### 3.4. Discussion

The biochar had no significant effect on plant growth for either species. The treatment with biochar did not produce a significantly different aboveground plant biomass or plant height than the treatment without biochar. Although most studies suggest biochar can improve tree growth (Thomas and Gale 2015), Dietrich and MacKenzie (2018) found that some reclamation soils such as FFMM do not have improved plant growth when biochar is added to the soil, however, when biochar is added to PMM plant growth is improved. Dietrich et al. (2017) found that biochar added to a mixture of 1:1 PMM to FFMM increased aspen biomass but not plant height. In our study, the 50/50 mix did not seem to benefit from the addition of biochar. Tian et al. (2012) found plant biomass increases when biochar was added to PMM in greenhouse trials, so perhaps biochar would have had a greater effect if the capping soils were only PMM. Biochar may be a more successful amendment when added to PMM alone rather than FFMM or a 50/50

mix due FFMM already being naturally high in pyrogenic carbon (Ohlson et al. 2009). Another factor could be biochar's ability to increase nutrient availability in a more nutrient PMM, such as K availability (Dietrich and MacKenzie 2018).

For both plant species, the average volumetric water content was higher in the biochar treatment. However, both treatments had had water content below the field capacity, so likely did not greatly impact growth. Biochar is known to increase water retention capability (Atkinson et al. 2010) which is a possible reason higher levels of volumetric water contents were found in the biochar treatments; however, this could also be a result of the smaller plants in the non-biochar treatment taking up less water from the soil.

Soil nutrient supply rates, as well as foliar nutrient concentrations, were similar between both treatments among nearly every nutrient. The exceptions are that Al supply rates in biochar amended soil were 37% smaller than in the treatment without biochar, and Ti foliar concentrations in the biochar treatment were less than half the concentration than the other treatment. Several studies demonstrate that biochar decreases metal bioavailability in the soil (Pan and Li 2013; Huang et al. 2013). This may explain the differences in Al supply rates and Ti concentrations, however would not have impacted the overall plant growth as the concentrations are still relatively low. If biochar was added directly to the tailings cake, we would have perhaps seen a larger effect.

### 3.5. Conclusion

In summary, the biochar had no effect on plant growth. Biochar does not seem to be a beneficial amendment when added to a 50/50 mix capping soil at the rate applied.



### 3.6. Tables and Figures

**Table 3.1.** Soil properties of subsoil and capping materials.

<b>Soil Description</b>	<b>pH</b>	<b>Electrical Conductivity (dS m<sup>-1</sup>)</b>	<b>Bulk Density (g cm<sup>3</sup>)</b>	<b>Field Capacity (% water content)</b>
FFT Cake	8.13 (0.19)	1.12 (0.05)	0.95 (0.06)	46.2
50/50 Mix	3.98 (0.01)	0.61 (0.01)	0.84 (0.12)	39.1
Biochar Mix	4.88 (0.33)	0.31 (0.01)	0.88 (0.09)	44.0

N=2-8

Values represent the means with standard deviation in brackets.

**Table 3.2.** Soil nutrient data from Plant-Root-Simulator (PRS) probes ( $\mu\text{g } 10 \text{ cm}^{-2} \text{ 28 Days}^{-1}$ ).

	<b>NO<sub>3</sub>-</b>	<b>NH<sub>4</sub></b>	<b>P</b>	<b>K</b>	<b>Ca</b>	<b>Mg</b>	<b>Al</b>	<b>B</b>	<b>Fe</b>	<b>S</b>	<b>Zn</b>
Treatment without biochar	118.4 (26.4)	< 2.0	2.05 (0.82)	29.55 (1.53)	1917 (120)	414.6 (32.1)	31.2 (1.3)	1.32 (0.30)	36.9 (13.2)	796 (130)	0.55 (0.13)
Treatment with Biochar	134.9 (71.1)	<	1.25 (0.09)	25.04 (1.20)	1962 (121)	433.3 (19.8)	19.7 (3.5)	0.88 (0.32)	14.4 (1.8)	979 (35)	0.67 (0.16)
P values	0.839		0.385	0.081	0.804	0.647	0.037	0.370	0.170	0.245	0.594

N=3

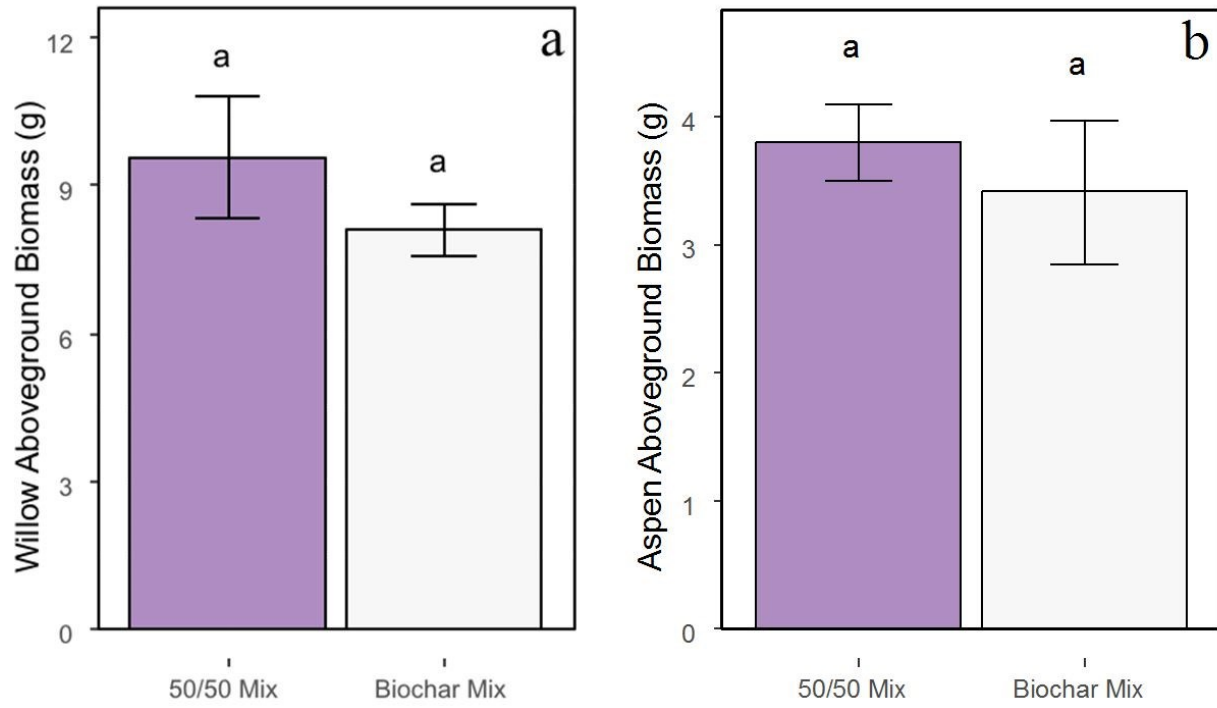
Values represent the means with standard errors in brackets. Values below the detection limit are represented by '<'.

**Table 3.3.** Foliar nutrient concentrations ( $\text{mg g}^{-1}$ ) of *Salix bebbiana*.

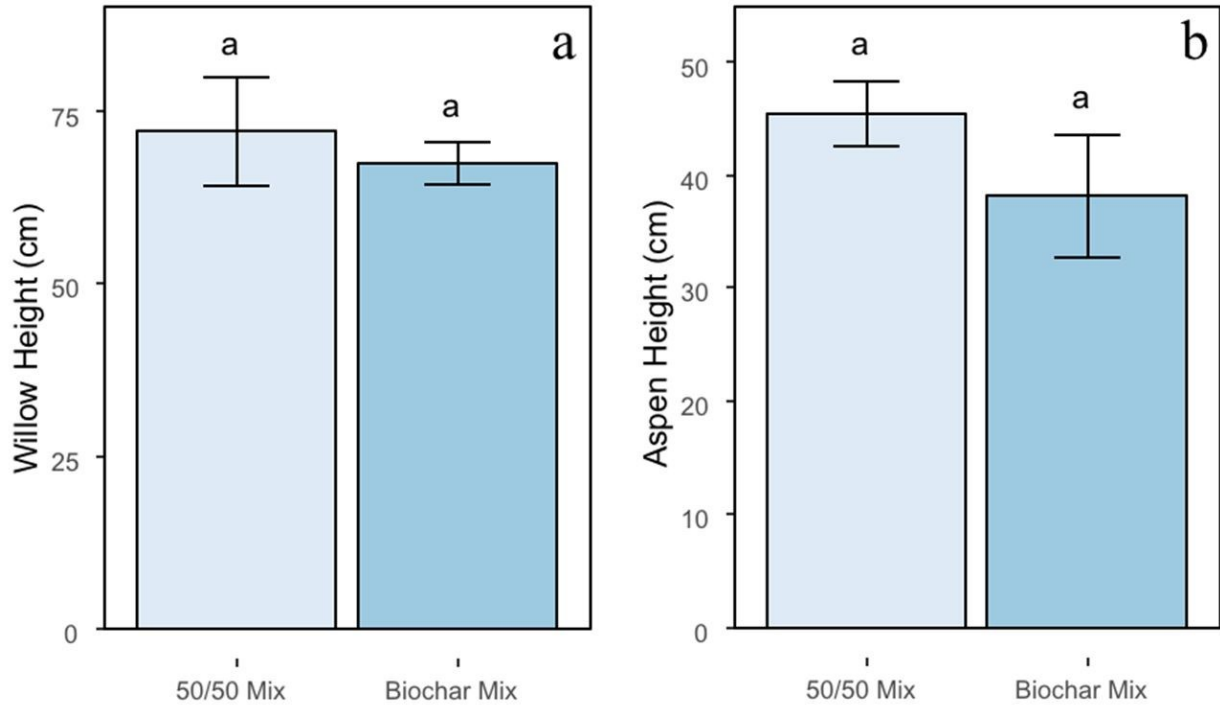
	<b>Al</b>	<b>B</b>	<b>Ca</b>	<b>Cr</b>	<b>Fe</b>	<b>K</b>	<b>Mg</b>	<b>P</b>	<b>S</b>	<b>Ti</b>	<b>Zn</b>
Treatment without Biochar	283 (76)	320 (25)	15024 (1056)	2.1 (0.5)	226 (51)	18513 (1523)	3860 (354)	1318 (148)	2655 (253)	6.7 (2.1)	135 (20)
Treatment with Biochar	163 (34)	380 (23)	13940 (1795)	2.0 (0.5)	191 (32)	19347 (1629)	4689 (513)	1631 (204)	3061 (228)	2.5 (0.4)	168 (22)
P values	0.143	0.101	0.637	0.927	0.549	0.721	0.232	0.258	0.255	0.043	0.298

N=7-9

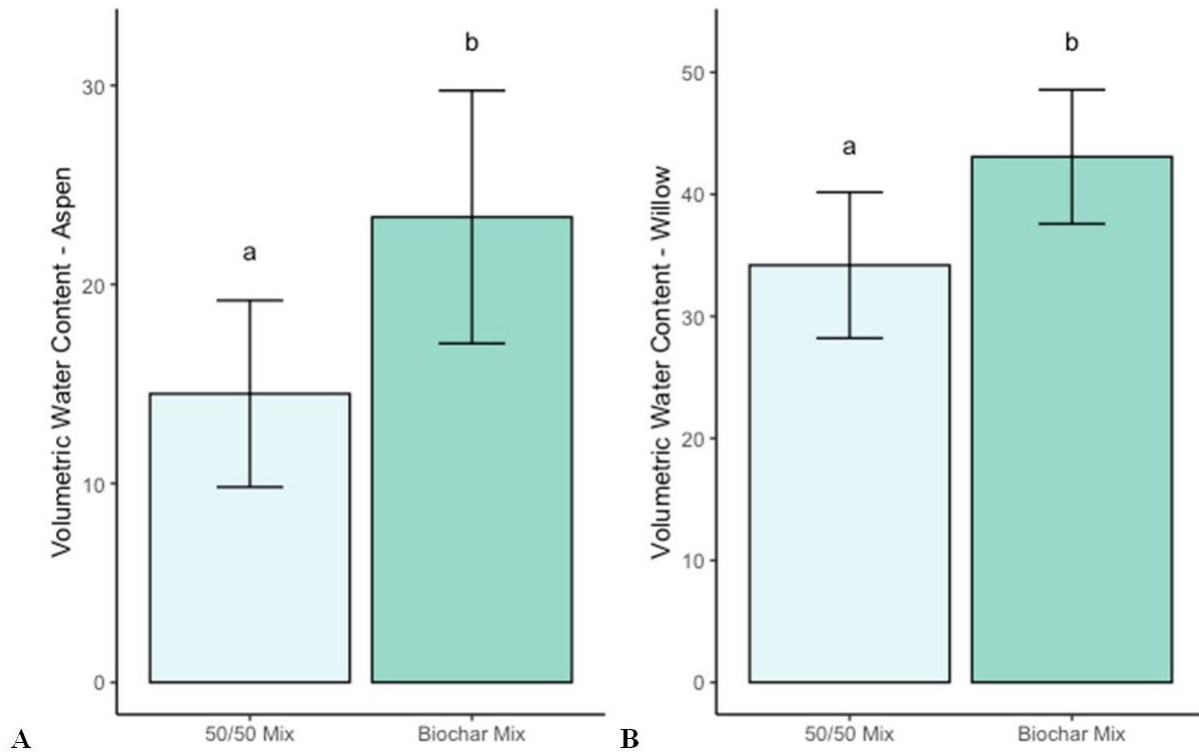
Values represent means with standard errors in brackets.



**Figure 3.1.** Aboveground biomass (g) for *S. bebbiana* (a) and *P. tremuloides* (b). Values represent the means with standard errors (n=6; p>0.05).



**Figure 3.2.** Plant height (cm) for *S. bebbiana* (a) and *P. tremuloides* (b). Values represent the means with standard errors (n=6; p>0.05).



**Figure 3.3.** Average soil volumetric water content for *S. bebbiana* (a) and *P. tremuloides* (b). Values represent the means with confidence levels (0.95, n=18-24).

## Chapter 4 – Conclusion

### 4.1. Study Implications and Recommendations

Understanding the findings from this study gives some insight for future management practices in oil-sands reclamation. The study found that even after centrifuge dewatering, high volumetric water content in FFT cake was still an issue affecting plant growth. Even after capping, water logging was an issue in shallower capping depths, especially for water sensitive species such as aspen. If FFT dewatered by centrifuge is to be used for reclamation purposes, high moisture content needs to be considered in management decisions.

Adding any soil cap resulted in a positive growth response, regardless of soil type or capping depth. However, deeper capping depths may want to be considered for long term growth. Due to limited reclamation materials, optimal minimal capping depth will need to be determined to maximize the use of such materials for long term reclamation success.

Both aspen and willow demonstrated they were potentially useful species to use in oil sands tailings reclamation. Considering willow had a greater tolerance to survive directly in FFT cake, was a good metal accumulator and is also more adapted to grow in high moisture conditions, *Salix bebbiana* is a promising species to be used in tailings reclamation. I could have potential use in other type of tailings and mining reclamation, and should be further studied.

## 4.2. Study Limitations

There were certain limitations to the study as well as aspects that could have been improved. First, the greenhouse itself is an inherent limitation to any study. Growing plants in such a controlled environment has benefits; however results can sometimes be less realistic and less transferrable to real-world conditions. Another key limitation is that the plants were grown in pots, and over a relatively short-period of time. The study could have been improved by incorporating more measurements such as belowground biomass, plant transpiration, chlorophyll content, etc.

## 4.3. Future Research

The use of dewatered mature fine tailings in the oil-sands reclamation process may become a common practice in the future, however, more research studies should be done to better understand the potential risks. Before incorporating dewatered tailings into regular reclamation practices, it would be encouraged to conduct a mesocosm experiment that investigates the long-term effects of growing different plants over FFT cake and the impact on the surrounding ecosystem, similar to what is described by Naeth et al. (2015). A greater variety of plant species that reflect targeted ecosystems should be used in such studies.

Future studies should investigate the belowground story to get a more detailed understanding of how plant growth is impacted by FFT cake. Root biomass, root metal concentrations and microbial community structures should be studied.



Biochar could still have benefits in tailings reclamation, especially due to its adsorption properties. Biochar emulates black carbon which is naturally found in the soils of ecosystems from the Athabasca region, and therefore could be beneficial when rebuilding novel ecosystems. More studies using biochar need to be conducted to understand the best way if at all to use biochar in tailing reclamation.

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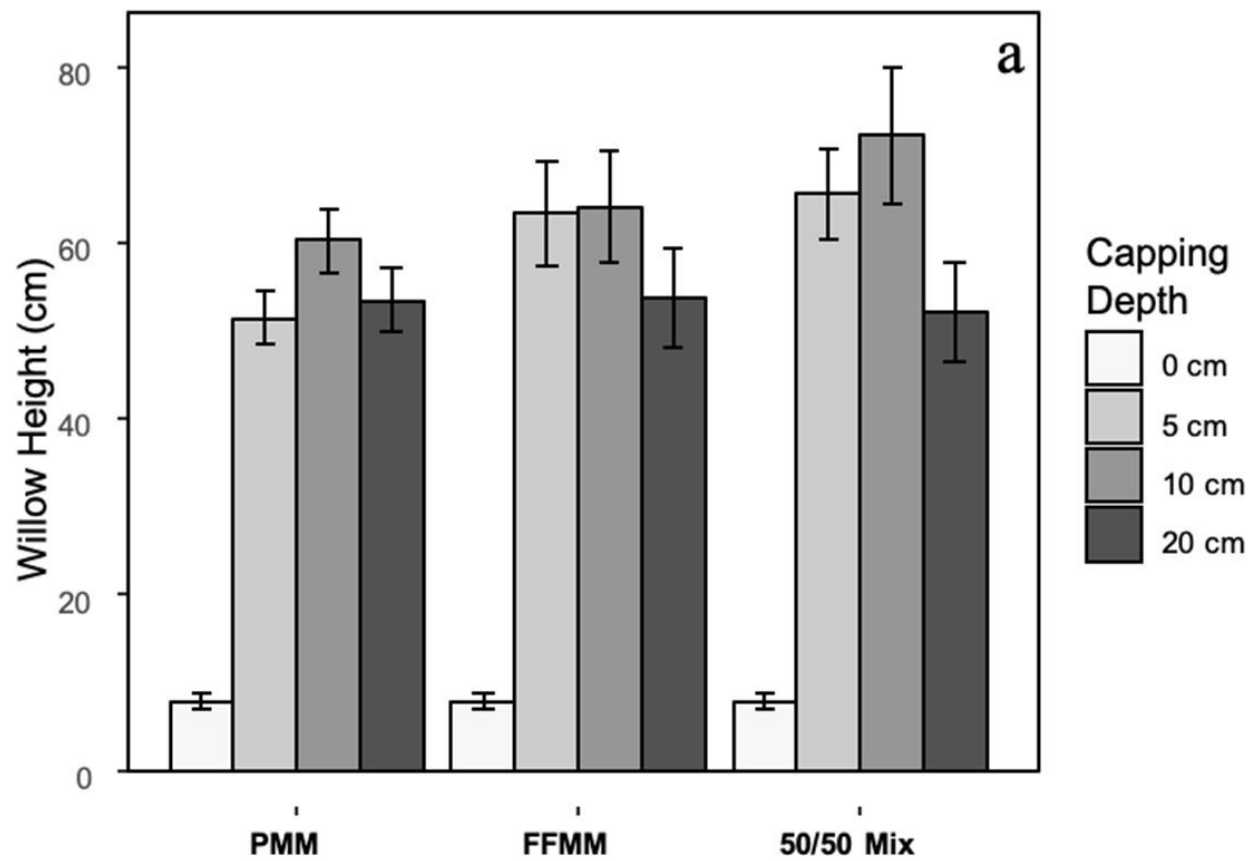
## Appendix

**Table A1.** Foliar nutrient concentrations (mg g<sup>-1</sup>) of *P. tremuloides*.

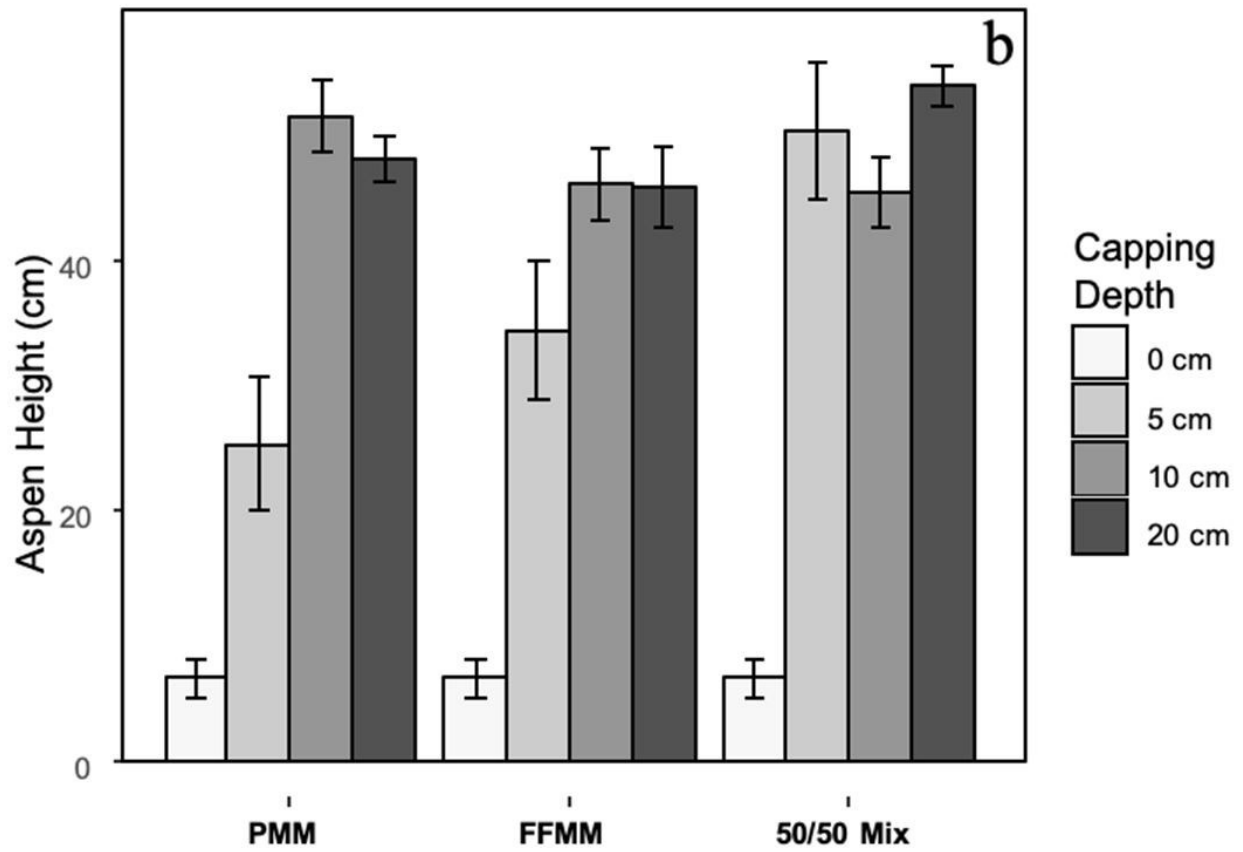
	Al	B	Ca	Cr	Fe	K	Mg	P	S	Ti	Zn
<b>Controls</b>											
FFT											
Cake	1.1 × 10 <sup>4</sup> (1723)	376 (38)	3.8 × 10 <sup>4</sup> (6950)	7.8 (1.6)	3898 (1057)	2866 (401)	5461 (679)	1165 (275)	5311 (669)	60.1 (5.2)	145 (26)
<b>PMM</b>											
5 cm	438 (197)	121 (13)	2.4 × 10 <sup>4</sup> (3949)	11 (5.6)	625 (245)	1.1 × 10 <sup>4</sup> (2251)	4525 (397)	1713 (213)	6511 (2140)	6.7 (2.1)	227 (29)
10 cm	162 (57)	120 (20)	2.0 × 10 <sup>4</sup> (977)	4.0 (1.1)	297 (117)	1.4 × 10 <sup>4</sup> (931)	4112 (193)	1166 (61)	3691 (355)	3.9 (1.0)	226 (21)
20 cm	102 (19)	52 (4.3)	1.7 × 10 <sup>4</sup> (809)	1.1 (0.3)	126 (18)	1.1 × 10 <sup>4</sup> (549)	3979 (204)	1379 (213)	3100 (341)	1.8 (0.3)	175 (27)
<b>FFMM</b>											
5 cm	252 (120)	85 (26)	1.7 × 10 <sup>4</sup> (3561)	8.4 (2.2)	214 (81)	1.1 × 10 <sup>4</sup> (2164)	4282 (839)	1189 (248)	3995 (914)	5.4 (1.9)	109 (24)
10 cm	194 (48)	139 (19)	2.2 × 10 <sup>4</sup> (1031)	3.8 (1.5)	164 (35)	1.7 × 10 <sup>4</sup> (1657)	5523 (348)	1367 (158)	3472 (321)	3.9 (1.0)	221 (25)
20 cm	227 (72)	53 (5.0)	1.9 × 10 <sup>4</sup> (2064)	2.9 (1.1)	192 (44)	1.4 × 10 <sup>4</sup> (1580)	5539 (406)	1816 (97)	3433 (226)	3.6 (0.8)	242 (18)
<b>50/50</b>											
<b>Mix</b>											
5 cm	95 (22)	151 (16)	1.8 × 10 <sup>4</sup> (597)	2.1 (0.5)	103 (14)	1.5 × 10 <sup>4</sup> (1224)	3860 (247)	1024 (113)	3054 (221)	2.2 (0.5)	158 (21)
10 cm	225 (86)	85 (9.6)	1.8 × 10 <sup>4</sup> (1213)	3.8 (1.3)	236 (32)	1.4 × 10 <sup>4</sup> (1132)	4626 (225)	1426 (138)	4323 (534)	4.5 (1.3)	223 (26)
20 cm	120 (25)	49 (3.8)	1.5 × 10 <sup>4</sup> (1388)	1.3 (0.4)	158 (32)	1.3 × 10 <sup>4</sup> (1265)	3913 (203)	1544 (205)	2279 (75)	2.1 (0.3)	184 (21)
<b>Study 2</b>											
<b>Biochar</b>											
<b>Mix</b>	227 (128)	143 (8.4)	1.8 × 10 <sup>4</sup> (1403)	5.4 (2.3)	407 (260)	1.4 × 10 <sup>4</sup> (989)	4720 (516)	1093 (81)	3318 (626)	4.2 (2.0)	222 (53)

N=5-7

Values represent means with standard errors in brackets.



**Figure A1.** Final plant height (cm) for *S. bebbiana*. Values represent the means with standard errors (n=6).



**Figure A2.** Final plant height (cm) for *P. tremuloides*. Values represent the means with standard errors (n=6).

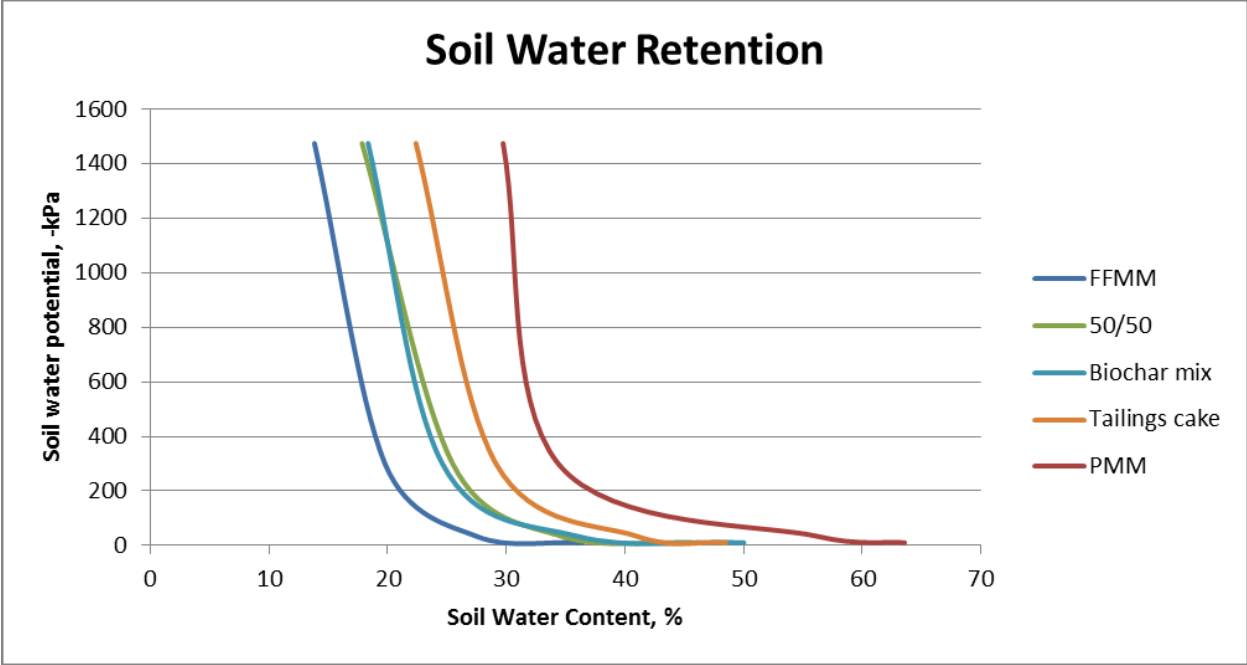


Figure A3. Soil water retention curve of different capping materials and tailings cake.



**Figure A4.** *S. bebbiana* growth after 16 weeks grown directly in FFT cake.



**Figure A5.** *S. bebbiana* growth after 16 weeks in 50/50 mix at 5 cm depth.





**Figure A6.** *P. tremuloides* growth after 16 weeks in 50/50 mix at 20 cm depth.



**Figure A7.** Dead *P. tremuloides* grown directly in FFT cake.