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Soluble calcium and sulphate excesses related to stress in Pinus contorta on peat amendments of reclaimed landscapes in the boreal oil sands region

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Abstract

The objective of this study was to evaluate impacts of three peat soil amendments containing variable amounts of soluble calcium and sulphate-S on soil quality and Pinus contorta (lodgepole pine) growth in a terrestrial boreal ecosystem. A 13-year old stand of lodgepole pine displayed various symptoms and degrees of nutrient deficiency on a reclaimed tailings sand dyke at an oil sands mining operation in northeastern Alberta, Canada and were representative of three unique peat-mineral soil amendments applied to a tailings pond slope in 1992. The poorest growth occurred on soil from a deep mesic peat deposit underlain by marl, the intermediate growth on a peat-mineral mix of shallow, fibric origin, and the best growth on soil of moist upland forest floor origin, with a cover of moss and litterfall. Transects were established in each of the three zones of variable pine performance to survey the vegetation and soil characteristics. Soil samples were analysed for salinity and soluble cations, reaction, and available nutrients in early fall, 2005. Soil moisture profiles to 160 cm were collected at each survey location during the 2006 growing season. Results indicated a negative relationship among pine performance with increasing soluble calcium and available sulphate-S. These relationships were stronger than indicators typically linked to poor peat amendment quality. Calcium and sulphate were the main contributors to electrical conductivity, and were also associated with elevated $CaCO_{3\ eq}$.

1 Introduction

Sphagnum spp. and *Carex* spp. peat from bogs and fens are often salvaged along with the underlying mineral soil substrates of glacial and post-glacial origin for use as a reclamation material in the Athabasca oil sands region. These amendments constitute the dominant reclamation material applied to upland sites in constructed reclaimed landscapes in the area. In their native state, *Sphagnum* spp. and *Carex* spp. dominated peatlands support a variety of ecosystems over a wide range of soil chemistry. Inorganic parameters including pH and salinity depend largely on surface hydrology at an ecosystem scale (Quinty and Rochefort, 2003). Relatively mineral rich fens are most often found in areas where surface water flow is slow yet continuous, and nutrients are continually in motion (Hájek et al., 2006). Surface water in fens also contain more soluble cations such as calcium and magnesium that associate with bicarbonate in the peat matrix soil solution and contribute to an elevated buffering capacity, keeping the pH near neutrality. Due to the relatively advanced decomposition stage in fens, nutrients are generally cycled faster in fens compared to bogs, and often contain more nutrients available for plants. Hydrological flow in bogs is generally stagnant, often accumulating humic acids and display retarded decomposition rate (Mitsch and Gosselink, 2000).

When peat soil is removed from its native hydric moisture conditions and introduced to dry reclamation sites, peat decay shifts from slowly formed anaerobic decomposition products of reduced and incompletely oxidised organic compounds toward an accelerated aerobic decomposition rate (St. Louis et al., 2003; Magnusson, 1993). During the growing season this overall shift to an oxygenated environment is accompanied by sharp increases in the overall mineralisation rates of sulphates, calcium and magnesium, especially during periods of repeated drying and re-wetting on the reclaimed slope (Verburg et al., 1999; Eimers et al., 2003). This process is exaggerated in peats that have reached relatively advanced stages of decomposition, such as mesic peats compared to less decomposed fibric peats (Whitfield et al., 2010).

The objective of this study was to identify the soil chemical properties inherent to peat-mineral mix reclamation soils of three unique origins that contribute to reduced reclamation success of subxeric sites targeted to a stand of lodgepole pine. Discussion of soil moisture is included to provide context to the soil chemistry results and to elucidate the influence of soil moisture on pine performance on the slope.

2 Methodology

2.1 Experimental area and amendments

The Dyke 2W experimental area (lat. 56°58'54"N, long. 111°31'03") is located on an upper, south facing tailings sand slope of about 20% to 30% grade. It is visible from Highway 63 on the south side of Tailings Pond 2/3 at the Suncor Oil Sands Project. Soil replacement on the dyke slope was initially conducted during the summer of 1992 under the direction and supervision of Suncor personnel. The slope was planted with equal stem densities (2,400 stems ha^{-1}) of lodgepole pine along the length of the study area in 1992 following soil replacement. Prior to seedling planting, the slope was broadcast seeded with an agronomic barley species to minimise erosion and competition among pioneer species with the pine seedlings.

The soil amendments along the slope were categorised as Deep Mesic Peat, Forest Floor Moss/Litter, Fibric, Humic (LFH) and Shallow Fibric Peat according to their determined soil amendment origins and visible relative variations of reclamation success; namely, stem density and height. As only one slope was assessed and similar sites in the project area were not identified during the initial site investigation that had similar amendment, substrate, and lodgepole pine regeneration, replication was an issue. This limitation prevents us from drawing broad conclusions regarding the inherent soil quality of the peat amendments used in this study beyond the Dyke 2W experimental area. The scope of the initial site investigation was to troubleshoot a specific reclamation performance issue on the slope. We maintain that heterogeneity in peat soil amendment chemistry is to be expected. With this in mind, Dyke 2W provides a unique observational study site where a group of related chemical factors appear to be expressed by unusually pronounced variation in pine performance.

2.1.1 Deep mesic peat - DM

This amendment originated from a mesic fen dominated deep peat deposit and was reported by soil salvage personnel on site to be underlain by a 30 cm thick layer of marl at the interface of the peat and the calcareous lacustro-till substrate. Following a brief period of storage at the Waste Area 21 stockpile on Suncor property, it was initially applied to a mean thickness of 20 cm on the western portion of Dyke 2W. For the sake of brevity, this amendment will be referred to hereafter as DM (Deep-Mesic).

2.1.2 Forest floor moss/LFH - FM

This amendment was reported to originate from a sandy upland site with a moist, moss covered forest floor that previously supported a black spruce and jack pine stand. This soil was placed immediately following salvage to a mean depth of 26 cm. This amendment will be referred to hereafter as FM (Forest-Moss).

2.1.3 Shallow fibric peat - SF

This amendment was observed to be a peat-mineral mix with a fibric peat organic component with a sandy loam to sandy clay loam mineral component high in coarse fragments. It was reported to originate from a shallow peat deposit on a transitional bog or poor fen vegetation community underlain by the weakly calcareous till common in the salvage areas at the time of removal. It was placed immediately following salvage to a mean depth of 52 cm on the eastern portion of Dyke 2W. This amendment will be referred to hereafter as SF (Shallow-Fibric).

2.2 Field methods

The on-site investigation was conducted over three days in mid October, 2005. At each of the three soil amendment types, nine equally spaced sampling locations were selected on a pre-determined grid pattern within a 0.2 ha plot, with a minimum distance of 7.5 m between sampling locations (along three transects of three locations each) for a total of 27 locations. Site characteristics were recorded at each location including, but not limited to, approximated pine stem height and diameter breast height (DBH) (where a pine stem was adjacent to the soil sample location) and thickness of amendment cover over tailings sand substrate. Soil samples were collected at 10 to 20 cm below the amendment surface at each location after excavating a shallow soil pit to the interface of the peat amendment and the tailings sand and collecting cursory profile descriptive data such as hand texture, root abundance, and coarse fragment volume estimates.

Average monthly air temperature and precipitation data between May 2004 and October 2006 were compared to the Canadian Climate Normals (1971–2000) for the weather station at the Fort McMurray airport to determine whether the climate data for the study period was representative of typical growing seasons in the area (Environment Canada, 2007).

2.3 Soil moisture

Sentek™ diviner access tubes were installed at each soil sampling location investigated during the soil sampling program. Electronic moisture data was obtained at 10 cm intervals from 10 to 160 cm below ground surface on 10 dates at two-week intervals during the late spring, summer, and early fall (June 1 to October 3) of 2006. Total profile soil water content (TSW, mm) was then calculated to 50 cm depth to compare TSW between soil amendments. The TSW 50 was calculated as the sum of the volumetric water content readings through the upper five 10 cm intervals of electronic moisture data readings (Singh et al., 1998). Other data included scaled frequency, volumetric water content (VMC, %), change in moisture storage, cumulative change in moisture storage, and soil temperature at 10 cm depth.

2.4 Laboratory analyses

All samples were immediately frozen in a chest freezer until submission to a commercial laboratory for analysis. All samples were analysed for the following parameters:

- Available nitrate (NO_3-N) (mg kg^{-1}) - 2.0 M KCl extract, determined by segmented flow analysis via cadmium reduction (Maynard et al., 2008).
- Available phosphate (PO_4-P) (mg kg^{-1}) - 0.25 M HOAc, 0.25 M NHOAc, 0.015 M NH_4F at pH 4.9 modified Kelowna extract solution, determined by colorimetry using ammonium molydate and ascorbic acid (Qian et al., 1994).
- Available potassium (K) (mg kg^{-1}) - 0.25 M HOAc, 0.25 M NHOAc, 0.015 M NH_4F at pH 4.9 modified Kelowna extract solution, determined by flame photometry (Qian et al., 1994).
- Available sulphate (SO_4-S) (mg kg^{-1}) - weak $CaCl_2$ extraction, determined by ICP-AES (Alberta Agriculture, 1987).
- Soluble Na, Ca, Mg, K (mg kg^{-1}) and EC (dS m^{-1}) (and calculated SAR) in deionised water saturation extract (Miller and Curtin, 2008).
- pH in saturated paste (Hendershot et al., 2008).
- $CaCO_{3\ eq}$ (%) – Five selected samples for each amendment were subsequently analysed by titration (Goh and Mermut, 2008).

2.5 Data analysis and interpretation

Analytical data sets were tested for normality using the D'Agostino-Pearson K2 test with alpha = 0.05. Non-normal concentration data were transformed by the log transformation prior to calculating means, standard errors, and making decisions of significance. The arcsine transformation was applied to non-parametric populations of ratio- and percentage-based data. Transformations of pH units into $[H^+]$ were performed to confirm validity of statistical decisions on normal distributions; however, simple means are presented here.

The differences between transformed and non-transformed mean pH values are negligible for these data sets and statistical decisions are valid despite treating these data as simple normal distributions. Statistical decisions were based on one-way analysis of variance (ANOVA) and Fisher's Least Significant Differences (LSD) paired tests with $p = 0.05$. Tabular data are supplemented with standard error (in brackets) and pair-wise LSD test results; columns with the same letter are not significantly different, except for soil moisture data which has significant differences compared across rows.

3 Results and discussion

The mean seasonal temperature (May to September) at the Fort McMurray airport in 2005 was 12.6°C, about 0.7°C below normal. May (10.0°C) and August (14.1°C) were particularly warm relative to those months of the previous year (5.7°C for May; 7.9° and 12.7°C for August). It was a cooler July in 2005 (16.2°C) compared to 2004 (18.0°C). Seasonal cumulative rainfall was 301 mm, slightly drier than the normal of 308 mm for the period from May to September (Environment Canada, 2007).

Table 1 shows the average amendment cover thickness and some basic pine growth statistics for each amendment. The FM amendment had a significantly higher average stem height (4.8 m) than the other amendments. The reclamation soil depth was greatest in SF (52 cm) and significantly smallest in the DM and FM (20 and 26 cm, respectively). The lack of a pattern attributing growth to placement depth suggests that amendment soil placement depth did not influence growth in this study. For example, the DM and FM amendments had similar placement depths; however, the stem height and density was significantly higher in FM than DM.

Table 1 Lodgepole pine growth characteristics for surviving stems among amendments and average peat-mineral thicknesses^{Z}

Amendment	Stem Height (m)	DBH^{Y} (cm)	Pine Adjacent Soil Sample Sites^{YW} (%)	Amendment Soil Placement Depth (cm)
DM	1.9 (0.2)^{V}b	2.6	44	20 (5)b
SF	2.5 (0.4)b	3.9	44	52 (5)a
FM	4.8 (0.2)a	5.9	100	26 (1)b

^{Z} As determined in October, 2005.
^{Y} For DM and SF, n = 4. For FM, n = 9.
^{W} Proportion of grid soil sample location in each amendment plot (max n = 9).
^{V} Values are averages (n = 9) with standard errors denoted by parentheses.
a,b Different letters within a column represent significant differences (Fisher's LSD) among peat-mineral amendments (p = 0.05).

3.1 Excess calcium and sulphate

Soluble calcium and $CaCO_{3\ eq}$ were significantly highest in the DM (Tables 2 and 3) followed by SF and FM. All pairwise comparisons were significantly different for $CaCO_{3\ eq}$, though there was no significant difference in soluble calcium between SF and FM.

Table 2 Peat-mineral amendment soluble cations and calcium ratios^{Z}

Amendment	Soluble Cations (mg kg^{-1} dry soil)				Ca/Mg Ratio^{Y}	Ca/Total Cation Ratio^{Y}
	Ca	K	Mg	Na		
DM	430 (63)^{W}a	7.0 (1.3)a	47 (9)a	8.9 (1.2)a	6.3 (0.6)a	0.83 (0.01)a
SF	63 (8)b	2.0 (0.4)b	16 (2)b	6.1 (0.4)b	2.4 (0.1)b	0.65 (0.01)b
FM	15 (2)b	1.6 (0.4)b	6 (1)b	4.3 (0.4)b	1.5 (0.0)b	0.50 (0.01)c

^{Z} As determined in October, 2005 and corrected to dry soil.
^{Y} Ratio calculated based on soluble cation concentrations converted to Meq 100g$^{-1.}$
^{W} Values are averages (n = 9) with standard errors denoted by parentheses.
a-c Different letters within a column represent significant differences (Fisher's LSD) among peat-mineral amendments (p = 0.05).

Table 3 Peat-mineral amendment reaction and salinity[Z]

Amendment	pH	EC (dS m^{-1})	SAR	$CaCO_3$ (%)
DM	7.3 (0.0)[Y]a	2.3 (0.2)a	0.1 (0.0)c	9.1 (1.5)[W]a
SF	7.3 (0.1)a	0.8 (0.1)b	0.3 (0.0)b	3.8 (0.4)b
FM	6.7 (0.1)b	0.4 (0.0)c	0.5 (0.0)a	0.8 (0.1)c

[Z] As determined in October, 2005.

[Y] Values are averages (n = 9) with standard errors denoted by parentheses.

[W] Values are averages (n = 5) with standard errors denoted by parentheses.

a-c Different letters within a column represent significant differences (Fisher's LSD) among peat-mineral amendments (p = 0.05).

The relative uptake of ions by plant roots in soils is governed by the soil solution concentration and gradients across the cell wall. The high calcium levels suggest there may be an accumulation of a substance around the plant root if in excess and more calcium is being supplied by mass transport than can be absorbed (Hausenbuiller, 1985). When expressed as a proportion of major cations, calcium represents 83% of the cations in the soil solution, followed by 65% on SF and 50% on FM. Calcium is typically the most abundant soluble cation in the soil solution (usually above 50% according to Henry (2003)), though excessive proportions, especially relative to magnesium (Ca/Mg ratio) can interfere with magnesium uptake as calcium and magnesium compete for carrier sites into roots (Ballard, 1986). Using Ballard's range of normal Ca/Mg ratios as a reference, all amendments appear to have Ca/Mg Ratios similar to the normal range of 3:1 to 5:1, with some statistically significant elevation in DM compared to the other amendments. Conversely, the FM Ca/Mg ratio was below the reported normal range, despite a lack of evidence of calcium deficiency in the pine.

Similar to soluble calcium, FM and SF had the lowest soluble sulphate (9 and 19 mg kg-1, respectively), with significantly higher sulphate in DM which averaged 591 mg kg-1 (Table 4). Further, the available NO_3-N/SO_4-S Ratio was between 21 and 25 times lower in DM than SF and FM. Additionally, sulphate accounted for 94 % of available nutrients (N, P, K, S) in DM, significantly higher than the 27 and 30 % of available nutrients in SF and FM, respectively. Similar to soluble calcium, this suggests mass transport and osmotic stresses are affecting stand growth at the sulphate absolute concentrations and proportions of DM.

Studies on *Sphagnum balticum* provided evidence that excess sulphates, in the absence of sufficient nitrogen to assimilate the excess into amino acids, damage the Photosystem II complex, thereby reducing the efficiency of photosynthesis (Granath et al., 2009). Pritchard et al. (2000) found calcium sulphate sequestered and deposited in the substomatal cavities of *Pinus palustris* needles in conditions of non-limiting calcium and sulphates in the soil. It was suggested that these deposited crystals prevent pathogen entry and decrease gas exchange by physically reducing the size of the stomatal aperture. Consequently, transpiration rates would also be reduced.

Testimonial site history evidence claims the origin to the calcium and sulphate concentrations is post-glacial lacustrine marl deposits, where a significant marl layer was encountered by soil salvage operators at the DM salvage site (Schurrenberger et al., 2003). A second origin of these ions is the release of calcium and sulphate from the peat material during decomposition where these ions will precipitate as gypsum during drying of the peat fraction (Litaor et al., 2005). It is likely that both sources are contributing factors of the ion levels encountered in DM, whereas sulphate release from the peat matrix alone is most likely from SF and FM. Additionally, sharp fluctuations in calcium content with depth in deep peat soils have been reported to correspond to relatively warm periods of the post-glacial Holocene, a legacy that is preserved in the reclamation soil amendment and contributes to highly variable calcium content of some soils (Arkhipov and Bernatonis, 2006). Each of these factors, in addition to those mentioned in the introduction, may be contributing to some degree to the trend of calcium and sulphates observed in the peat amendments.

Table 4 Peat-mineral amendment available nutrient status and sulphate ratiosZ

Amendment	Available Nutrients (mg kg^{-1} dry soil)				NO_3-N/SO_4-S Ratio	SO_4-S/ Total Avail. Nutrient Ratio
	NO_3-N	PO_4-P	K	SO_4-S		
DM	2.3 (0.4)[Y]ab	1.0 (0.0)b	24 (4)b	591 (125)a	0.01 (0.00)b	0.94 (0.01)a
SF	3.1 (0.4)a	2.9 (0.8)b	44 (8)a	19 (6)b	0.25 (0.05)a	0.27 (0.05)b
FM	1.7 (0.1)b	10.6 (2.6)a	16 (4)b	9 (1)b	0.21 (0.02)a	0.30 (0.05)b

[Z] As determined in October, 2005 and corrected to dry soil.
[Y] Values are averages (n = 9) with standard errors denoted by parentheses.
a,b Different letters within a column represent significant differences (Fisher's LSD) among peat-mineral amendments (p = 0.05).

3.2 Phosphorus and nitrogen

Phosphate-P was significantly lower in DM and SF compared to FM (Table 4). The absolute concentrations suggest a possible phosphorus deficiency in each of DM and SF, in the presence of alkaline pH and high soluble calcium, under which conditions precipitation of insoluble Ca-P salts are favoured (Zhu et al., 1994). Further, calcareous soils, as observed in DM and to a lesser extent SF, are especially low in available phosphorus; also by the precipitation of Ca-P (Diamond, 1985).

Nitrogen status was represented by the available NO_3-N fraction, and did not display a pattern consistent with a nitrogen deficiency in the soil particular to any one amendment. The lowest and least variable soil was FM, with about 2 mg kg^{-1}. DM had an equivalent NO_3-N average to FM, though it had greater variability (Table 4). It is acknowledged that the NO_3-N represents only a portion of the labile N pool and that sampling for N in the early fall is not the optimal time of year to measure this parameter without seasonal monitoring. Nevertheless, the NO_3-N proxy of nitrogen status for the studied amendments yielded results consistent with NO_3-N levels obtained on similar peat amendments applied to tailings sand in the same geographical setting during a mid-spring sampling event in 2004 (Hemstock et al., 2010). Based on the observed pattern, it appeared that nitrogen status may be a factor in the lodgepole growth on the site as it pertained to the available NO_3-N/SO_4-S Ratio described previously.

3.3 pH and salinity

The average pH of FM was significantly lower than SF and DM, which were slightly alkaline (Table 3). The lower pH in FM is attributed to elevated soluble calcium and $CaCO_{3\ eq}$ of DM and SF, and to greater pine biomass, resulting in increased needle litter on the soil surface layer of FM (Yavitt and Fahey, 1986). Needle litterfall is also assumed to have contributed to the pH of the native soil condition for the FM source, as it occurred under a pine forest (Jobbagy and Jackson, 2003).

It is not well understood to what extent pH may have affected pine growth in this study other than a possible link to phosphorus deficiency. As SF was an equivalent pH to that of DM, a direct relationship between rooting zone pH and pine growth, alone, could not be determined. It is otherwise well known that lodgepole pine species tolerate an acidic environment, a sentiment consistent with surface conditions Bulmer and Krzic (2003) found in natural forest soils of north eastern British Columba. Bulmer and Simpson (2005), however, used a medium textured mineral soil with alkaline pH containing plentiful PO_4-P which was not evidenced to interfere with the lodgepole pine growth on their study investigating moisture and compaction.

The combination of in situ calcium carbonate formation, the mechanical mixing of deep carbonates into DM and SF, and the inherent high buffering capacity of the peat fraction makes these amendments highly resistant to acidification (Carrow et al., 2002). The prevailing conditions on these amendments may render the soil incapable of supporting vegetation communities that require acidic and non-saline conditions to establish and survive into the later stages of succession (Purdy et al., 2005).

Electrical conductance for SF and FM were < 1 dS m^{-1} at all sites, and thus non-saline. DM averaged 2.3 dS m^{-1}, and significantly higher than SF and FM. Sodium adsorption ratio (SAR) was expectedly low, with a maximum of 0.5 in FM, as the high presence of calcium readily displaces the low sodium levels on the exchange sites (Sansom et al., 1998).

3.4 Soil moisture

The total soil water content to a depth of 50 cm (TSW 50) showed highest cumulative soil water in SF, with DM and FM having similar moisture conditions throughout the growing season (Figure 1). SF had higher TSW 50 than DM and FM, seemingly largely attributable to having the greatest cover soil thickness (52 cm), while DM and FM had similar cover soil thickness (CEMA, 2006). Average volumetric water content (VMC) indicated that the least variable and driest profiles of the wettest and driest day were FM and DM, due to the similarities in cover thickness of the amendments (data not shown). The general increase in moisture downwards through the profile suggests cooler temperatures and a lack of a deep root system for water uptake.

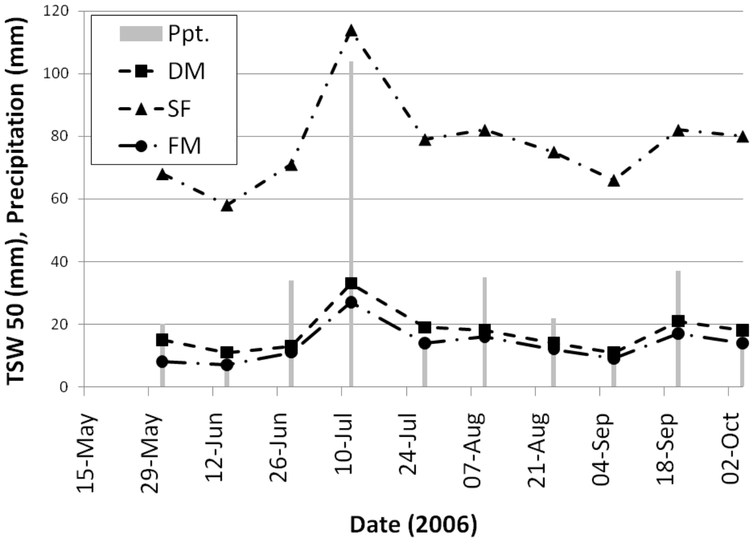

Figure 1 Total soil water to 50 cm (TSW 50) in the 2006 growing season for all treatments with diviners installed

4 Summary and conclusions

This study identified soil chemical evidence for possible nutrient deficiencies in lodgepole pine based on the inverse relationship between high proportions and absolute concentrations of soluble calcium and sulphate in some peat-mineral reclamation soils and poor pine growth performance. The presence of elevated carbonates in some amendments contribute to high resistance to acidification of the upper soil profile by leaching, and such alkaline reclamation soil may be detrimental to lodgepole pine growth with respect to phosphorus availability, and are correlated to the presence of high calcium and sulphate, suggesting a similar origin or in situ condition for release into the soil matrix. The saline conditions observed in DM were driven mainly by the high calcium and sulphate levels. The FM amendment supported a relatively normal pine stand and demonstrated that tailings sand can provide a suitable substrate for subxeric reclaimed sites with minimal application of an appropriate reclamation soil.

An expanded version of this study is presently underway to address our current shortcomings of replication in attempt to identify a broader pattern in reclamation soil quality from various peat amendments with respect to pine performance. Study of foliar analysis is planned in an attempt to positively quantify the extent of various nutrient deficiencies in the live pine crown foliage. Additional qualitative characterisation of the peat amendments will also be included, as well as additional data on soil sulphur pools and micronutrients. Considerable research on lodgepole pine foliar nutrition already exists which may provide a valuable benchmark for this study (Amponsah et al., 2005; Sanborn et al., 2005).

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