

**IMPACTS OF SURFACE APPLIED ALKALINE-TREATED BIOSOLIDS
ON SPRUCE PLANTATION SOILS AND VEGETATION
IN NOVA SCOTIA, CANADA**

by

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ABSTRACT

Decades of acid deposition in northeastern North America has resulted in base cation (Ca, Mg, K) loss and increased exchangeable Al concentrations in many forest soils across the region. In Nova Scotia, Ca has been identified as a potentially limiting nutrient affecting current or future forest productivity in many areas – including plantation sites where nutrient demands are increased through intensive management. In this study, the use of alkaline-treated biosolids (ATB) on spruce plantations was investigated. It was hypothesized that ATB could be a good source of Ca in Ca-limited sites, while providing an environmentally sound end-use for this waste-stream product.

Through field and greenhouse trials, it was found that ATB can increase mineral soil Ca and base cation / Al ratios in Ca-limited sites, while also increasing forest floor pH and reducing exchangeable Al³⁺ concentrations. In addition, ATB treatments did not result in any significant accumulation and/or leaching of NO₃⁻ or trace metals, nor to any impact on ground vegetation abundance or diversity in the juvenile plantations assessed. It did, however, improve Ca, K, P, and Mn concentrations in white spruce crop tree foliage, while also slightly increasing diameter increment over two growing seasons.

With appropriate consideration of soil and site characteristics, use of ATB could be an effective way to offset current or future Ca deficits associated with base cation depletion and intensive harvesting in Nova Scotia spruce plantations, but it may not result in any short-term increases in yield due to lack of significant impact on N availability and/or the presence of other limiting nutrients. In addition, ATB produced using cement kiln dust as the alkaline stabilizer is a poor source of Mg, and the possibility of creating major Ca:Mg imbalances and/or inducing Mg shortages needs to be addressed when applying ATB to plantation soils.

Although initial results support the possible use of ATB in spruce plantation nutrient management, further research is needed to assess the longevity of ATB treatment impacts on plantation soils and vegetation, as well as the associated cost-effectiveness of ATB use versus other amendments such as lime and wood ash.

LIST of ABBREVIATIONS and SYMBOLS

AAS	Atomic Absorption Spectrophotometry
Al, Al ³⁺	Aluminum, Aluminum ion
Al(OH) ₃	Aluminum Hydroxide
ANOVA	Analysis of Variance
ATB	Alkaline-Treated Biosolids
% BS	Percent Base Saturation
C, CO ₂	Carbon, Carbon Dioxide
C:N	Carbon : Nitrogen Ratio
Ca, Ca ²⁺	Calcium, Calcium ion
CaCl ₂	Calcium Chloride
CaCO ₃	Calcium Carbonate
Cd, Cd ²⁺	Cadmium, Cadmium ion
Cu, Cu ²⁺	Copper, Copper ion
CEC	Cation Exchange Capacity
CES	Cation Exchange Sites
CKD	Cement Kiln Dust
Cl, Cl ⁻	Chlorine, Chloride ion
°C	Degrees Celsius
% CF	Percent Coarse Fragments
cm	Centimetres
cmol ⁺ kg ⁻¹	Centimoles positive charge per kilogram
Db	Bulk Density
dbh	Diameter at Breast Height
EC	Electrical Conductivity
eq	Equivalents
eq ha ⁻¹	Equivalents per hectare
eq ha ⁻¹ yr ⁻¹	Equivalents per hectare per year
Fe, Fe ²⁺ / Fe ³⁺	Iron, Iron ions
g cm ⁻³	grams per cubic centimetre
g eq ⁻¹	grams per equivalent
GIS	Geographic Information System
H, H ⁺ , H ₂ O	Hydrogen, Hydrogen ion, Water
HCl	Hydrochloric acid
ICP	Inductively Coupled Plasma Mass Spectrometry
J mol ⁻¹ °C ⁻¹	Joules per mole per degree Celsius
K, K ⁺	Potassium, Potassium ion
KCl	Potassium Chloride
kg	Kilograms

kg ha ⁻¹ yr ⁻¹	Kilograms per hectare per year
km ²	Square kilometres
kPa	Kilopascals
m	Metres
M	Molar
m ³ ha ⁻¹	Cubic metres per hectare
m ³ ha ⁻¹ yr ⁻¹	Cubic metres per hectare per year
MARS	Microwave Accelerated Reaction System
Mg ha ⁻¹	Mega grams per hectare
mg kg ⁻¹	Milligrams per kilogram
MAI	Mean Annual Increment
MBC	Microbial Biomass Carbon
MCv	Percent Moisture Content by Volume
Mg, Mg ²⁺	Magnesium, Magnesium ion
MMAI	Merchantable Mean Annual Increment
Mn, Mn ²⁺	Manganese, Manganese ion
N, NO _x , NO ₃ ⁻ , NH ₄ ⁺	Nitrogen, Nitrogen Oxides, Nitrate ion, Ammonium ion
N	Normal (when associated with HCl concentration)
Na, Na ⁺	Sodium, Sodium ion
NaHCO ₃	Sodium Bicarbonate
NBM-NS	Nutrient Budget Model – Nova Scotia
NH ₄ OAc	Ammonium Acetate
% OM	Percent Organic Matter
P, PO ₄ ³⁻	Phosphorous, Phosphate ion
Pb, Pb ²⁺	Lead, Lead ion
pH	Potential of Hydrogen
PRS TM -probes	Plant Root Simulator probes
qdbh	Quadratic Mean Diameter at Breast Height
S, SO _x , SO ₂ , SO ₄ ²⁻	Sulphur, Sulphur Oxides, Sulphur Dioxide, Sulphate ion
SMB	Soil Microbial Biomass
SusMAI	Sustainable Mean Annual Increment
T-MCv	Threshold Moisture Content by Volume
t ha ⁻¹	Tonnes per hectare
µg 10cm ⁻²	Micrograms per 10 square centimetres
US, USA	United States, United States of America
Zn, Zn ²⁺	Zinc, Zinc ion

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Kevin Keys

CHAPTER 1

INTRODUCTION

BACKGROUND

Plantation forestry falls under the category of intensive forest management with its possible use of site preparation, improved growing stock, density management, competition control, and short rotation lengths to generate increased yields. In Nova Scotia, maximum productivity in fully stocked, natural softwood stands is estimated to be about $5 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ on sites of average quality – using red spruce (*Picea rubens*) as the reference tree – with actual values typically between 2 and $3 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ due to variation in stocking levels and species composition (NSDNR 2011). In contrast, productivity under plantation management is predicted to be about $7 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ with full stocking, or about $6 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ with typical stocking levels. In terms of yield, this equates to a doubling of predicted harvest volume from $150 \text{ m}^3 \text{ ha}^{-1}$ on typical unmanaged stands to about $300 \text{ m}^3 \text{ ha}^{-1}$ from plantations at peak mean annual increment (NSDNR 2011).

To realize potential volume gains from plantation management over the long-term, and to meet sustainable forest management objectives, there must be a sufficient supply of nutrients to offset increased exports due to harvesting. However, based on output from a new steady-state nutrient budget model described by Keys et al. (2016), many plantation sites in Nova Scotia may not be able to continuously meet desired yield targets over time without the use of soil amendments (see Chapter 2). Based on initial model runs, the nutrient predicted to be most limiting in Nova Scotia plantations is calcium (Ca). This is in keeping with other studies from eastern North America which show declines in forest soil Ca stocks from a combination of acid

deposition and harvesting (Freedman et al. 1986; Federer et al. 1989; Adams et al. 2000; Huntington 2005).

Calcium Depletion and Acid Deposition

Acid deposition is the end-product of various reactions between sulphur oxides (SO_x), nitrogen oxides (NO_x), and water in the atmosphere that reaches earth as precipitation and fog (wet deposition); or as gases, acid aerosols, and particles (dry deposition) (Morrison 2005). Major sources of SO_x and NO_x include fossil fuels (refining, power generation, transportation), mining and smelting, and production and use of fertilizers (Morrison 2005; Rice and Herman 2012). In northeastern North America, sulphur dioxide (SO₂) emissions have accounted for the majority of acidity in deposition (Lynch et al. 2000; Niemi 2005). As a whole, SO₂ emissions increased steadily in North America from the mid-1800s, peaking in the 1970s before starting to decline (Figure 1.1). This decline coincided with initial recognition of “acid rain” as an environmental problem in the early 1970s (CATCTAP 1983), recognition that eventually led to legislation designed to address acid deposition pollution – the 1970 *Clean Air Act* (US), 1990 *Clean Air Amendments* (US), and 1991 *Air Quality Accord* (US and Canada).

Data from the Canadian Council of Ministers of the Environment (CCME) (2013) show significant declines in wet deposition of sulphate (SO₄²⁻) and nitrate (NO₃⁻) between 1990 and 2010 reflecting the decrease in SO₂ and NO_x emissions over that period (Figures 1.2 and 1.3). Wet deposition of non-sea-salt SO₄²⁻ in Nova Scotia decreased from approximately 8-20 kg ha⁻¹ yr⁻¹ to 4-12 kg ha⁻¹ yr⁻¹, while NO₃⁻ decreased from approximately 6-15 kg ha⁻¹ yr⁻¹ to 3-9 kg ha⁻¹ yr⁻¹. However, despite these significant reductions, overall recovery of northeastern forests and surface waters from decreased acid deposition has not materialized as quickly as hoped (e.g.,

Houle et al. 2006; Johnson et al. 2008; Warby et al. 2009; Lawrence et al. 2012). Indeed, some projections suggest it could take several decades for many sites to naturally recover, due in part to (i) reduced acid neutralizing capacity in impacted soils and (ii) desorption of SO_4^{2-} that can continue to promote base cation depletion (Driscoll et al. 2001).

In addition, large areas of forest may still be receiving excess deposition despite emission reductions. For example, approximately 38% of eastern Canadian forests (range 3% to 61%) and 29% of New England forests (range 4%-52%) were still subject to acid deposition exceedances 10 years after signing of the *Air Quality Accord* – based on critical loads modelling using 2002-2003 data (NEG-ECP 2007). For eastern Canada, Nova Scotia had the greatest exceedance estimate (61% of mapped forest area) (Figure 1.4). Although acid deposition levels have continued to decline since 2002-2003 (Keys et al. 2016), there continues to be susceptibility to acid exceedance in Nova Scotia due, in part, to low weathering inputs from the coarse, acidic till soils that dominate many parts of the province (Whitfield et al. 2006).

The impacts of long-term (and continuing) acid deposition on northeastern forests have been well studied since the 1970s. Extensive and ongoing research has identified the following trends and impacts (from reviews by Lawrence et al. 1999; Driscoll et al. 2001; Schaberg et al. 2001; Driscoll et al. 2003; Watmough and Ouimet 2005; Fenn et al. 2006; Lawrence et al. 2012; and Duarte et al. 2013):

- Long-term acid deposition has caused increased soil acidification and depletion of exchangeable base cations [calcium (Ca^{2+}), magnesium (Mg^{2+}), potassium (K^+), and sodium (Na^+)] which has led to a general decline in percent base saturation (% BS) and pH, and an increase in bioavailable forms of aluminum (Al^{3+}). Acidification and depletion trends are variable, however, due to variation in acid deposition levels, soil buffering capacity, base cation uptake by vegetation, and base cation deposition levels.

- Concomitant with base cation depletion has been a general increase in soil sulphur (S) and nitrogen (N) concentrations in forest soils which are implicated in the slow or negligible recovery of many streams and lakes from reduced acid deposition – mainly through ongoing desorption and leaching of excess SO_4^{2-} ions with Al^{3+} and hydrogen (H^+). Another contributing factor is decreased soil % BS which is directly linked to decreased acid neutralizing capacity (i.e., less ability to neutralize acidity despite reductions in inputs).
- A general association between increasing soil acidification and declining tree growth has been observed since the middle of the 20th century, but impacts are variable and/or confounded by other contributing factors (e.g., species mix, climate gradients, and other disturbances agents).
- In addition to reduced productivity, chronic soil acidification can lead to reduced forest health and increased sensitivity to abiotic stresses, insect defoliation, and disease, especially on marginal sites (with respect to species suitability) and sites with poor buffering capacity. This is mainly due to nutrient deficiencies and “aluminum stress”.
- Nitrogen deposition can potentially increase productivity in N-limited ecosystems (fertilizer effect), but can also lead to nutrient imbalances and/or base cation depletion on sites where N is not limiting and/or where nitrification rates are enhanced leading to excess NO_3^- leaching.

Calcium Depletion and Forest Harvesting

In addition to acid leaching losses (and in the absence of fire), the other main source of nutrient removal from forest sites is through periodic harvesting (Federer et al. 1989). These nutrient losses occur directly through biomass removal, as well as indirectly through post-harvest leaching losses associated with increased forest floor and/or slash mineralization (Adams 1999). While it is relatively easy to estimate direct nutrient removals by using harvest mass data and

species-specific biomass concentrations (e.g., Pardo et al. 2005), potential post-harvest leaching losses are harder to predict and quantify because they depend on site-specific soil and vegetation conditions, timing and extent of harvest, post-harvest weather patterns, etc. As a result, post-harvest leaching losses can be negligible to substantial (e.g., Adams 1999).

Calcium Depletion and Aluminum Stress

There are several pH-buffering mechanisms in soil associated with different pH ranges. In general, soils are mainly buffered through silicate mineral weathering (i.e., base cation release) when pH is between about 6.2 and 5.0. This changes to mainly base cation exchange buffering when pH is between 5.0 and 4.2, and then Al and/or iron (Fe) hydroxide buffering when pH is about 4.2 or less (Tomlinson 1990). Using several forest soil samples from eastern Canada, Clark and Hill (1964 *in* Reuss and Walthall 1990) showed pH to be relatively consistent (between 4 and 5) over a large range of $\text{Ca}^{2+} + \text{Mg}^{2+}$ percent saturation values (20%-90%), thus indicating the dominance of base cation exchange in buffering pH in these soils.

In soils that have been impacted by acid deposition, concentrations of SO_4^{2-} , NO_3^- , and H^+ are increased. If increases in mobile SO_4^{2-} and NO_3^- are not offset by plant and microbial uptake (SO_4^{2-} and NO_3^-) and mineral soil adsorption (SO_4^{2-}), they will be leached from the soil. Given the requirement for electro-neutrality in aqueous solutions, cations must also be leached with excess anions, and when soil pH is in the range associated with cation exchange buffering (i.e., about 4.2-5.0), base cations (Ca^{2+} , Mg^{2+} , K^+ , Na^+) will make up most of the required positive charge in soil leachate. If these base cation losses are not offset by inputs from soil weathering and/or atmospheric deposition, then base cation depletion will occur – the overall

impact of which depends on initial base cation stores (particularly Ca^{2+} , Mg^{2+} , and K^+), the rate of loss, and the crossing of deleterious threshold values (to be discussed further below).

Base cation depletion and associated decreases in soil pH will eventually lead to increased Al^{3+} in solution via two mechanisms. As noted above, when soil pH drops into the low 4.0 range, further acid buffering becomes more and more associated with release of Al^{3+} from hydroxide minerals (simplistically represented by: $3\text{H}^+ + \text{Al}(\text{OH})_3 \leftrightarrow \text{Al}^{3+} + 3\text{H}_2\text{O}$) rather than through cation exchange, thereby increasing the concentration of exchangeable Al^{3+} in soil while also limiting the drop in pH. Despite this increase in available Al^{3+} , lower charged base cations can still be dominant in soil solution due to the stronger affinity of tri-valent Al^{3+} for cation exchange sites. However, when % BS drops to about 15%-20%, release of Al^{3+} into solution increases significantly (Reuss and Johnson 1985 *in* Reuss and Walthall 1990) and Al^{3+} becomes a major component of soil solution and leachate.

Increases in bioavailable Al^{3+} can cause stress and growth loss in plants, as well as high inputs of Al^{3+} into surface waters (with related toxicity impacts). Reduced plant growth is mainly due to reduced nutrient availability or imbalances, Al^{3+} interference with base cation uptake, and reduced fine root growth from Al^{3+} toxicity (e.g., Rengel 1992; Ouimet and Camiré 1995; Godbold et al. 2003; Lawrence et al. 2005; de Wit et al. 2010). Impacts of increased Al and decreased Ca availability on the health of red spruce and sugar maple (*Acer saccharum*) have been particularly well documented (e.g., DeHayes et al. 1999; Schaberg et al. 2006; Huggett et al. 2007; Ouimet et al. 2008; Long et al. 2009; Boyce et al. 2013).

The 15% BS threshold below which “aluminum stress” occurs (Cronan and Grigal 1995) is well established in the literature, and Driscoll et al. (2001) suggest 20% BS as a general value for assessing chemical recovery from acid deposition. Results from Whitfield et al. (2006) and

Keys et al. (2016) suggest that many forest soils in Nova Scotia are currently below one or both of these thresholds. However, the range in % BS values calculated from historic soil survey reports also suggest that not all soils have been equally impacted from acid deposition across the province (Table 1.1). Variation in % BS values shown in Table 1.1 likely reflect differences in acid deposition levels, buffering capacity, sampling year, natural variability, and limited sample size.

Although all base cations are affected by acid depletion and harvesting, it is Ca that has been the focus of most concern and related research in northeastern forests. This is due to:

- The dominant Ca fraction in soils compared to other bases (e.g., Lawrence et al. 1999),
- The documented widespread loss of Ca (e.g., Likens et al. 1998; Watmough and Dillon 2003; Bedison and Johnson 2010),
- The importance of Ca in overall forest ecosystem health – both terrestrial and aquatic (e.g., Schaberg et al. 2001; Houle et al. 2006),
- The importance of Ca in commercial tree productivity and health (e.g., DeHayes et al. 1999; Huggett et al. 2007), and
- The significant potential for added Ca loss through commercial harvesting compared to other bases (e.g., Federer et al. 1989; Adams et al. 2000).

In contrast, impacts of Mg and K depletion are mainly associated with overall increases in acidity (i.e., reduced % BS) and potential nutrient imbalances, but less commonly with outright limitations (e.g., Ouimet and Camiré 1995). This is somewhat in contrast to European forests where Mg deficiencies have more often been implicated in forest declines (e.g.,

Tomlinson 1990). Results of forest nutrient budget assessments in Nova Scotia generally support northeastern trends, with Ca and N estimated to be the main potential limiting nutrients, K occasionally a nutrient of concern, and Mg of little concern with respect to overall limitations (see Chapter 2).

Calcium Depletion and Climate Change

There are many potential impacts of climate change on forest health and productivity, both positive and negative. Campbell et al. (2009) discuss several of these in relation to northeastern forests. Broadly speaking, the most pertinent with respect to acid deposition, base cation depletion, and forest nutrient management are:

- changes in atmospheric carbon dioxide (CO₂) concentrations,
- changes in growing season length,
- changes in species composition,
- changes in soil weathering and nitrification potential,
- changes in precipitation patterns and leaching potential, and
- changes in soil organic matter contents and distribution.

Changes in atmospheric CO₂ concentrations and growing season length: Enhanced tree growth from increased available CO₂ and longer growing seasons could be a positive impact of climate change (Boisvenue and Running 2006). For example, growing degree days are predicted to increase significantly over most of Nova Scotia in the next 50 years (Bourque et al. 2008). However, for growth gains to be realized, trees also require adequate moisture and nutrients.

Even if moisture is not limiting, widespread base cation depletion could restrict potential growth due to limiting nutrient stores and Al interference with base cation uptake. Furthermore,

carbon (C) resources may be shifted more to root production than to aboveground biomass to offset damage caused by Al toxicity (Lapenis et al. 2013). If trees are being stressed by low nutrient or moisture conditions, longer growing seasons may only serve to increase these stresses, along with associated vulnerabilities to insect and disease attacks, frost damage, etc.

Changes in species composition: Climate change is expected to promote major tree species shifts in northeastern forests as habitat and competitive conditions change (Mohan et al. 2009). It is suggested that hardwood cover will increase overall at the expense of softwood cover, with species like balsam fir (*Abies balsamea*) and spruces (*Picea spp.*) being particularly impacted (Mohan et al. 2009; CCFM 2009). Notwithstanding other climate change influences on soil fertility, changes in species distribution will directly impact nutrient budgets and depletion trends through differences in canopy interception levels, rooting patterns, nutrient demands, biomass concentrations, and litter production characteristics (amount, timing, and quality).

In Nova Scotia, species habitat suitability modelling under current and projected climate scenarios clearly show the potential for decreased softwood and increased hardwood cover across the province (Bourque et al. 2008). However, how these potential shifts manifest themselves will depend on several factors including site conditions, disturbance patterns, and management impacts (Steenberg et al. 2011 and 2013). For example, Steenberg et al. (2013) discuss how red spruce presence may be significantly decreased by the combined impacts of climate change and harvesting versus climate change alone – a result of increased competitive advantage of post-harvest successional species like red maple (*Acer rubrum*) and trembling aspen (*Populus tremuloides*) under climate change conditions. Soil nutrient status will also play an important role with respect to climate change impacts on forest cover (e.g., LaFleur et al. 2010), but has yet to be fully integrated into forest management and climate change modelling in

the province. For example, in the red spruce scenario described above, projected impacts could be exacerbated by stresses associated with base cation depletion.

Changes in soil weathering potential and nitrification rates: Weathering is the main source of new base cation inputs in soils. Along with parent material characteristics, potential inputs are directly related to weathering rates as influenced by soil temperature and moisture conditions. Although climate change is expected to lead to warmer average temperatures which would promote weathering, the impact on precipitation (and related soil moisture) may be quite variable, adding to, or subtracting from, the positive temperature influence. One of the main areas of concern with respect to base cation budgets (and Ca in particular) is the potential for soil weathering to offset losses caused by depletion and harvesting. Where Ca is the main limiting nutrient, weathering responses related to changing climate conditions could be a key issue for predicting future forest growth (Campbell et al. 2009).

In addition to weathering rate impacts, climate induced changes in temperature and moisture regimes could result in increased rates of N nitrification in some forest soils, especially where C:N ratios have been lowered by increased N deposition. Nitrification can even occur in low pH soils when there is excess NH_4^+ present (Aber et al. 1989). This increase in NO_3^- production could lead to additional base cation leaching if this mobile anion is not taken up by plants and microbes. Leaching potential is also related to the timing and intensity of precipitation events (as discussed below).

Changes in precipitation patterns and leaching potential: Climate change is expected to result in significant changes to forest watershed hydrology in northeastern North America (Campbell et al. 2009). Expected changes include: (i) increased winter precipitation (much of it falling as rain), (ii) reduced snowpack and shortened snow season, and (iii) increased likelihood

and severity of damaging rainstorms (Frumhoff et al. 2007). A potential ramification of these changes is increased leaching of base cations and NO_3^- outside of the growing season (when uptake demands are low) and/or during storm events. There is also concern that more frequent soil freezing events (associated with low snowpack cover) could cause root damage and further leaching losses (Groffman et al. 2012). SO_4^{2-} leaching may also be enhanced by increased “watershed wetness” (Mitchell and Likens 2011). Overall, the potential for increased leaching is a concern for base cation budgets under future climate conditions (Huntington et al. 2009).

Changes in soil organic matter: Assuming adequate moisture contents, soil organic matter decomposition rates should be enhanced with the increase in average air temperatures predicted with climate change. Increased mineralization could, at least in part, provide the nutrients needed to realize potential growth gains associated with enhanced CO_2 concentrations and longer growing seasons. However, these potential inputs are not new sources of nutrients, only cycled inputs subject to potential loss through leaching and/or future harvesting. As such, their release and availability may only temporarily mask or delay productivity problems associated with base cation depletion and long-term nutrient deficits.

Organic matter in acidic forest soils is present in a variety of forms and size fractions (Reuss and Walthall 1990). Of particular interest are soil solution organic acids and colloidal material associated with pH-dependent CEC (Reuss and Walthall 1990; Johnson 2002). In coarse-textured forest soils (i.e., clay content $\leq 10\%$), organic matter is strongly correlated with effective CEC and is the main source of exchange sites in these soils (Johnson 2002). Al and H chemistry are intimately tied to the reactive organic matter fraction in acidic forest soils (Ross et al. 2008), so any impacts of climate change on soil organic matter contents and distribution will certainly impact nutrient status of these soils, especially coarse-textured soils that are already the

most sensitive to leaching. Also, given the fundamental connection between organic matter dynamics and soil organisms, decomposition and distribution will also depend on soil flora and fauna responses to changing soil temperature and moisture regimes (as influenced by pH and N dynamics).

Calcium-Based Soil Amendments

Given the well documented impacts of acid deposition and harvesting on base cation nutrient stores (as possibly exacerbated by climate change), it is perhaps surprising that there has been little to no use of soil amendments to mitigate the effects of increased acidity and base cation depletion in northeastern forest soils. Although there have been scattered watershed liming treatments conducted to offset stream acidity (e.g., Smallidge et al. 1993; Clair and Hindar 2005) and various research trials aimed at assessing Ca and/or lime amendment impacts on forest ecosystems (notably in the Hubbard Brook Experimental Forest in New Hampshire), these have not resulted in any widespread or routine use of soil amendments in the region (Moore et al. 2015). This is despite recognition of the potential for liming amendments to improve forest soil conditions (Tomlinson 1990; Lawrence et al. 2016).

Lime has been used in some European countries since the 1980s to mitigate the impacts of acidic deposition (e.g., Tomlinson 1990; Nilsson et al. 2001), and there is increasing use of wood ash in Nordic countries to help offset the impacts of biomass harvesting and acidic deposition on long-term site productivity (e.g., Levin and Eriksson 2010). In addition to nutrient management, use of wood ash in these forests also “closes the loop” with respect to using woody biomass for energy, and provides an environmentally sound end-use for this waste product. Use of wood ash in Canada is also being investigated as a potential soil amendment to offset nutrient

losses. In a recent trial in Ontario, Pugliese et al. (2014) found ash amendments to be of overall benefit with little negative impact on soil chemical or biological processes. However, they also indicated that ash properties (and therefore appropriate application rates) were variable and dependent on source supply.

Despite the potential for lime or wood ash amendments to offset soil acidity and base cation depletion, there can be negative impacts if these amendments are improperly used. For example, when application rates are too high: (i) microbial, meso-fauna, and ground vegetation communities can be significantly altered; (ii) organic matter decomposition rates can be greatly accelerated; and (iii) NO_3^- production can be increased beyond ecosystem demands leading to increased leaching (Kreutzer 1995; Pitman 2006). As is usually the case in forestry, these risks are soil and/or site-type specific. In addition, treatment responses can be highly variable, as recently discussed by Reid and Watmough (2014) in their meta-analysis of liming and wood ash treatment effects. These authors found that, in general, soil pH and foliar Ca concentrations were more responsive to liming treatments, whereas tree growth and % BS were more responsive to ash treatments. However, there were also a significant number of studies assessed where neither treatment resulted in any significant response, or where response varied by site conditions.

Papermill sludge and municipal biosolids (sewage sludge) have also been used as forest soil amendments in some jurisdictions (Matysik et al. 2001). Papermill sludge can be a significant source of base cations (e.g., Evanylo et al. 2004), but its potential use is limited by source location and the type of pulping process used which affects sludge chemistry (Scott and Smith 1995). In addition, this material often has a very high C:N ratio which can negatively impact post-treatment decomposition and N availability (Matysik et al. 2001). Municipal biosolids that meet regulatory standards for land application are mainly a source of N and

phosphorous (P) (Cogger et al. 2006), unless they have been alkaline-treated in which case they are considered more of a liming amendment (USEPA 2000).

Alkaline treatment involves mixing biosolids with an alkaline material such as cement kiln dust (CKD) to generate pH levels unfavorable for pathogen growth. Class A biosolids requirements (no detectable pathogens) can be achieved when the pH of the mixture is maintained at or above 12 for at least 72 hours, with a temperature of 52 °C maintained for at least 12 hours during that time (USEPA 2000). Although alkaline-treated biosolids (ATB) are routinely used in agriculture, their use in northeastern forests has been minimal to date (Banaitis et al. 2009). Appropriate use of ATB could, however, offset nutrient deficiencies and base saturation declines caused by acid deposition and forest harvesting, while also providing another end-use for this waste-stream product. It also represents a potentially untapped market for ATB which could be of economic and social benefit. Initial routine use of soil amendments like ATB in northeastern forests would seem most appropriate in plantation sites where nutrient demands are the greatest and amendment application the easiest.

OBJECTIVES

To quantify the potential need for liming amendments in Nova Scotia forest soils, and to evaluate the potential for using ATB to offset current or predicted Ca deficits in intensively managed plantation sites, a research project was initiated with the following objectives:

- Assess current condition and long-term nutrient sustainability of a range of spruce plantation soils/sites in central Nova Scotia.

- Comprehensively assess the effects of surface applied ATB on soils and vegetation in two representative white spruce (*Picea glauca*) plantations.
- Further evaluate ATB as a potential liming amendment by comparing nutrient and metal ion dynamics in ATB, lime, and fly ash amended soils under controlled greenhouse conditions.

The bulk of this thesis (Chapters 2 to 6) is comprised of one published and four draft manuscripts. Chapter 2 describes a nutrient budget model developed for Nova Scotia along with field work conducted to assess current soil and site conditions in 25 spruce plantations. Data collected were used to model long-term sustainability of plantation management regimes and to identify site types most in need of Ca amendments. Chapters 3 to 5 describe results of two field trials where ATB was surface applied under white spruce plantations in central Nova Scotia. Experimental design, forest floor responses, and surface soil ion flux responses are described and discussed in Chapter 3, followed by discussion of mineral soil and leachate chemistry responses (Chapter 4) and vegetation responses (Chapter 5). Chapter 6 describes a greenhouse trial where ATB, lime, and fly ash were applied to uniform soil samples collected from field trial sites to assess and compare amendment related ion fluxes over a 10-week period. The last chapter (Chapter 7) presents summary conclusions and recommendations for future work.

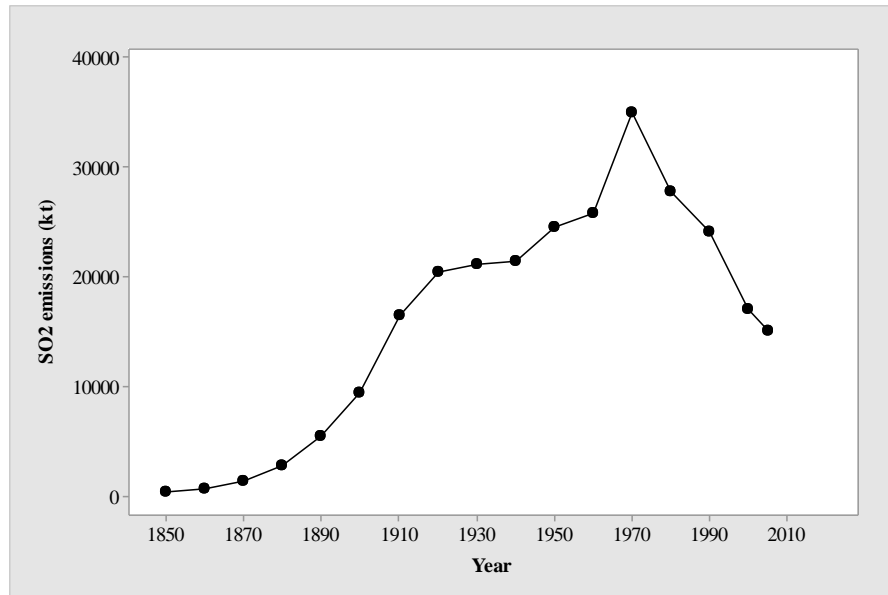


Figure 1.1. Estimated total SO₂ emissions for the United States and Canada by decade (from Smith et al. 2011).

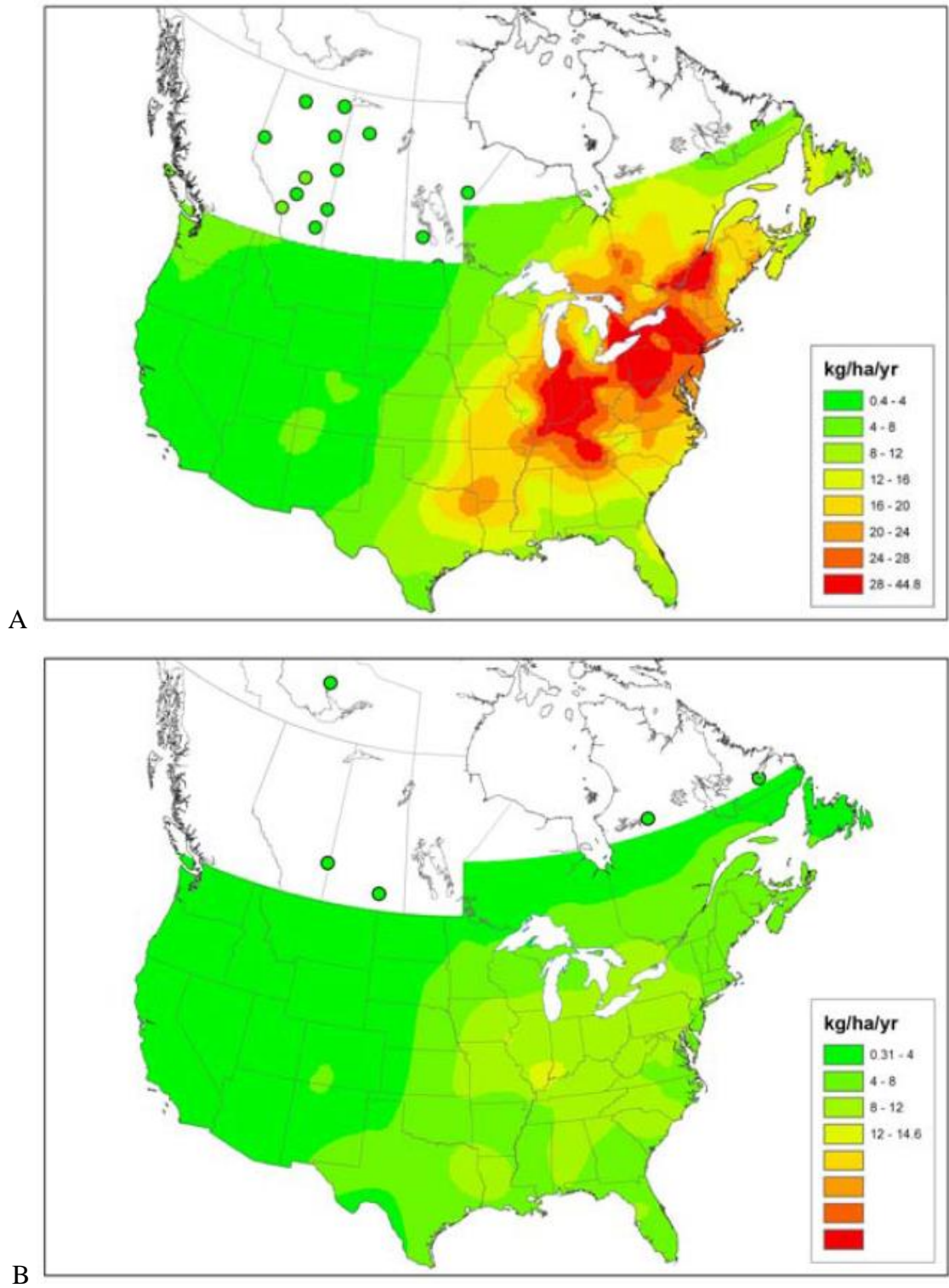


Figure 1.2. Wet deposition of non-sea-salt SO_4^{2-} in 1990 (A) and 2010 (B) for the United States and eastern Canada (from CCME 2013).

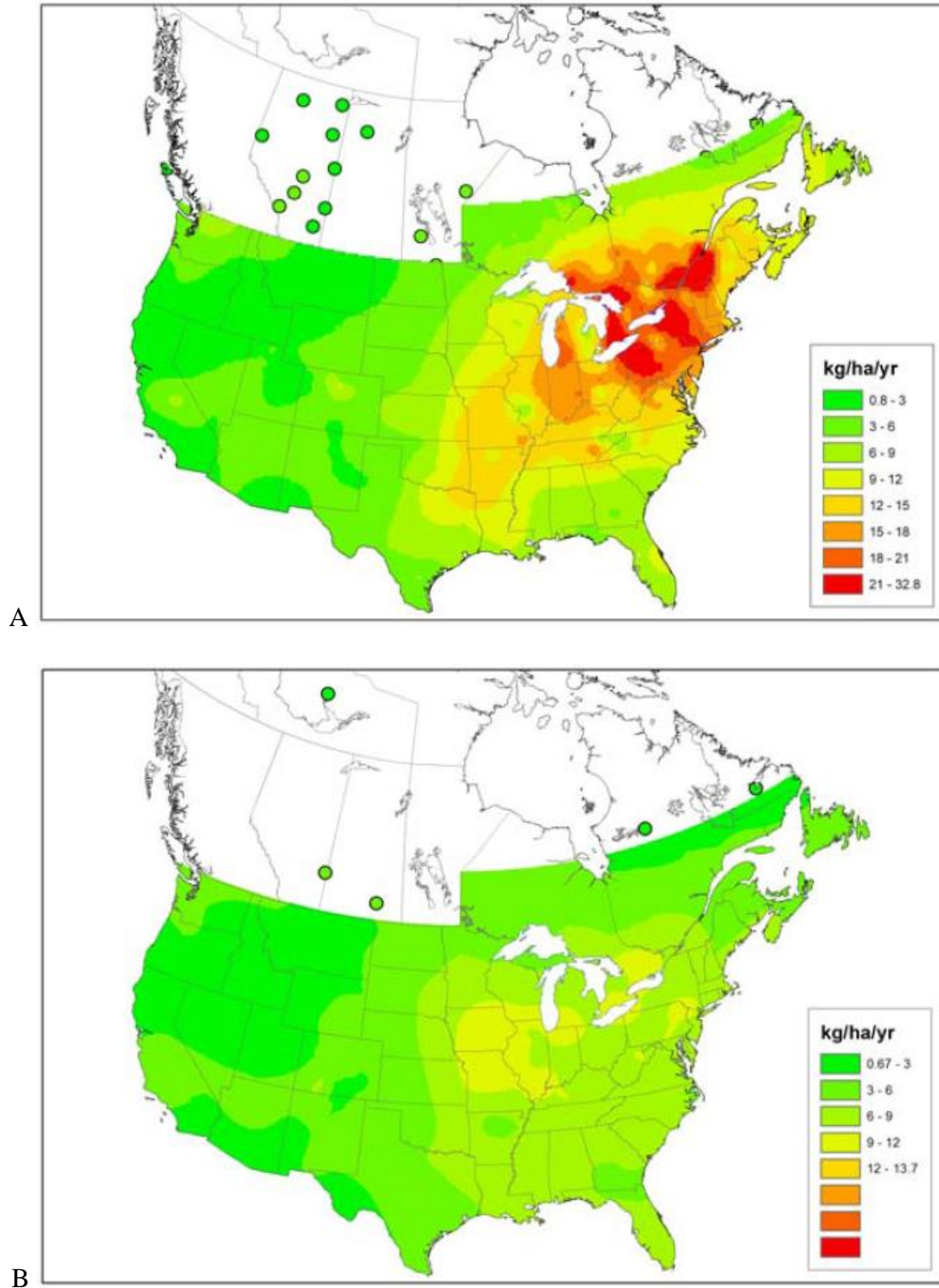


Figure 1.3. Wet deposition of NO_3^- in 1990 (A) and 2010 (B) for the United States and eastern Canada (from CCME 2013).

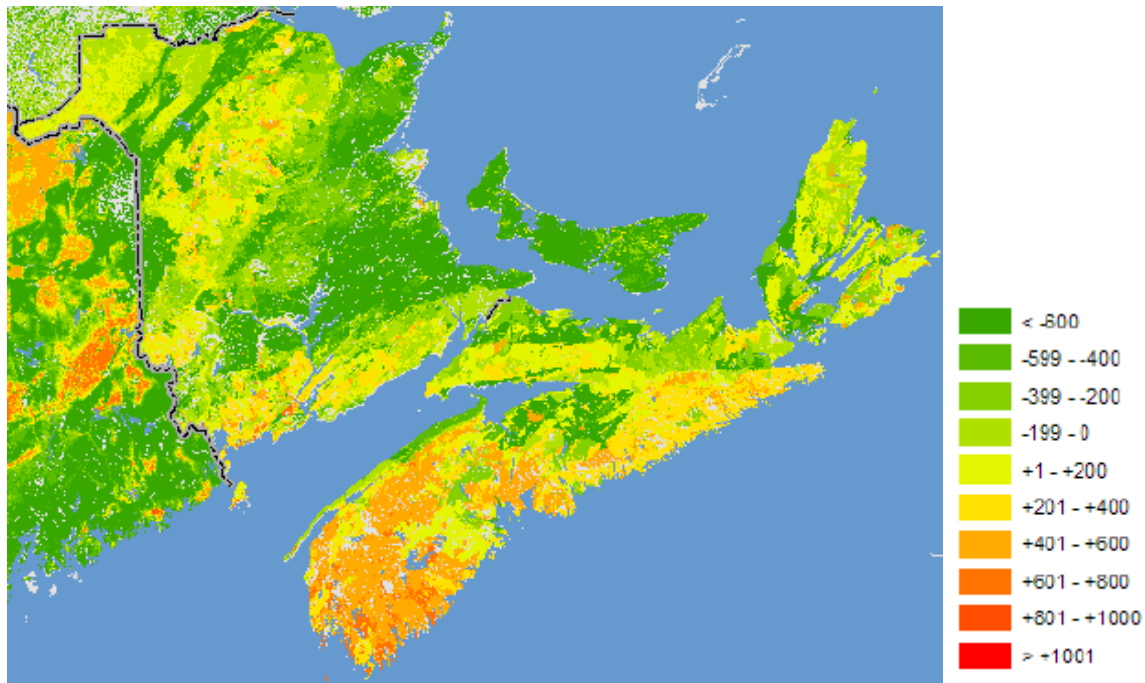


Figure 1.4. Critical load exceedances (expressed as mole equivalents) for the Maritime Provinces based on 2002 deposition data ($\text{eq ha}^{-1} \text{yr}^{-1}$). In this context, critical load is defined as the level of exposure to acidic sulphur (S) and nitrogen (N) compounds below which no harmful effects are known to occur. Positive numbers represent exceedance while negative numbers represent non-exceedance (from NEG-ECP 2007).

Table 1.1. Percent base saturation (% BS) range for 10 dominant soil series found in Nova Scotia as calculated from available data in soil survey reports (for non-agricultural soils). Values do not include Na and are based on effective CEC reference values.

Soil Series	BS%	# of Profiles
Gibraltar	3-11	3
Halifax	2-6	4
Queens	4-59	10
Thom	3-42	7
Bridgewater	3-9	6
Millbrook	6-24	5
Wolfville	5-24	4
Shulie	3-44	5
Westbrook	3-29	6
Cobequid	8-17	3

CHAPTER 2

A SIMPLE GEOSPATIAL NUTRIENT BUDGET MODEL FOR ASSESSING FOREST HARVEST SUSTAINABILITY ACROSS NOVA SCOTIA, CANADA *

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ABSTRACT

A geospatial GIS-linked spreadsheet model (Nutrient Budget Model – Nova Scotia: NBM-NS) was developed for Nova Scotia to assess the long-term sustainability of forest harvest scenarios as constrained by primary nutrient inputs and outputs due to atmospheric deposition, soil weathering, and leaching. Harvest scenarios refer to user-defined, stand-specific removal rates of bolewood, bark, branches, and foliage based on current or projected forest inventories. These scenarios are evaluated within the context of existing data layers for current climate (mean annual precipitation and air temperatures), atmospheric deposition (N, S, Ca, Mg, K), and soil/substrate types, supplemented by species-specific look-up tables containing expected

biomass fractions and nutrient concentrations. This paper introduces this model to assess relative site quality and limiting nutrients for red spruce and sugar maple across Nova Scotia. This is followed by an output comparison involving 25 spruce plantations whereby NBM-NS determinations derived using “default” soil survey data are compared with those derived using plantation-specific soil data. Model output shows that (i) Ca and N are the main growth limiting nutrients across Nova Scotia, (ii) currently projected plantation yields are generally not sustainable on sites underlain by slowly weathering soils, (iii) current soil base cation contents are generally lower than what is reported in historic soil survey reports, and (iv) model results are expected to vary within the context of changing climate, acid deposition levels, and data accuracy.

KEYWORDS

Forest Nutrition Management, Biomass Harvesting, Sustainable Forest Management, Forest Plantation Management

INTRODUCTION

Although sustainable forest management can be defined in many ways, sustainability of forest harvesting is ultimately dependent on the availability of growth-limiting nutrients (Ågren, 1985; Sverdrup and Rosen 1998). Harvesting and acid depletion lead to nutrient losses from a site, thereby reducing soil/site nutrient pools (Tew et al. 1986). If these exports exceed primary nutrient inputs, then repeated harvesting coupled with soil acidification stress would be expected to cause nutrient deficiencies (de Vries et al. 1995; Oja and Arp 1996) leading to declines in forest health and productivity (Ouimet et al. 2001). Although the potential for productivity

decline is usually associated with forest biomass or whole-tree harvesting (e.g., Thiffault et al. 2011), nutrient deficiencies are also possible with more conventional stem-only harvesting if harvest demands are high or sites are nutrient poor.

In simple nutrient budget terms, demand refers to nutrients removed from a site through forest harvesting and acid depletion, whereas supply refers to nutrient inputs from atmospheric deposition and soil weathering (Moayeri 2000). A sustainable balance is achieved when demand is less than or equal to supply in the context of a complete harvest rotation; i.e., all nutrients lost through harvesting and depletion need to be replenished before the next rotation comes into effect. In this regard, forest nutrient budgets can vary widely across landscapes based on differences in climate, atmospheric deposition, soil type, and vegetation type (Ranger and Turpault 1999).

Allometric relationships using traditional forest inventory data can be used to calculate harvest-based nutrient removals in considerable detail. This includes tracking species-specific nutrient removals by bolewood, bark, branch, and foliage biomass compartments (e.g., Freedman et al. 1986; Arthur et al. 1999). Atmospheric deposition, soil acidification, and weathering inputs can be estimated with regional survey datasets and maps (e.g., Arp et al. 2001; Ouimet et al. 2006; Nasr et al. 2010). Finally, necessary soil data can be acquired from general surveys or site-specific assessments.

This paper describes a regional nutrient budget model (NBM-NS) developed for Nova Scotia, Canada that can be used (i) to estimate and map landscape and stand-level primary nutrient supplies in relation to modelled atmospheric deposition and soil weathering rates, and (ii) to relate these supplies to potential harvest demands from current or projected stand inventory data, with or without imposing acid rain induced soil acidification. This is followed by

a case study showing how NBM-NS was used to assess the long-term nutrient sustainability of 25 spruce plantations across several soil and site types in central Nova Scotia within the context of currently available province-wide data layers for climate, atmospheric deposition, and soils.

METHODS

Model Development

NBM-NS is a geospatial, GIS-linked spreadsheet model that focusses on linking major nutrient and acid loading drivers, namely calcium (Ca), magnesium (Mg), potassium (K), nitrogen (N), and sulphur (S), to forest growth in terms of per hectare per year biomass accumulations. Only primary nutrient inputs and outputs are considered. Secondary nutrient cycling processes, while important, are assumed to only result in a temporal redistribution of nutrients within forest stands. Potential nutrient losses from episodic fires are also not considered because fires in actively managed forests are generally suppressed, while impacts of historic fires are, in part, reflected by current site/soil conditions. The following geospatial databases were used for NBM-NS input and ArcMap processing:

- Total mean annual atmospheric deposition coverage layers for S and N, and wet atmospheric deposition coverage layers for Ca, Mg, and K were obtained from the National Atmospheric Chemistry Database and Analysis System (NATChem), Meteorological Service of Canada (Ro and Vet 2002; 50 km² resolution). Base cation layers were adjusted for total deposition using a series of wet:dry deposition ratios generated by way of a watershed study for southwestern Nova Scotia (Yanni 1996; Yanni et al. 2000).

- Climate data coverages for mean annual precipitation rates and air temperature were obtained from Environment Canada's National Climate Archive (Canadian Daily Climate Data, 2004-2008; 10 km² resolution; AAFC 2009).
- Provincial forest inventory stand coverage was obtained from the Nova Scotia Department of Natural Resources.
- Provincial Soil Survey Reports and Maps were obtained from the Canadian Soil Information System (CanSIS) database from Agriculture and Agri-Foods Canada (<http://sis.agr.gc.ca/cansis/publications/surveys/ns/index.html>).

Soil parameters within NBM-NS include: (i) percent clay (% Clay); (ii) percent organic matter (% OM); (iii) percent coarse fragments (% CF); (iv) bulk density (Db); (v) potential rooting depth (Depth); (vi) percent base saturation (% BS); (vii) percent total nitrogen (% N); (viii) effective (unbuffered) cation exchange capacity (CEC); (ix) Ca, Mg, and K fractions on cation exchange sites; and (x) total cation exchange sites (CES). All these data were compiled or calculated for each soil series across Nova Scotia using available soil survey data (Appendix 2.1) and/or derived pedo-transfer functions (Noseworthy 2011). Predicted data were then reviewed and adjusted, as needed, to match criteria in soil series descriptions and trends observed from approximately 1,500 soil pits assessed for the Nova Scotia Forest Ecosystem Classification (FEC) project (Neily et al. 2013). For example, predicted % Clay could not be outside the range defined for the soil series, and soil depth needed to be within the range found in related FEC plots. Final “default” soil values were then assigned to respective GIS soil series polygons mapped across the province.

Soil weathering rates in NBM-NS are estimated using the Clay Content method (Sverdrup et al. 1990; de Vries et al. 1992) – adjusted for *Db*, % *CF*, and % *OM* – which calculates the total rate of base cation weathering (BC_{we}^0) within a soil matrix as follows:

$$BC_{we}^0 = Db \text{ Depth} \left(1 - \frac{CF}{100}\right) EXP \left(\left(\frac{A}{(2.6 + 273)} \right) - \left(\frac{A}{(273 + Tann)} \right) \right) \left(\frac{ClassX}{100} \right) \left(1 - \frac{OM}{100}\right),$$

where *Db* is the soil bulk density ($g \text{ cm}^{-3}$), *Depth* is the soil rooting space depth (cm), *CF* is the soil coarse fragment content (%), *A* is the Arrhenius pre-exponential factor ($3600 \text{ J mol}^{-1} \text{ }^\circ\text{C}^{-1}$; Sverdrup et al. 1990), the constant 273 is the conversion from degrees Celsius to Kelvin, 2.6 is a reference temperature for northern climates ($^\circ\text{C}$) (de Vries 1991), *Tann* is the mean annual air temperature ($^\circ\text{C}$), and *OM* is the soil organic matter content (%).

ClassX refers to soil texture dependent parent material class weathering estimations (de Vries 1991) calculated as:

$$Class\ 1 = 56.7 \text{ Clay} - 0.32 \text{ Clay}^2,$$

$$Class\ 2 = 500 + 53.6 \text{ Clay} - 0.18 \text{ Clay}^2,$$

$$Class\ 3 = 500 + 59.2 \text{ Clay},$$

$$Class\ 4 = 1500 + 59.2 \text{ Clay},$$

where *Clay* refers to average % *Clay*.

Class 1 represents acidic substrates such as those comprised of sand, gravel, and glacial tills high in acidic rock (granite, quartzite, non-calcareous sandstones, etc.). *Class 2* represents

intermediate substrates such as marine sediments; medium to fine textured glacial tills derived from slates, shales, and mudstones; and medium to coarse textured tills containing less acidic rock than Class 1. *Class 3* represents more basic substrates such as gabbro and basalt dominated glacial tills, along with nutrient-enriched alluvium deposits. *Class 4* represents calcareous substrates such as limestone, gypsum, calcareous sedimentary deposits, and related tills. All soil series polygons were assigned one of these substrate classes based on their parent material attributes.

All compiled and derived data layers were geospatially aligned and intersected with provincial forest inventory maps, using ArcMap procedures, to create a spatial ecounit layer that informs about mean annual atmospheric deposition rates (precipitation, S, N, Ca, Mg, K), air temperature, tree species composition, soil conditions, and weathering rates for each mapped forest stand. Continuous raster data (deposition and temperature) were averaged for each ecounit. Where ecounits were associated with more than one soil series polygon, ecounit values were area-weighted by soil polygon composition.

Harvestable biomass (M_{Har}) compartments are calculated per stand ($Mg\ ha^{-1}$) using an oven-dry wood-density correct formulation:

$$M_{Har} = \sum_{i=1}^{\infty} \left[\begin{array}{l} (D_i V_i) \text{Bolewood}_i + \\ (D_i V_i) \text{Bark}_i + \\ (D_i V_i) \text{Branch}_i + \\ (D_i V_i) \text{Foliage}_i \end{array} \right],$$

where subscript i denotes each tree species in the stand (%), D_i represents species-specific stem densities taken from Gonzalez (1990), V_i is the total merchantable volume of each species in the

stand ($\text{m}^3 \text{ha}^{-1}$), and *Bolewood*, *Bark*, *Branch*, and *Foliage* denote the Lambert et al. (2005) generated biomass fractions, each prorated to stem biomass as follows:

$$\text{Compartment biomass} / \text{Stem biomass} = A_i \exp [B_i \ln(\text{DBH}_i)]$$

where DBH_i refers to diameter at breast height (tree of average basal area), and A_i and B_i are species and compartment-specific biomass proration numbers (Appendix 2.2, Noseworthy 2011).

Nutrient losses due to harvesting (X_{Harvest}), expressed in eq ha^{-1} , are calculated from:

$$X_{\text{Harvest}} = \sum_{i=1}^{\infty} \left[\begin{array}{l} (D_i V_i) (Stem_i [X_{\text{Bolewood}}]_i) + \\ (D_i V_i) (Bark_i [X_{\text{Bark}}]_i) + \\ (D_i V_i) (Branch_i [X_{\text{Branch}}]_i) + \\ (D_i V_i) (Foliage_i [X_{\text{Foliage}}]_i) \end{array} \right] 10^6 / X_{g/eq}$$

where X refers to one of four nutrients (Ca, Mg, K, N), $[X_i]$ refers to biomass nutrient fraction, and the subscript g/eq denotes the equivalent weight of element X (Ca = 20, Mg = 12.2, K = 39.1, N = 14; g eq^{-1}). Listed in Appendix 2.2, by selected tree species, are (i) oven-dried bolewood densities, (ii) stem-based A and B biomass proration coefficients by tree compartment, and (iii) biomass N, Ca, Mg, and K concentrations per biomass compartment.

Total uptake of base cations (BC_{up}) and nitrogen (N_{up}) for each forest site were calculated from:

$$BC_{up} = (Ca_{HS} + Mg_{HS} + K_{HS}) / Age \text{ and } N_{up} = N_{HS} / Age,$$

where the subscript *HS* denotes user-determined harvest scenarios (i.e., the proportion of bolewood, bark, branch, and foliage removed) and *Age* is the average stand age (years). All uptake equations are expressed in eq ha⁻¹ yr⁻¹.

Nutrient deficiencies refer to the difference between nutrient inputs (atmospheric deposition and weathering) and nutrient outputs due to harvesting. A positive value (gain) indicates a sustainable harvest rate, while a negative value (deficit) indicates an unsustainable harvest rate (assuming repeated harvests under the same harvest scenario and stand/site conditions). For nitrogen, potential gains or deficits are estimated from: $N_{def} = N_{dep} - N_{up}$. For base cations, potential gains or deficits are estimated from $X_{def} = X_{dep} + X_{we} - X_{up}$, with *X* referring to Ca, Mg, or K. All gains or deficits are expressed in eq ha⁻¹ yr⁻¹.

In terms of base and acid cation leaching due to the combination of atmospheric deposition and vegetative uptake, it follows that:

$$BC_{le} = BC_{dep} + BC_{we} - BC_{up},$$

with subscripts *dep*, *we*, *up* and *le* referring to atmospheric deposition, soil weathering, uptake by vegetation, and leaching, respectively. All terms are expressed in eq ha⁻¹ yr⁻¹.

The base cation leaching rate for upland forest soils was estimated from the law of mass action and charge conservation given by:

$$K_{exch} = \frac{CES - BC + \Delta x}{BC - \Delta x} \frac{BC_{le} + \Delta x}{AC_{le} - \Delta x},$$

where K_{exch} is the cation exchange ratio (set at 10 in order to reflect the adsorption preference of mineral soil surfaces for H^+ and Al^{3+} over adsorption of competing base cations – NEG-ECP 2001; Nasr et al. 2010), CES refers to the sum of soil cation exchange sites ($eq\ ha^{-1}$), BC is the sum of exchangeable soil base cations ($eq\ ha^{-1}$), Δx is the annual exchange of acid to base cations (leaching or accumulation) that shifts base saturation from the current state (BS) to the final state (BS_f). BC_{le} and AC_{le} are the base and acid cation leaching rates, with AC_{le} denoting H^+ and Al^{3+} ions given by:

$$AC_{le} = S_{dep} + N_{dep} - N_{up} + BC_{up} = \frac{1}{K_{exch}} \frac{CES - BC}{BC} BC_{le},$$

and S_{dep} denoting atmospheric S deposition. For soils with AC_{le} and $BC_{le} \ll BC$, one obtains:

$$\Delta x \approx \frac{100 (BS - BS_f)}{BS BS_f (K_{exch} - 1 + 100/BS)} BC_{le},$$

with BS and BS_f as % base saturations when $\Delta x \neq 0$ (i.e., current base saturation) and when $\Delta x = 0$ (i.e., final base saturation), with $BS = 100 BC/CES$ and:

$$BS_f = \frac{100}{1 + K_{exch} AC_{le} / BC_{le}}.$$

Sustainability of a harvest scenario is determined by way of the Sprengel-Liebig Law of the Minimum (van der Ploeg et al. 1999). This is related to (i) harvest deficiencies alone or (ii) in combination with acid-induced base cation depletion estimates:

$$\min \left[N_{def}^{Export}, Ca_{def}^{Export}, Mg_{def}^{Export}, K_{def}^{Export}, 0 \right], \quad \underline{\text{or}}$$

$$\min \left[N_{dpl+def}^{Export}, Ca_{dpl+def}^{Export}, Mg_{dpl+def}^{Export}, K_{dpl+def}^{Export}, 0 \right],$$

where $\min[...]$ selects the growth-limiting nutrient for each stand. Each harvest scenario is considered sustainable when minimum values as evaluated above are greater than zero. A sustainable harvest rate (expressed as sustainable mean annual increment – *SusMAI* – in $\text{m}^3 \text{ha}^{-1} \text{yr}^{-1}$ of solid wood) is then derived based on (i) the annual input of the most limiting nutrient, (ii) the mass-weighted nutrient concentration per harvested biomass, and (iii) tree species composition.

Case Study

For the plantation survey (25 spruce plantations, Figure 2.1, Table 2.1), soils were sampled during fall, 2012 from three representative locations in each plantation based on visual assessment of ground conditions (distance between plots was about 100 m or more). Pits were shovel-excavated and samples collected at two depths (approximately 0-15 cm and 30-45 cm) for chemical and texture analysis. A soil corer was used to collect bulk density samples at these same depths. Visual estimates of potential soil rooting depth and coarse fragment volume were also recorded for each pit.

Soil samples were analyzed individually for chemistry and Db, and were pooled by plantation and depth for texture analysis. All chemical analyses were carried out by Analytical Services, Nova Scotia Department of Agriculture using standard in-house procedures (i) Mehlich 3 extraction followed by ICP analysis for base cations, (ii) LECO combustion for N, (iii) loss-on-ignition at 450 °C for 1 hour for % OM, and (iv) summation of base cations and exchangeable acidity for % BS and CEC. Clay content (hydrometer method) and Db analyses (CF corrected) were carried out at Dalhousie University Agricultural Campus in Truro, NS. Chemical data were expressed on an area basis using measured Db before calculating final average values for each plantation soil.

RESULTS and DISCUSSION

Model Projections across Nova Scotia

Total atmospheric deposition maps for S, N, Ca, Mg, and K are shown in Figure 2.2. N and S deposition follow a west-east gradient (high to low) directly related to inputs from the northeast United States. Base cation trends are more variable, with Ca deposition being highest along the northwest (Bay of Fundy) coast and northern Cape Breton Island, and lowest along the Atlantic coast and central mainland. Deposition patterns for Mg and K are relatively the same with the lowest levels found in north central parts of the province, increasing eastward into Cape Breton and (to a lesser extent) along the southwest coast. Figure 2.3 presents a range of default soil data determined or derived from provincial soil survey reports (% clay, % carbon, weatherability class), or based on more recent forest soil assessments (soil depth, % BS). These maps show the range of soil conditions across Nova Scotia that influence site productivity.

To compare relative site productivity across the province based on default soil data, atmospheric deposition, and weathering inputs (without the confounding influence of variable tree cover), NBM-NS was run assuming all stands had the same species and stocking levels. One run assumed 80-year-old red spruce (*Picea rubens*) with qdbh of 20 cm and 60% stocking with an average merchantable volume of 247 m³ ha⁻¹ (Figure 2.4). The resulting estimates for sustainable mean annual increment (SusMAI) values ranged from less than 3.0 to more than 8.0 m³ ha⁻¹ yr⁻¹ (Figure 2.4). These values were mainly driven by soil characteristics and substrate type, with the lowest values associated with very coarse, acidic (Class 1), shallow soils; and the highest with deeper, medium to fine-textured (Class 2 and higher) soils. SusMAI values were also modified by atmospheric deposition, especially where N was calculated to be the growth limiting nutrient. For example, estimated red spruce SusMAI on Gibraltar soil (a well-drained, coarse-textured, Class 1 soil limited by Ca) only ranged from 5.2 to 5.4 m³ ha⁻¹ yr⁻¹, whereas values on Queens soil (an imperfectly drained, fine-textured Class 2 soil limited by N) ranged from 7.3 to 9.0 m³ ha⁻¹ yr⁻¹.

Another run assuming uniform cross-province coverage of 80-year-old sugar maple (*Acer saccharum*) with qdbh of 22 cm, 60% stocking, and average merchantable volume of 210 m³ ha⁻¹ resulted in estimated SusMAI values ranging from less than 1.0 to more than 3.5 m³ ha⁻¹ yr⁻¹ (Figure 2.4). *Note:* As a rule of thumb, hardwood MAI in Nova Scotia is about half that of softwood on the same site due to differences in growth form and basal area capacity (NSDLF 1990; McGrath 2011). Estimated SusMAI trends for sugar maple and red spruce mainly differ by growth-limiting nutrient, with Ca being more growth limiting for sugar maple than for red spruce (Figure 2.5) because of higher Ca demands (Appendix 2.2). The strip of K-limited area along the

northwest (Bay of Fundy) coast was mainly associated with shallow Rossway soils on thin glacial till deposits over basalt bedrock.

Plantation survey

A summary of measured plantation soil data is presented in Table 2.2. Entries are based on pooled plantation data, not individual samples, and provide insight into the similarities and differences between various soil series. For example, Halifax and Cobequid soils are derived from gravelly, sandy loam tills, but are differentiated by rock type, and – hence – by mineralogy (MacDougall et al. 1963; Webb et al. 1991). In contrast, Queens soils are derived from non-gravelly, loam to clay loam till (Webb et al. 1991). Sampling results within these soils were variable, with Mg and K fraction data having consistently high coefficient of variation values (21-68%).

Table 2.3 reports on field-determined and corresponding NBM-NS default soil data for the surveyed plantations. In all cases, field-determined values for % BS and % N were lower than corresponding default data, with relative decreases ranging from -37% to -82% (% BS) and -19% to -71% (% N). These differences would, at least in part, be due to the agricultural bias of early soil surveys in Nova Scotia. In addition, Nova Scotia soils were subject to acid rain caused soil acidification over several decades prior to the systematic US and Canada-wide reductions in SO₂ emissions since 2000 (Whitfield et al. 2006). The low field-determined % BS values found were similar to those reported by Whitfield et al. (2006). Field-determined CEC values were also generally lower than default values (range -59% to +21%). Trends in CEC were, however, poorly correlated with changes in % Clay and % OM. This could, in part, be due to small sample

size and appreciable scatter between field-determined and modelled CEC values (Meyer et al. 1994).

Differences between default and actual base cation (Ca, Mg, K) fractions showed variable trends (range from -58% to +221%), with field-determined values generally greater than default values. Simultaneous increases in Ca, Mg, and K fractions would be due to the assigned fractional value given to Na in NBM-NS. The approximate weathering fraction reported by Whitfield et al. (2006) for Na is 0.30. As can be derived from Table 2.3, actual Na fractions from sampled soils ranged from 0.09 to 0.19, i.e., in agreement with Yanni et al. (2000) who found exchangeable Na to be generally low in western Nova Scotia despite high rates of atmospheric Na deposition (a considerable portion of exchangeable Na^+ is thought to be lost through Cl^- leaching).

For the remaining parameters, differences between default and actual values for % Clay (-32% to +36%), % CF (-33% to +125%), Db (-10% to +25%), Depth (-13% to + 10%), and pH (-6% to +2%) showed no clear trends; and although field-determined % OM values were higher for all soils (+5% to +113%), absolute differences only varied from 0.4% to 2.3%. In general, differences between site-specific and survey derived default values should be expected. Nevertheless, differences so accruing can affect NBM-NS output. Hence, using site-specific data is important for verifying and improving NBM-NS modelled results.

A summary of NBM-NS output using default and mean plantation soil data, together with projected plantation growth data, is presented in Table 2.4 (*Harvesting Only* losses) and Table 2.5 (*Harvesting + Depletion*). All harvesting assumed 100% removal of stem wood and bark with 0% removal of branches and foliage (i.e., a very efficient conventional stem-only harvesting

processed at the stump). A summary of plantation regime sustainability by model configuration is given in Table 2.6.

The MAI Diff. entries in Tables 2.4 and 2.5 were used to compare model calculated SusMAI with merchantable mean annual increment (MMAI) by projected management regime. Negative MAI Diff. values indicate that the projected (or desired) yields over the long term (several rotations with same yield expectations) cannot be sustained without increased nutrient inputs or reduced harvest expectations. When MAI Diff. is positive, projected yields are predicted to be nutrient sustainable over the long term under current atmospheric deposition conditions. In both cases, growth limiting nutrient(s), i.e., those that constrain SusMAI, are listed in order of importance. Several points arise from Tables 2.4-2.6:

- Based on comparisons with NBM-NS output, approximately $\frac{1}{4}$ to $\frac{1}{2}$ of the assessed plantation sites have non-sustainable MMAI yield expectations.
- Best MAI-sustainability outcomes are obtained by enabling NBM-NS depletion functions and using site-specific soil data. This occurs when the $K_{exch} AC_{le}/BC_{le}$ specified BS_f value is larger than the field-determined BS value. In this case, base cation depletion calculations generate a positive result whereby some BS_f -determined leaching of base cations can be taken up by vegetation while still maintaining current BS value. However, soil quality, tree health, and rooting vigour must be considered as well (see discussion below).
- Plantations with non-sustainable MMAI values are mainly associated with low soil weathering classes (especially Class 1) and/or tree species with high nutrient demands (e.g., Norway spruce – *Picea abies*).
- Ca and N are the most common growth limiting nutrients.

To better understand how site-specific soil data and model configuration influences NBM-NS output, a sample plantation with a range of output is examined in further detail (Table 2.7). Entries in this table refer to default and site-specific soil data for Plantation 19 (white spruce underlain by Perch Lake soil), and to the changes in predicted SusMAI by adjusting each individual soil parameter for the *Harvest Only* and *Harvest + Depletion* scenarios. As shown, the largest single impact on SusMAI under the *Harvest Only* scenario (+53%) comes from the 71% increase in Ca fraction above its default value (Ca being the growth limiting nutrient at this site, Table 2.4). The 25% increase in clay also has a positive impact on SusMAI (+17%), but this would – in part – be compensated by default differences in soil depth and density. Overall, using plantation versus default data as NBM-NS input leads to an estimated 60% SusMAI increase (to $4.6 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$). This increase, however, is still not sufficient to match the projected (or desired) MMAI of $5.6 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ (Table 2.4).

Under the *Harvest + Depletion* scenario, SusMAI may be greater or smaller than what is estimated by harvest deficiency alone, with Table 2.7 suggesting an increase in SusMAI towards $6.1 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$. Highest SusMAI expectations via NBM-BS are obtained when all available base cations obtained through weathering and atmospheric deposition are taken up by vegetation. This, however, would allow for no replenishment of depleted base cations on CES, and BS_f could theoretically drop to zero while tree growth and fine root vigour would be substantially impaired by way of increased Al^{3+} concentrations (Driscoll et al. 2001; Duarte et al. 2013). Therefore, when % BS levels are low, it is even more important to monitor base cation removals from harvesting so as not to exacerbate the problem by way of increased MMAI expectations. Cronan and Grigal (1995) suggest 15% BS as a threshold below which “aluminum stress” occurs in forest soils, and Driscoll et al. (2001) suggest 20% BS as a general value for assessing soil

recovery from already incurred acid deposition impacts. Any % BS value can be directly assigned within NBM-NS, so sustainable harvest rates can be based on these threshold values rather than using current or default values. In the case of Plantation 19, assigning % BS values of 15% and 20% resulted in estimated SusMAI values of $3.7 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ and $2.9 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ respectively (using site-specific data for other variables). Both of these values are below the projected (or desired) MMAI of $5.6 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ for this site, as well as the SusMAI value of $4.6 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ obtained when considering harvesting deficiencies alone.

NBM-NS Assumptions and Limitations

Based on its design, NBM-NS can only evaluate a single site condition and harvest scenario combination over time. As such, this model does not accommodate changing cover types or multiple interventions over time within a single model run. Other site productivity components such as potential ground disturbance and climate change impacts also need to be considered. In addition, NBM-NS does not account for N-fixation, or for seepage effects on nutrient budgets in low-lying locations. While long-term nutrient sustainability across forest sites can be addressed, current input data may require site-specific corrections. For example, Ca has long been considered a nutrient of concern in eastern North America (Freedman et al. 1986; Federer et al. 1989; Adams et al. 2000; Huntington 2005; Campbell et al. 2009), and this is supported by NBM-NS output which identifies Ca as the most frequent growth limiting element in addition to N. As suggested above, loss of Ca (and other base cations) from decades of acid deposition has probably contributed to the low % BS values found in plantation soils compared to values derived from historic soil surveys.

With Ca recognized as a common limiting nutrient in eastern forest soils (Huntington 2005), it is important to accurately model soil weathering rates. Whitfield et al. (2006) showed weathering rates estimated via the Clay Content method to be comparable to those obtained using more elaborate methods (Zirconium Depletion and PROFILE model) for five Nova scotia sites with very low clay contents (30-130 eq ha⁻¹ yr⁻¹). These values are generally lower than those generated by NBM-NS for this study (Table 2.8), but plantation soils had higher clay contents and/or a higher substrate weatherability class than the acid soils assessed by Whitfield et al. (2006). The range in estimated weathering input shown in Table 2.8 (196-967 eq ha⁻¹ yr⁻¹) is in keeping with values compiled by Ouimet and Duchesne (2005) for other northeastern sites. This confluence of results increases overall confidence over and above any single estimate (Futter et al. 2012), and justifies the use of the more easily applied Clay Content method within NBM-NS.

The main problem in using the Clay Content method comes from appropriately assigning soil/substrate weathering classes. For the NBM-NS case study, Halifax, Perch Lake, Queens, Millbrook, Bridgewater, and Diligence soils were easily assigned a weathering class, but Cobequid and Thom soils required *Class 1* to *Class 2* intermediate assignments. In future, weathering rates could also be impacted by climate-induced changes in soil temperature and moisture regimes, with longer growing seasons potentially enhancing not only soil weathering, but also base cation uptake during the growing season and base cation leaching outside the growing season (Oja and Arp 1996; Campbell et al. 2009; Huntington et al. 2009).

NBM-NS can be used as both a landscape planning tool (using default data) and a stand-level planning tool (using site-specific soil data). This study suggests, however, that care must be taken when using old soil survey data to initialize nutrient budget models such as NBM-NS.

Nova Scotia is one of the few provinces in Canada that has essentially all of its area covered by soil surveys, but the accumulated database may not be reflective of current forest soil conditions, as apparent from the case study change towards lower % BS values. The model is, however, easily updated as new soil data become available.

Atmospheric deposition data used in NBM-NS can also be updated when available. For this paper, we used the 2002 atmospheric deposition maps for S, N, Ca, Mg and K from Environment Canada. However, according to Canada's NATChem database for atmospheric deposition, the Canadian Air and Precipitation Monitoring Network (CAPMoN) stations at Kejimikujik National Park (Latitude 44:26:0, Longitude 65:12:21 and Jackson (Acadia: Latitude 45:35:35, Longitude 63:50:30) suggest the following volume-weighted trends:

- a steady decline for SO_4^{2-} deposition since 1985 with SO_4^{2-} deposition = $49.6 - 0.0244(1983 + \text{number of years})$, $R^2 = 0.75$;
- a more scattered decline pattern for $\text{NO}_3\text{-N}$ deposition, with NO_3^- deposition = $29.5 - 0.0145(1983 + \text{number of years})$, $R^2 = 0.54$;
- a fairly steady deposition rate for NH_4^+ at 0.15 mg/L ($R^2 = 0.0003$), and
- a slight increase for Ca^{2+} , Mg^{2+} , and K^+ combined at 8 $\mu\text{eq/L}$ ($R^2 = 0.094$).

These trends imply a general reduction in atmospheric acid deposition, which – in turn – would reduce soil acidity, increase soil base saturation, but lower the amount of available soil N. As a result, N rather than Ca and K could become more growth limiting.

Management Implications

Although differences in site quality and species suitability are well understood by forest managers, it is often assumed that increased yields predicted through intensive management are sustainable on any given site. This perception is likely due to (i) the long time frames associated with forest management, including plantation management, (ii) the relatively short history of intensive forest management in Canada, (iii) gradual rather than abrupt changes in growth limiting factors, and (iv) the lack of effective tools to evaluate forest nutrient sustainability.

Although not perfect, models like NBM-NS (combined with necessary GIS data) allow forest managers to better evaluate planned management regimes with respect to regional and local nutrient inputs, and to make adjustments to accommodate predictable nutrient deficits. For example, species selection, percent removals, and rotation lengths can all be adjusted to varying degrees to reduce the amount and timing of nutrient outputs and related shortfalls. In some cases, as in intensive plantation management, soil amendments could also be applied to offset nutrient losses from harvesting and continued soil leaching.

Although recent NATChem data shows that acid deposition across Nova Scotia has decreased, the problem and legacy of base cation depletion is still a concern in northeastern forests (e.g., Driscoll et al. 2001; Watmough 2005; Johnson et al. 2008; Warby et al. 2009). Also, while decreasing N inputs can potentially reduce cation leaching where N is not limiting, it also means less available N for sites that are N-limited, thereby potentially reducing potential productivity on these sites.

CONCLUSIONS

This paper describes a geospatial, GIS-linked nutrient budget model (NBM-NS) that can be used to:

- predict long term sustainability of forest harvesting regimes in relation to primary nutrient input and output estimates;
- estimate sustainable mean annual increment (SusMAI) values for individual stands or sites by determining limiting nutrient levels; and
- develop nutrient-based site quality assessments based on soil/substrate characteristics and atmospheric deposition data.

Although specifically designed for use in Nova Scotia, a similar model could be developed for any region assuming necessary data are available. When used to assess the sustainability of 25 sample spruce plantations, NBM-NS predicted nutrient declines for several sites, suggesting the need to adjust long-term yield expectations and/or management regimes for these site types.

It is recommended that nutrient budget assessments (such as provided by NBM-NS) become an integral component of sustainable forest management planning, especially when considering intensive management regimes or biomass harvesting for energy. Nutrient assessments are even more important in areas that have been impacted by long-term acid deposition since harvest removals can exacerbate declines in base cation levels (especially Ca) in affected soils. In doing so, it is important to ensure that model input is as accurate as possible, since budget estimates are directly related to soil/site conditions.

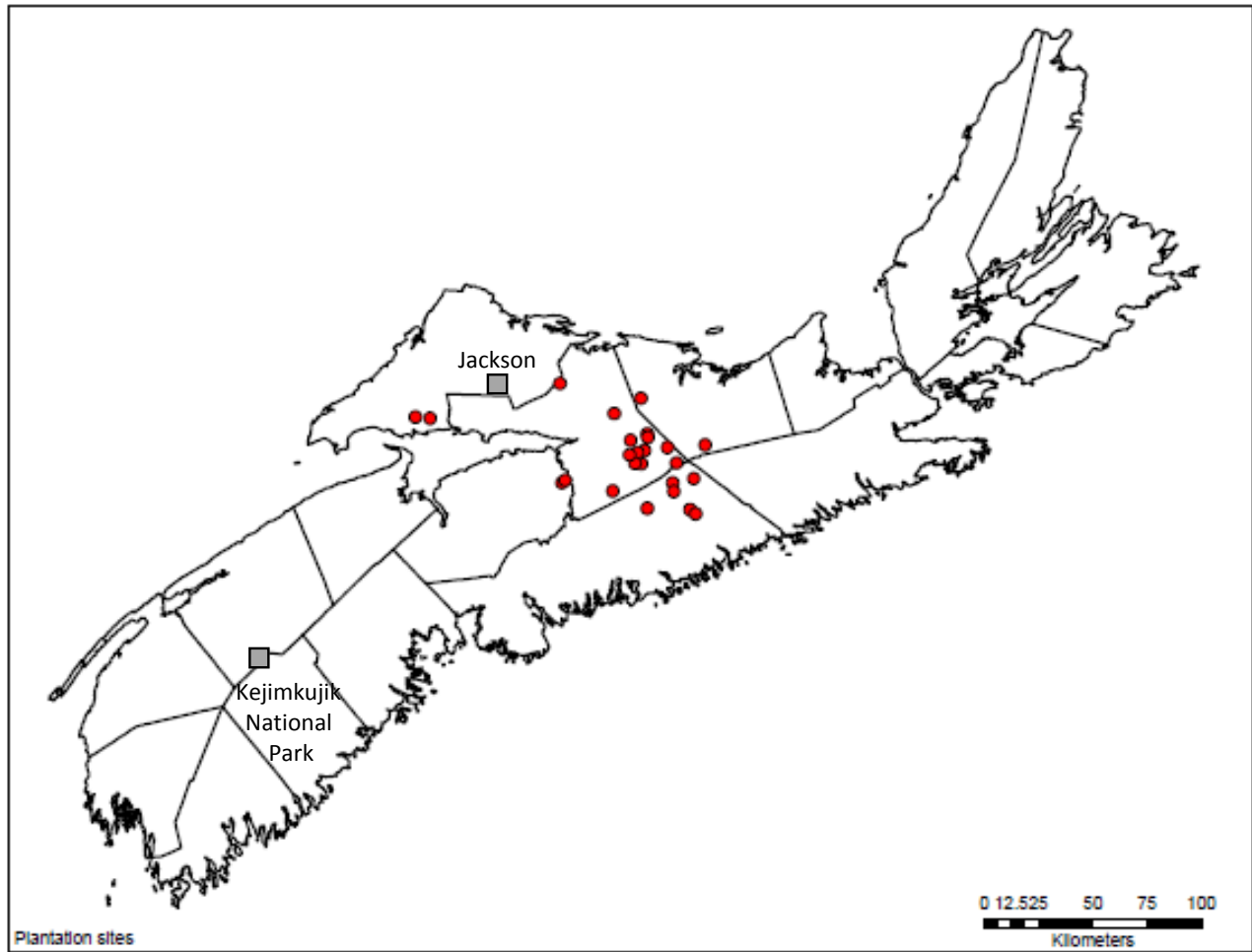


Figure 2.1. Case study plantation locations in central Nova Scotia, also showing the NATChem database locations for the CAPMon atmospheric deposition monitoring stations at Kejimikujik National Park and Jackson.

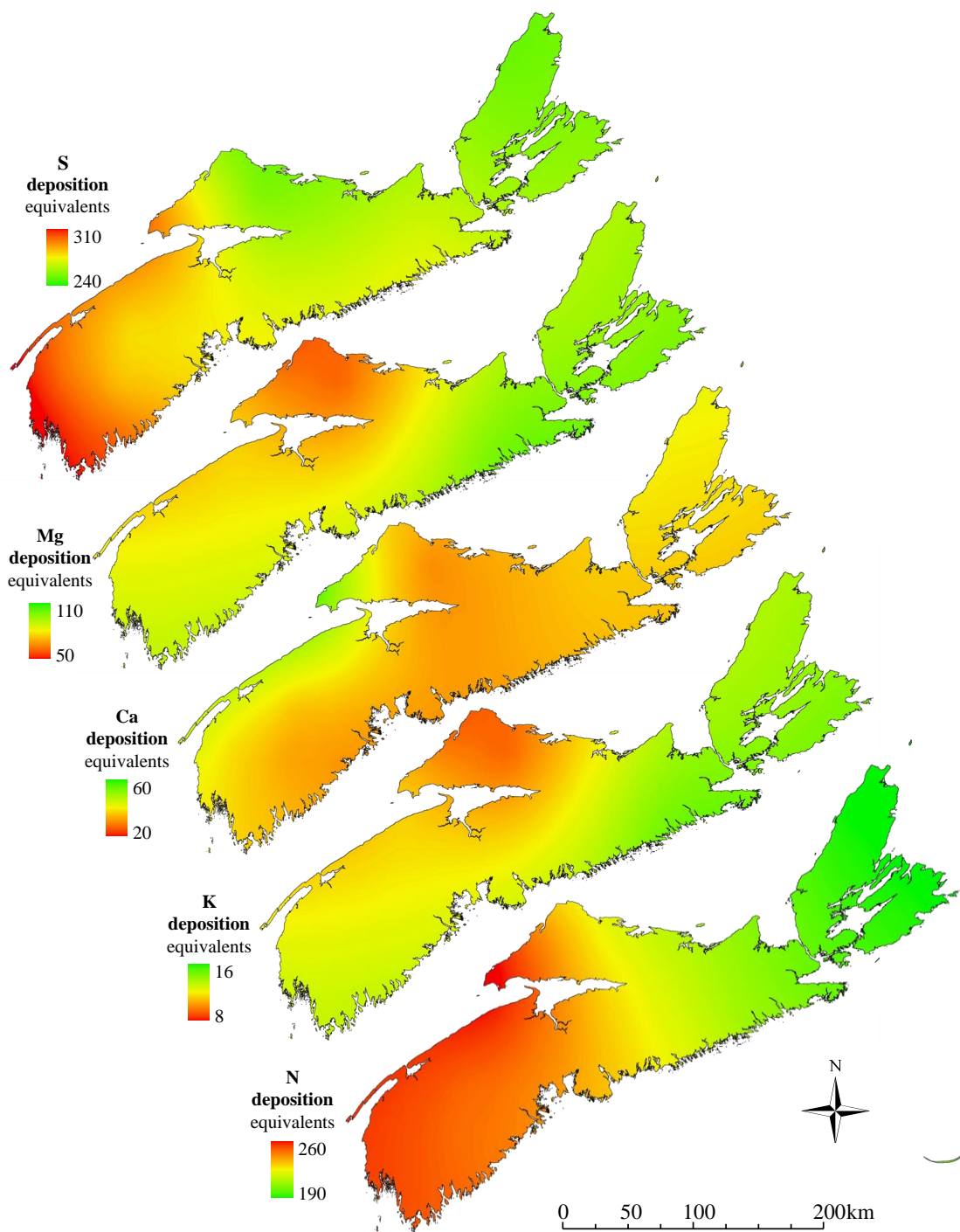


Figure 2.2. Estimated wet deposition rates for sulphate ($S-SO_4^{2-}$), magnesium (Mg^{2+}), calcium (Ca^{2+}), potassium (K^+), and nitrogen ($N-NO_3^- + N-NH_4^+$) based on the National Atmospheric Chemistry Database and Analysis System (NATChem) dataset.

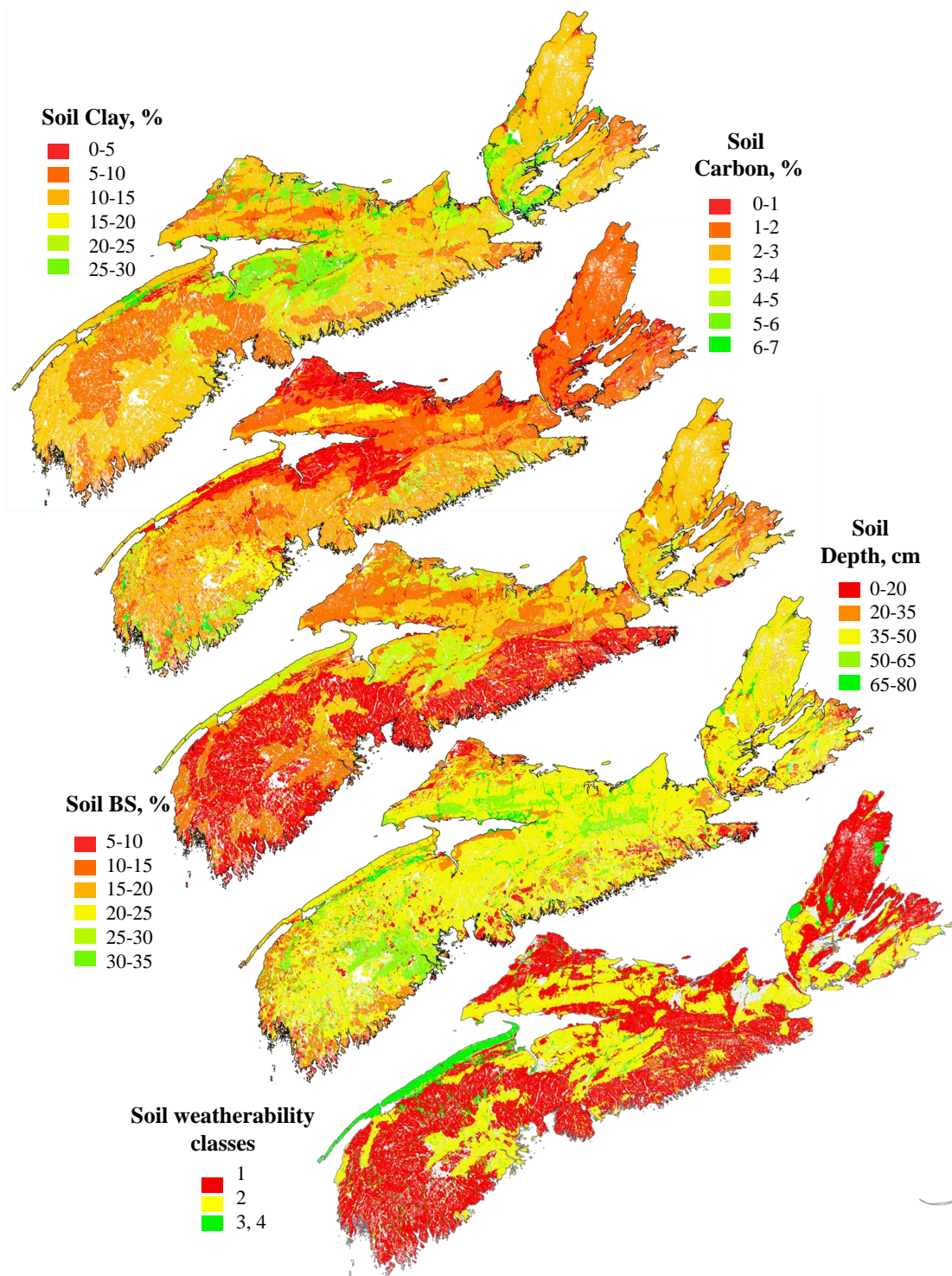


Figure 2.3. Distribution of soil properties (clay %, carbon %, base saturation %, soil depth) and assigned soil substrate weatherability classes for Nova Scotia. Maps do not include water or organic soil. Note: Soil % BS estimates are based on current available data, not historic soil survey data.

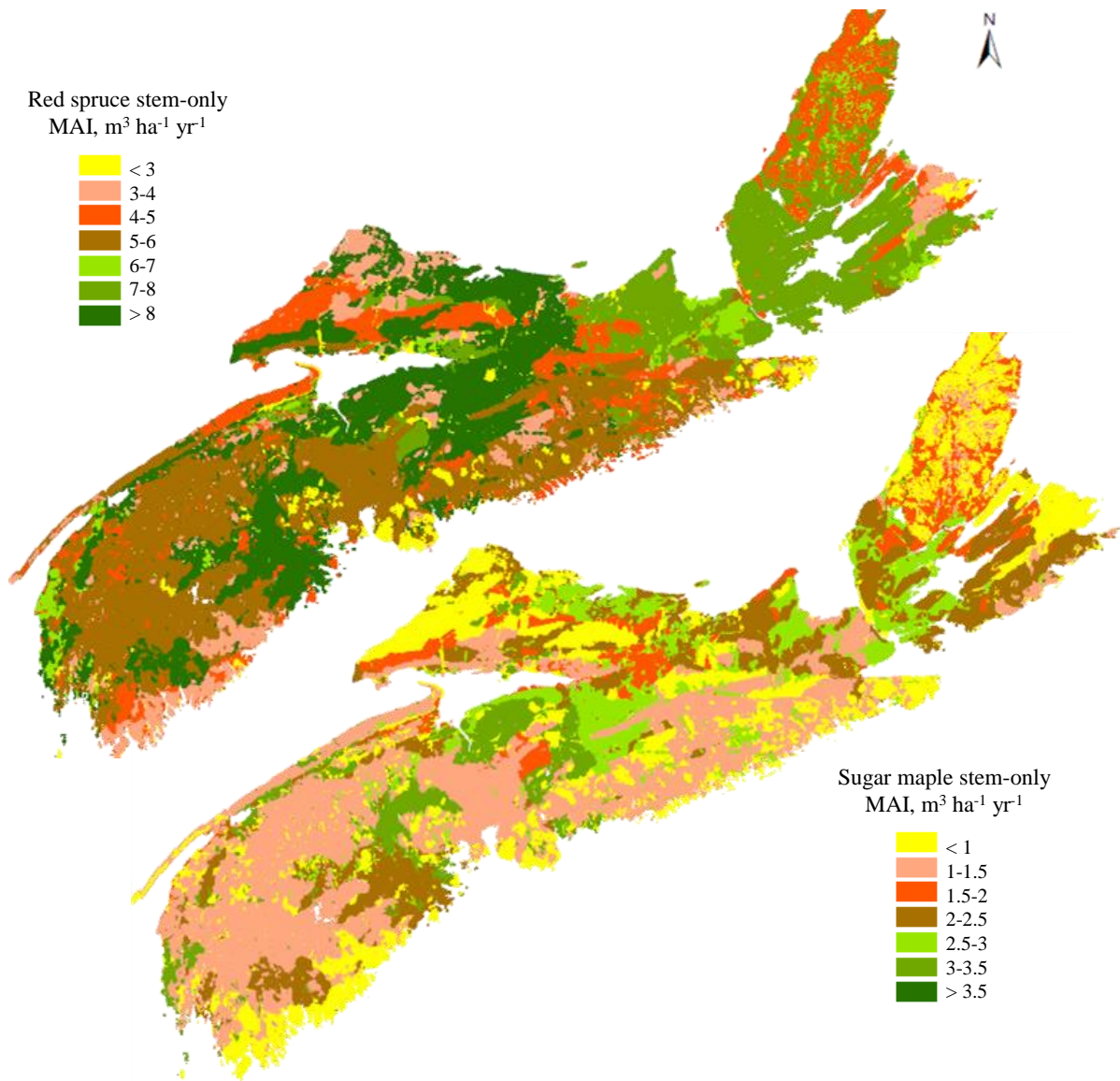


Figure 2.4. Predicted sustainable mean annual increment (MAI) across Nova Scotia generated by the NBM-NS model assuming default soil conditions and uniform red spruce (top) and sugar maple (bottom) cover.

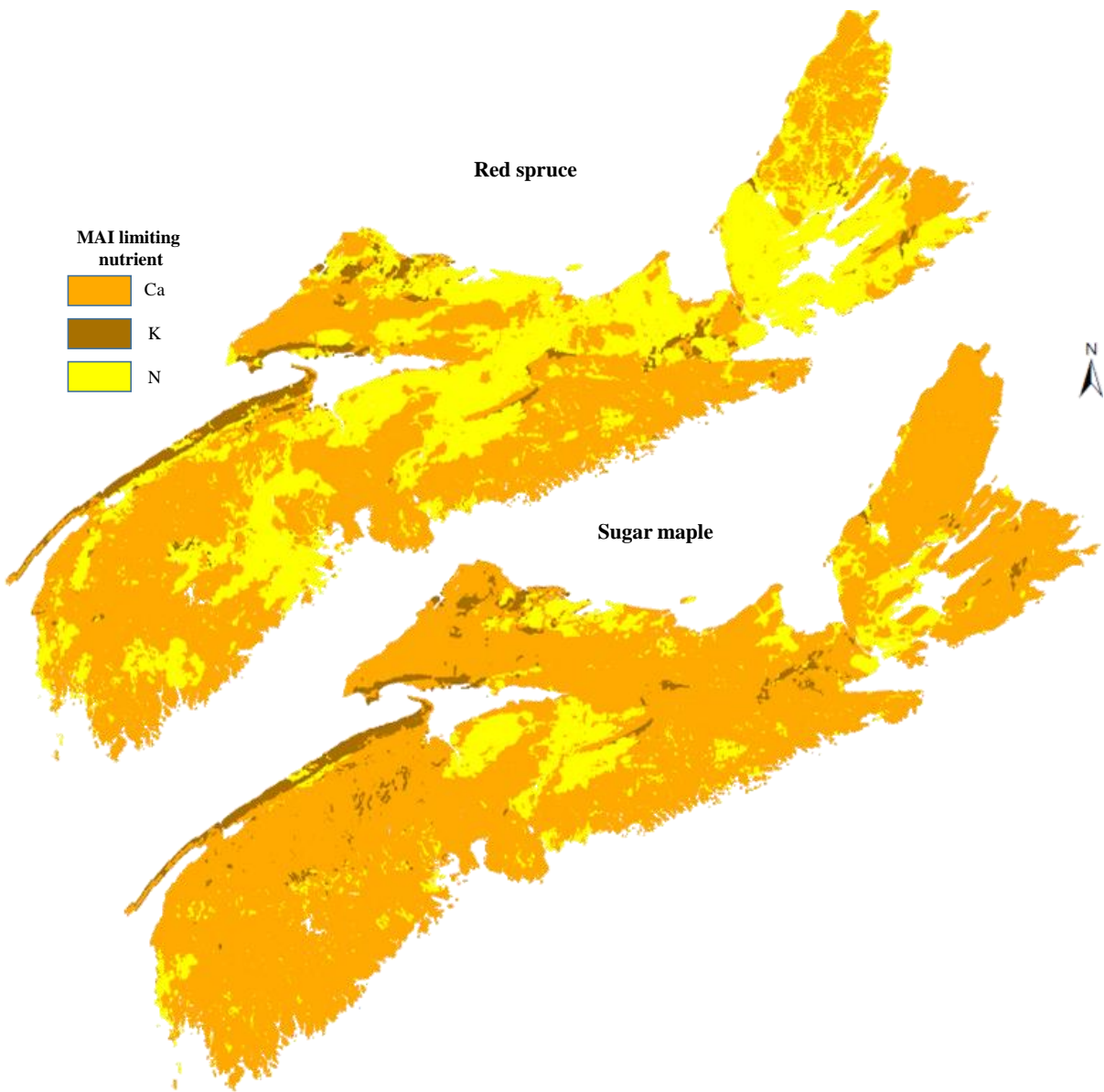


Figure 2.5. Map showing predicted range of growth limiting nutrients across Nova Scotia under (A) uniform red spruce cover and (B) uniform sugar maple cover. Orange = calcium (Ca), Yellow = nitrogen (N), Brown = potassium (K).

Table 2.1. Summary of sampled soils and plantations.

Soil series	Plantations	Spruce species	Age range (yrs)
Bridgewater	2	white/black	7-16
Cobequid	4	white/black/red	8-21
Halifax	4	white/black	6-18
Millbrook	4	white/black	5-12
Perch Lake	3	white/Norway	5-16
Queens	4	white/black/Norway	6-28
Thom	3	white/red	7-12
Diligence	1	white	4-8

White spruce (*Picea glauca*), black spruce (*Picea mariana*), red spruce (*Picea rubens*), Norway spruce (*Picea abies*).

Table 2.2. Summary of measured plantation soil attributes.

Soil Series	Statistic	% Clay	% OM	% CF	Db (g cm ⁻³)	Depth (cm)	% BS	Ca- Fraction	Mg- Fraction	K- Fraction	pH (H ₂ O)	% N	CEC (cmol + kg ⁻¹)
Halifax	Mean	8	5.1	32	1.06	55	4.7	0.54	0.15	0.12	4.8	0.17	7.8
(SWC-1)	Stdev.	4	1.2	9	0.13	7	2.3	0.14	0.05	0.03	0.1	0.02	1.3
	CV(%)	46	23	27	13	13	49	26	35	22	2	11	17
Queens	Mean	23	2.2	11	1.51	37	15.7	0.53	0.27	0.11	4.7	0.09	7.4
(SWC-2)	Stdev.	2	0.2	6	0.01	2	6.9	0.13	0.06	0.07	0.1	0.01	1.1
	CV(%)	8	10	50	1	6	44	24	22	68	2	10	15
Cobequid	Mean	11	6.3	29	1.05	54	4.6	0.54	0.15	0.15	4.7	0.19	9
(SWC-2)	Stdev.	3	1.2	3	0.11	17	0.8	0.07	0.05	0.04	0.2	0.03	1
	CV(%)	25	19	12	10	31	17	13	32	27	4	18	11
Millbrook	Mean	26	3.4	23	1.32	40	9.3	0.6	0.15	0.14	4.6	0.13	7.3
(SWC-2)	Stdev.	4	0.8	1	0.09	3	3.2	0.04	0.05	0.05	0.1	0.03	0.6
	CV(%)	14	22	5	6	7	35	7	34	32	2	24	9
Thom	Mean	16	4.9	34	1.09	53	4.9	0.59	0.14	0.14	4.7	0.16	7.9
(SWC-2)	Stdev.	5	0.7	12	0.13	7	1.4	0.19	0.05	0.08	0	0.01	0.9
	CV(%)	33	14	35	12	13	28	32	38	60	0	7	11
Perch Lake	Mean	15	4.9	30	1.11	52	6.4	0.58	0.12	0.14	4.7	0.17	7.3
(SWC-1)	Stdev.	3	0.7	6	0.08	11	2.2	0.24	0.07	0.08	0.1	0.02	0.2
	CV(%)	20	14	21	8	21	34	41	59	61	1	13	3
Bridgewater	Mean	14	5.2	35	1.13	48	4.4	0.44	0.2	0.17	4.8	0.18	6.7
(SWC-2)	Stdev.	5	0.3	15	0.04	9	0.6	0.01	0.05	0.04	0.1	0.03	0.5
	CV(%)	34	5	42	3	20	14	2	27	21	2	14	7
Diligence	Mean	34	4.3	13	1.3	35	6.3	0.4	0.27	0.23	4.5	0.13	9.2
(SWC-2)	Stdev.	na	na	na	na	na	na	na	na	na	na	na	na
	CV(%)	na	na	na	na	na	na	na	na	na	na	na	na

*SWC = assigned Soil Weatherability Class

Table 2.3. Comparison of soil survey derived (Default) and measured (Plantation) soil attributes.

Soil Series	Data Source	% Clay	% OM	% CF	Db (g cm ⁻³)	Depth (cm)	% BS	Ca- Fraction	Mg- Fraction	K- Fraction	pH (H ₂ O)	% N	CEC (cmol + kg ⁻¹)
Halifax (SWC-1)	Default	12	4.5	30	1.10	50	15.0	0.44	0.21	0.05	4.8	0.58	9.5
	Plantation	8	5.1	32	1.06	55	4.7	0.54	0.15	0.12	4.8	0.17	7.8
	% Diff.	-32	14	7	-4	10	-69	21	-32	166	1	-71	-18
Queens (SWC-2)	Default	25	1.5	5	1.50	40	25.0	0.44	0.12	0.14	4.9	0.27	10.9
	Plantation	23	2.2	11	1.51	37	15.7	0.53	0.27	0.11	4.7	0.09	7.4
	% Diff.	-10	47	125	1	-9	-37	22	117	-25	-4	-67	-33
Cobequid (SWC-2)	Default	15	5.0	35	1.10	55	25.0	0.52	0.12	0.06	4.8	0.24	8.4
	Plantation	11	6.3	29	1.05	54	4.6	0.54	0.15	0.15	4.7	0.19	9.0
	% Diff.	-23	26	-16	-4	-2	-82	4	26	135	-2	-19	8
Millbrook (SWC-2)	Default	20	3.0	15	1.40	40	20.0	0.49	0.15	0.06	4.5	0.29	17.7
	Plantation	26	3.4	23	1.32	40	9.3	0.60	0.15	0.14	4.6	0.13	7.3
	% Diff.	32	14	51	-6	-1	-54	22	0	156	2	-56	-59
Thom (SWC-2)	Default	12	3.5	30	1.20	50	20.0	0.51	0.06	0.13	4.7	0.40	19.0
	Plantation	16	4.9	34	1.09	53	4.9	0.59	0.14	0.14	4.7	0.16	7.9
	% Diff.	36	41	15	-10	7	-75	16	144	5	-1	-59	-58
Perch Lake (SWC-1)	Default	12	4.5	35	1.20	60	20.0	0.34	0.29	0.07	5.0	0.24	6.1
	Plantation	15	4.9	30	1.11	52	6.4	0.58	0.12	0.14	4.7	0.17	7.3
	% Diff.	21	9	-14	-8	-13	-68	70	-58	89	-6	-28	21
Bridgewater (SWC-2)	Default	15	5.0	20	0.90	55	15.0	0.50	0.14	0.06	5.0	0.25	10.3
	Plantation	14	5.2	35	1.13	48	4.4	0.44	0.20	0.17	4.8	0.18	6.7
	% Diff.	-7	5	73	25	-12	-70	-13	46	193	-4	-29	-35
Diligence (SWC-2)	Literature	30	2.0	20	1.50	35	15.0	0.43	0.20	0.07	4.5	0.28	21.2
	Plantation	34	4.3	13	1.30	35	6.3	0.40	0.27	0.23	4.5	0.13	9.2
	% Diff.	12	113	-33	-13	0	-58	-8	37	221	-1	-54	-56

*SWC = assigned Soil Weatherability Class

Table 2.4. NBM-NS output for sampled plantations – Harvesting Only losses.

Plantation	Soil Series	Species	Projected MMAI (m ³ ha ⁻¹ yr ⁻¹)	NBM Output - Default Data			NBM Output - Plantation Data		
				SusMAI (m ³ ha ⁻¹ yr ⁻¹)	MAI Diff. (m ³ ha ⁻¹ yr ⁻¹)	Limiting Nutrients	SusMAI (m ³ ha ⁻¹ yr ⁻¹)	MAI Diff. (m ³ ha ⁻¹ yr ⁻¹)	Limiting Nutrients
1	Bridgewater	bS	7.8	6.4	-1.3	Ca/K	5.0	-2.8	Ca
2	Bridgewater	wS	7.3	6.0	-1.3	Ca/K	4.6	-2.7	Ca
3	Cobequid	rS	7.9	9.2	1.3	N	9.2	1.3	N
4	Cobequid	wS	5.5	6.0	0.5	Ca	5.4	-0.1	Ca
5	Cobequid	wS	5.6	6.1	0.5	Ca	5.5	-0.1	Ca
6	Cobequid	bS	5.7	6.6	0.9	Ca	6.0	0.3	Ca
7	Halifax	wS	7.3	3.0	-4.3	Ca/K	2.7	-4.6	Ca/K
8	Halifax	wS	7.3	3.0	-4.3	Ca/K	2.6	-4.7	Ca/K
9	Halifax	bS	7.8	3.2	-4.6	Ca/K	2.8	-4.9	Ca/K
10	Halifax	wS	7.3	3.0	-4.3	Ca/K	2.7	-4.7	Ca/K
11	Thom	wS	7.3	7.7	0.4	N	6.9	-0.4	Ca
12	Millbrook	bS	7.8	8.0	0.3	N	8.0	0.3	N
13	Millbrook	bS	7.8	8.2	0.4	N	8.2	0.4	N
14	Millbrook	wS	7.3	7.7	0.3	N	7.7	0.3	N
15	Thom	wS	5.6	7.8	2.2	N	6.9	1.4	Ca
16	Millbrook	wS	7.3	7.7	0.4	N	7.7	0.4	N
17	Perch Lake	NS	8.8	2.8	-5.9	Ca/N/K	4.6	-4.2	Ca/N
18	Perch Lake	wS	7.1	2.9	-4.3	Ca/K	4.6	-2.5	Ca
19	Perch Lake	wS	5.6	2.9	-2.7	Ca	4.6	-1.0	N/Ca
20	Queens	NS	10.4	7.7	-2.7	N/Ca	7.7	-2.7	N
21	Queens	wS	7.3	7.8	0.5	N	7.8	0.5	N
22	Queens	wS	7.1	8.1	0.9	N	8.1	0.9	N
23	Queens	bS	7.6	8.5	0.8	N	8.5	0.8	N
24	Diligence	wS	7.3	5.8	-1.5	Ca	7.7	0.4	N
25	Thom	rS	8.0	8.3	0.3	N	8.3	0.3	N

* bS = black spruce, wS = white spruce, rS = red spruce, NS = Norway Spruce.

Table 2.5. NBM-NS output for sampled plantations – Harvesting + Depletion.

Plantation	Soil Series	Species	Projected MMAI (m ³ ha ⁻¹ yr ⁻¹)	NBM Output - Default Data			NBM Output - Plantation Data		
				SusMAI (m ³ ha ⁻¹ yr ⁻¹)	MAI Diff. (m ³ ha ⁻¹ yr ⁻¹)	Limiting Nutrients	SusMAI (m ³ ha ⁻¹ yr ⁻¹)	MAI Diff. (m ³ ha ⁻¹ yr ⁻¹)	Limiting Nutrients
1	Bridgewater	bS	7.8	7.2	-0.5	Ca	7.8	0.1	Ca
2	Bridgewater	wS	7.3	6.8	-0.5	Ca	7.3	0.0	Ca
3	Cobequid	rS	7.9	9.2	1.3	N	9.2	1.3	N
4	Cobequid	wS	5.5	5.8	0.4	Ca	7.5	2.0	N
5	Cobequid	wS	5.6	5.5	-0.1	Ca	8.1	2.5	N
6	Cobequid	bS	5.7	5.9	0.2	Ca	9.0	3.3	N
7	Halifax	wS	7.3	1.4	-5.9	Ca/K	3.0	-4.3	Ca/K
8	Halifax	wS	7.3	1.3	-6.0	Ca/K	2.8	-4.4	Ca/K
9	Halifax	bS	7.8	1.4	-6.3	Ca/K	3.1	-4.6	Ca/K
10	Halifax	wS	7.3	1.4	-6.0	Ca/K	2.9	-4.4	Ca/K
11	Thom	wS	7.3	7.7	0.4	N	7.7	0.4	N
12	Millbrook	bS	7.8	8.0	0.3	N	8.0	0.3	N
13	Millbrook	bS	7.8	8.2	0.4	N	8.2	0.4	N
14	Millbrook	wS	7.3	7.7	0.3	N	7.7	0.3	N
15	Thom	wS	5.6	7.8	2.2	N	7.8	2.2	N
16	Millbrook	wS	7.3	7.7	0.4	N	7.7	0.4	N
17	Perch Lake	NS	8.8	1.3	-7.5	Ca/N/K	5.6	-3.2	Ca/N
18	Perch Lake	wS	7.1	1.5	-5.6	Ca/K	5.9	-1.3	Ca
19	Perch Lake	wS	5.6	1.8	-3.8	Ca/K	6.1	0.5	Ca
20	Queens	NS	10.4	7.7	-2.7	N	7.7	-2.7	N
21	Queens	wS	7.3	7.8	0.5	N	7.8	0.5	N
22	Queens	wS	7.1	8.1	0.9	N	8.1	0.9	N
23	Queens	bS	7.6	8.5	0.8	N	8.5	0.8	N
24	Diligence	wS	7.3	5.5	-1.8	Ca	7.7	0.4	N
25	Thom	rS	8.0	8.3	0.3	N	8.3	0.3	N

* bS = black spruce, wS = white spruce, rS = red spruce, NS = Norway Spruce.

Table 2.6. Number of plantations with predicted SusMAI less than projected MMAI by model configuration.

NBM-NS Configuration	Plantations with Predicted SusMAI < Projected MMAI
Default soil data with Harvest Only losses	11 (44%)
Plantation soil data with Harvest Only losses	13 (52%)
Default soil data with Harvest + Depletion	12 (48%)
Plantation soil data with Harvest + Depletion	7 (28%)

Table 2.7. NBM-NS output for Plantation 19 showing changes in SusMAI for each change in soil parameter. Predicted SusMAI with all parameters adjusted is also listed for reference purposes.

Parameter	Soil Data			SusMAI (m³ ha⁻¹ yr⁻¹) Harvest Only			SusMAI (m³ ha⁻¹ yr⁻¹) Harvest + Depletion		
	Default	Plantation	% Diff.	Default	Plantation	% Diff.	Default	Plantation	% Diff.
Depth (cm)	60	52	-13	2.9	2.6	-10	1.8	1.2	-33
% Clay	12	15	25	2.9	3.4	17	1.8	2.8	54
% CF	35	30	-14	2.9	3	6	1.8	2.1	19
Db (g cm⁻³)	1.2	1.11	-7	2.9	2.7	-6	1.8	1.5	-18
% OM	4.5	4.9	9	2.9	2.9	0	1.8	1.8	-1
% BS	20	6.4	-68	2.9	2.9	0	1.8	3.6	103
Ca Fraction	0.34	0.58	71	2.9	4.4	53	1.8	2.5	42
Mg Fraction	0.29	0.12	-58	2.9	2.9	0	1.8	1.8	0
K Fraction	0.07	0.14	89	2.9	2.9	0	1.8	1.8	0
All Parameters Adjusted:				2.9	4.6	60	1.8	6.1	243

Table 2.8. NBM-NS calculated soil weathering inputs based on the Clay Content method using measured case study soil data.

Soil Series	Weather-ability Class	Calculated Total Base Cation Input (eq ha⁻¹ yr⁻¹)	Estimated Ca Input (eq ha⁻¹ yr⁻¹)	Estimated Mg Input (eq ha⁻¹ yr⁻¹)	Estimated K Input (eq ha⁻¹ yr⁻¹)
Halifax	1	196	105	28	23
Queens	2	967	515	256	103
Cobequid	2	480	259	73	71
Millbrook	2	836	503	129	118
Thom	2	568	336	78	80
Perch Lake	1	359	207	43	50
Bridgewater	2	494	216	100	85
Diligence	2	961	384	258	219

Appendix 2.1. Chronological order of Nova Scotia Soil Survey Reports.

Year	Author	Title	Number*	Scale
1943	Harlow and Whiteside	Soil Survey of the Annapolis Valley Fruit Growing Area	DA Publication 752	1:63,360
1945	Whiteside et al.	Soil Survey of Cumberland County, Nova Scotia	NSSS Report No. 2	1:126,720
1948	Wicklund and Smith	Soil Survey of Colchester County, Nova Scotia	NSSS Report No. 3	1:126,720
1950	Cann and Wicklund	Soil Survey of Pictou County, Nova Scotia	NSSS Report No. 4	1:126,720
1954	Cann et al.	Soil Survey of Hants County, Nova Scotia	NSSS Report No. 5	1:126,720
1954	Cann and Hilchey	Soil Survey of Antigonish County, Nova Scotia	NSSS Report No. 6	1:126,720
1958	Cann and Hilchey	Soil Survey of Lunenburg County, Nova Scotia	NSSS Report No. 7	1:63,360
1959	Cann and Hilchey	Soil Survey of Queens County, Nova Scotia	NSSS Report No. 8	1:63,360
1960	Cann et al.	Soil Survey of Yarmouth County, Nova Scotia	NSSS Report No. 9	1:63,360
1961	MacDougall et al.	Soil Survey of Shelburne County, Nova Scotia	NSSS Report No. 10	1:63,360
1962	Hilchey et al.	Soil Survey of Digby County, Nova Scotia	NSSS Report No. 11	1:63,360
1963	Cann et al.	Soil Survey of Cape Breton Island, Nova Scotia	NSSS Report No. 12	1:100,000
1963	MacDougall et al.	Soil Survey of Halifax County, Nova Scotia	NSSS Report No. 13	1:63,360
1964	Hilchey et al.	Soil Survey of Guysborough County, Nova Scotia	NSSS Report No. 14	1:63,360
1965	Cann et al.	Soil Survey of Kings County, Nova Scotia	NSSS Report No. 15	1:63,360
1969	MacDougall et al.	Soil Survey of Annapolis County, Nova Scotia	NSSS Report No. 16	1:63,360
1973	Nowland and MacDougall	Soils of Cumberland County, Nova Scotia	NSSS Report No. 17	1:63,360
1987	Langille	Soils of the Kentville Research Station and Sheffield Farm	NSSS Report No. 21	1:5,000
1988	Holmstrom	Soils of the Cambridge Station Area of Nova Scotia	NSSS Report No. 25	1:20,000
1989	Holmstrom and Thompson	Soils of the Annapolis Valley Area of Nova Scotia	NSSS Report No. 22	1:20,000
1989	Webb et al.	Soils of the Cobequid Shore Area of Nova Scotia	NSSS Report No. 23	1:20,000
1989	Patterson and Thompson	Soils of the Northumberland Shore Area of Nova Scotia	NSSS Report No. 24	1:20,000
1990	Webb	Soils of Pictou County, Nova Scotia	NSSS Report No. 18	1:50,000
1991	Webb et al.	Soils of Colchester County, Nova Scotia	NSSS Report No. 19	1:50,000
1993	Langille et al.	Supplement to Soils of the Annapolis Valley Area of Nova Scotia	NSSS Report No. 26	1:20,000
1995	Webb and Langille	Soils of the Nappan Research Farm, Nova Scotia	NSSS Report No. 20	1:5,000

Notes: Surveys from 1943-1973 were published by Canada Department of Agriculture
 Surveys from 1987-1995 were published by Agriculture Canada
 Canada Department of Agriculture (DA) Publication 752 is also recognized as Nova Scotia Soil Survey (NSSS) Report No. 1.

Appendix 2.2. Bolewood density (Gonzalez 1990), stem to compartment biomass conversion coefficients A and B (Noseworthy 2011), and biomass nutrient concentrations (Pardo et al. 2005) for plantation and common tree species in Nova Scotia.

Species	Bolewood Density (g cm ⁻¹)	Biomass Compartment	A	B	N (mg kg ⁻¹)	Ca (mg kg ⁻¹)	Mg (mg kg ⁻¹)	K (mg kg ⁻¹)
White/Norway spruce	0.393	Bolewood	0.8174	0.0249	654	943	101	343
		Bark	0.2641	-0.2504	3,560	12,949	666	2,418
		Branches	0.6443	-0.4703	3,750	5,851	514	2,503
		Foliage	3.6451	-1.0856	10,526	10,532	919	5,247
Black spruce	0.445	Bolewood	0.8172	0.0248	630	874	138	342
		Bark	0.2621	-0.2470	2,400	9,966	555	1,542
		Branches	0.4762	-0.4060	2,592	3,996	430	1,352
		Foliage	2.8232	-1.0756	8,372	7,045	893	4,238
Red spruce	0.425	Bolewood	0.8458	0.0172	640	690	96	220
		Bark	0.1881	-0.1734	2,773	6,685	445	1,635
		Branches	0.0043	1.0108	2,738	3,381	442	1,826
		Foliage	0.0564	0.1571	10,187	4,084	970	5,446
Balsam fir	0.367	Bolewood	0.8257	0.0080	918	823	204	921
		Bark	0.1778	-0.0466	4,616	7,394	636	2,566
		Branches	0.1082	0.1456	3,919	3,812	505	2,569
		Foliage	0.8350	-0.7255	12,746	7,497	806	4,222
White pine	0.365	Bolewood	0.8426	0.0085	780	516	101	324
		Bark	0.1623	-0.0586	3,544	4,223	613	1,473
		Branches	0.0473	0.3387	4,088	3,034	573	1,946
		Foliage	0.2400	-0.3249	12,779	2,827	1,154	4,469
Sugar maple	0.702	Bolewood	0.7962	0.0328	976	1,301	198	691
		Bark	0.3820	-0.3560	5,114	22,280	600	3,119
		Branches	0.1998	0.0940	3,365	6,313	390	2,101
		Foliage	0.2379	-0.5871	19,486	9,337	1,537	7,551
Red maple	0.586	Bolewood	0.8281	0.0226	885	1,121	204	803
		Bark	0.2377	-0.2329	4,332	13,016	468	1,985
		Branches	0.1429	0.1624	3,092	4,655	421	1,704
		Foliage	0.4206	-0.8024	16,958	7,638	2,041	6,827
Yellow birch	0.649	Bolewood	0.9169	-0.0090	1,026	701	155	433
		Bark	0.0911	0.0816	5,672	10,283	423	1,243
		Branches	0.1447	0.2385	4,600	4,130	363	1,130
		Foliage	0.5310	-0.7686	23,490	9,624	2,558	10,241
White birch	0.588	Bolewood	0.8234	0.0127	924	775	185	514
		Bark	0.1875	-0.0846	3,639	6,846	413	1,201
		Branches	0.1875	0.0633	3,913	4,413	533	1,594
		Foliage	0.7582	-0.8548	19,165	7,222	2,247	8,645
Trembling aspen	0.424	Bolewood	0.7881	0.0136	1,298	2,239	343	1,119
		Bark	0.2188	-0.0665	4,497	12,038	1,053	2,631
		Branches	0.1042	0.0600	5,046	9,736	1,156	2,767
		Foliage	0.3400	-0.8310	21,136	10,599	2,082	7,813

CHAPTER 3

FOREST FLOOR CHEMISTRY AND MINERAL SOIL ION EXPOSURE AFTER SURFACE APPLICATION OF ALKALINE-TREATED BIOSOLIDS UNDER TWO WHITE SRUCE (*Picea glauca*) PLANTATIONS IN NOVA SCOTIA, CANADA

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ABSTRACT

Two field trials were established to evaluate the use of alkaline-treated biosolids (ATB) to offset current or predicted Ca deficits in Nova Scotia forest soils under juvenile white spruce (*Picea glauca*) plantations. At the rates applied (7.5 and 15 t ha⁻¹ wet weight), ATB treatments led to significant increases in total and available Ca within the forest floor and surface mineral soil, significant increases in forest floor pH, significant or near-significant decreases in exchangeable forest floor Al³⁺ concentrations, and negligible leaching of metals (Cu, Cd, Pb, Zn) for the 2-year duration of study. Near-surface PO₄³⁻-P availability was also slightly enhanced after an initial delay period. However, despite relatively high K concentrations in the ATB product used, there were no significant increases in forest floor K concentrations, suggesting a relatively rapid

release and movement of K^+ to deeper soil layers compared to Ca^{2+} . There were also no significant increases in forest floor Mg concentrations, nor in total and available N. Results suggest that ATB could be a good source of Ca in Ca-limited sites, but nutrient imbalances may be a problem on sites where K and Mg depletion has also occurred or where N is also limiting.

KEYWORDS

Alkaline-Treated Biosolids; Spruce Plantations; Forest Floor Chemistry; Ion Exposure; Base Cations

INTRODUCTION

Northeastern US and eastern Canadian forests have been affected by decades of acid deposition resulting in increased acidity, base cation depletion, and increased Al availability in many affected soils (Watmough and Ouimet 2005; Lawrence et al. 2012). This has contributed to an overall reduction in potential site productivity in many areas, and an increased susceptibility to further environmental stresses (Schaberg et al. 2001; Duarte et al. 2013). Although acid deposition levels have decreased since adoption of the 1990 *Clean Air Amendments* (US), and 1991 *Air Quality Accord* (US and Canada) (CCME 2013), the overall recovery of forests from decreased acid deposition has not progressed as quickly as hoped (e.g., Houle et al. 2006; Warby et al. 2009; Lawrence et al. 2012). Indeed, some projections suggest it could take several decades for many sites to recover naturally, due in part to (i) reduced acid neutralizing capacity in impacted soils, (ii) desorption of SO_4^{2-} that can continue to promote base cation depletion, and (iii) potential leaching impacts related to ongoing NO_3^- deposition (Driscoll et al. 2001). In addition to acid deposition, timber harvesting can also contribute to a decline in base cation

nutrients (especially Ca) through periodic removal of stem wood and bark (e.g., Freedman, et al. 1986; Federer et al. 1989; Adams et al. 2000; Huntington 2005). Sustainable management in northeastern forests must therefore consider the balance between nutrient outputs via acid leaching and periodic harvesting, and nutrient inputs from soil weathering, atmospheric deposition, and (potentially) application of soil amendments (see Chapter 2).

Despite the well documented impacts of acid deposition (e.g., Driscoll et al., 2003; Watmough and Ouimet, 2005, Fenn et al., 2006; Lawrence et al., 2012; and Duarte et al., 2013) there has been little use of soil amendments to mitigate the effects of increased acidity and base cation depletion in northeastern forest soils (Moore et al. 2015). In contrast, lime has been used in some European countries since the 1980s to mitigate the impacts of acid deposition (e.g., Tomlinson 1990; Nilsson et al. 2001), with wood ash also being used to offset both acid deposition and harvesting impacts (e.g., Levin and Eriksson 2010). In addition to lime and wood ash, alkaline-treated biosolids (ATB) are also considered a liming amendment and nutrient supplement, but their use in forests has been minimal to date (Banaitis et al. 2009).

Initial use of soil amendments in northeastern forests would seem most appropriate in areas under intensive management (such as plantations) where the goal is to increase fibre yields over time through a combination of silviculture treatments and shorter rotation lengths. For example, typical productivity in spruce plantations in Nova Scotia, Canada, is estimated to be about $6 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ which equates to a doubling of predicted harvest volume at peak mean annual increment (approx. $300 \text{ m}^3 \text{ ha}^{-1}$) compared to extensively managed stands (NSDNR 2011). However, based on outputs from a new steady-state nutrient budget model described by Keys et al. (2016), many plantation sites in Nova Scotia may not be able to continuously meet

desired yield targets over time without the use of soil amendments, with Ca identified as a main limiting nutrient (see Chapter 2).

To evaluate the potential for ATB to offset current or predicted Ca deficits in Nova Scotia forest soils, two field trials were established to measure impacts of surface applied ATB on white spruce (*Picea glauca*) plantation soils and vegetation. To our knowledge, this is the first time ATB has been applied to conifer plantations in northeastern North America to assess its potential role in plantation nutrient management. This chapter describes field study design and discusses ATB treatment effects on plantation forest floor chemistry and surface soil ion exposure. Effects on mineral soil chemistry, leachate chemistry, and vegetation are reported in Chapters 4 and 5.

METHODS

An ATB product manufactured by N-Viro Systems Canada and trademarked as Halifax Soil Amendment™ was used in this study. The product is categorized as a Class A biosolid under Nova Scotia Department of Environment regulations (NSE 2010) and is sold commercially under an approved fertilizer label. The patented N-Viro process is known as Advanced Alkaline Stabilization with Subsequent Accelerated Drying. In this process, an alkaline admixture (e.g., cement kiln dust) is added to dewatered biosolids, mixed, heated, and dried. This process eliminates almost all pathogens found in the untreated sludge (USEPA 2000) and produces an agriculture-grade liming material and fertilizer.

Two 10-15-year old upland white spruce plantations were selected for independent field trials in central Nova Scotia, Canada (Figure 3.1). Sites were chosen to be broadly representative of two dominant soil types in the province. Site 1 was moderately well to imperfectly drained

and underlain by a shaly loam soil derived from slate till. Site 2 was well to rapidly drained and underlain by a gravelly/cobbly sandy loam soil derived from granitic till. Both sites had originally supported shade-tolerant hardwood or mixed wood stands that were converted to spruce plantations after harvest.

Treatments compared at each site were a one-time surface application of 7.5 and 15 t ha⁻¹ ATB (wet weight), referred to as low (L) and high (H) rates respectively, along with an untreated control (C). Treatments were applied to Site 1 in September 2012, and to Site 2 in June, 2013, with post-treatment measurements at both sites taken until November, 2014. Target application rates were based on typical ATB product values for Ca (20%) and moisture content (33%) (N-Viro Systems Canada, pers. comm.) which corresponded to Ca applications of about 1,000 and 2,000 kg ha⁻¹. These rates were in the same range as other Ca amendment studies conducted in the past (e.g., Matzner et al. 1985; Long et al. 1997; Juice et al. 2006), as well as a more recent ATB forestry trial in Maine, USA (Banaitis et al. 2009).

Nine 40 m x 40 m plots were established at each location allowing for three replicates of each treatment. Site conditions required different plot layouts at each location, with a Latin square design employed at Site 1 and a linear plot design (with dispersion) employed at Site 2 (Figure 3.2). Plots receiving ATB applications were divided into square quadrats measuring 3.7 m x 3.7 m in low rate plots and 2.6 m x 2.6 m in high rate plots with each quadrat receiving approximately 10 kg of ATB delivered in marked buckets. ATB treatments were applied manually in each quadrat, including under crop trees, taking care to ensure a uniform distribution.

Ca concentrations in ATB samples collected at both trial sites were in the expected 20% range and relatively consistent (coefficient of variation 6-12%) (Table 3.1). Most other elements

also showed consistent concentrations (coefficient of variation less than 10%), but some variability in ATB batches was observed. Based on field assessments and quarterly N-Viro analysis data, ATB moisture content during application at both sites averaged about 38% rather than the typical value of 33%. After adjusting for moisture content and average Ca concentration, estimated Ca application rates were 957 kg ha⁻¹ and 1,914 kg ha⁻¹ (Site 1), and 935 kg ha⁻¹ and 1,870 kg ha⁻¹ (Site 2), for the low and high ATB rates respectively (Table 3.1). Due to analytical problems, cadmium (Cd) and lead (Pb) concentrations and calculated loading rates were determined using average quarterly chemical analysis data obtained from N-Viro Systems Canada (Table 3.1).

A systematic sampling scheme was used to assess soil and vegetation parameters in each plot (Figure 3.3). Since the focus of this chapter is on forest floor chemistry and surface soil ion exposure, only details related to these assessments are presented here. Pre- and post-treatment forest floor samples (combined F and H horizons) were collected in summer and late fall of each year for chemical analysis. Samples consisted of pooled sub-samples from three systematically located points in each plot (Figure 3.3) providing one sample per plot and a final sample size of three for each treatment. Samples were analyzed for pH, total calcium (Ca), total magnesium (Mg), total potassium (K), total phosphorous (P), total nitrogen (N), total sulphur (S), total carbon (C), total iron (Fe), total manganese (Mn), total zinc (Zn), and exchangeable aluminum (Al³⁺). Al was only assessed in pre-treatment and late fall samples.

Ion exposure was assessed using PRSTM-probes (Western Ag Innovations Inc., Saskatoon, SK, Canada). A PRSTM (Plant Root Simulator) probe is an ion exchange membrane encased in plastic that provides a dynamic measure of ion flux to a quantifiable surface area and represents plant nutrient supply rates for the duration of burial (Western Ag 2010). Although

more commonly used in agriculture, PRSTM-probes have also been used in forest soil assessments (e.g., Harrison and Maynard 2013; Johnson et al. 2014). In this study, we refer to the cumulative capture of ions by PRSTM-probes as a measure of ion exposure rather than flux since probes were used year-round and potential exposure levels reflected more than just plant nutrient supply rates.

Eight sets of cation and anion probes were used in each plot with probes inserted at an approximate angle of 15 degrees until tops were flush with surface mineral soil immediately below the forest floor. This resulted in probe membranes being centred around 10 cm from the surface. Probes were installed just before plots were treated, with post-treatment sampling taking place approximately bi-weekly for the first six weeks after ATB application (to avoid overloading the probes), then monthly for most of the remaining study period (when soils were not frozen or snow covered). Burial duration was approximately two months per sampling period for the last year of assessment. Probes were replaced after each sampling period to allow for continuous monitoring (15 sampling events at Site 1 and 10 events at Site 2). All probes were cleaned with de-ionized water shortly after retrieval and shipped to Western Ag Innovations Inc. for extraction and analysis. Probes were extracted in batches of four to give two average cation (Ca^{2+} , Mg^{2+} , K^+ , $\text{NH}_4^+\text{-N}$, Fe^{3+} , Mn^{2+} , Al^{3+} , Cd^{2+} , Cu^{2+} , Pb^{2+} , Zn^{2+}) and anion ($\text{NO}_3^-\text{-N}$, $\text{SO}_4^{2-}\text{-S}$, $\text{PO}_4^{3-}\text{-P}$) measurements per plot per assessment period. This equated to a sample size of six for each ion for each treatment. Randomly selected blank probes handled in the same manner as others (except not deployed in the field) were also submitted for analysis on a periodic basis to check for potential contamination, with no contamination problems found.

Sample Analysis and Statistical Procedures

Sample analysis: Amendment Ca, Mg, K, P, and metals were determined through nitric acid digestion using a Microwave Accelerated Reaction System (MARS) followed by AAS analysis. Amendment pH and electrical conductivity (5:1 water) were measured using an ExStik EC500 meter (Extech Instruments). Amendment and forest floor total C, N, and S were measured using a LECO induction furnace. Forest floor exchangeable Al^{3+} was determined through 1M KCl extraction followed by AAS analysis. Other forest floor elements were determined through dry ashing (400 °C) followed by HCl digestion and colorimetric analysis (P) or AAS analysis (all other elements). Forest floor pH was measured using 0.01 CaCl_2 solution (1:1 ratio) and a benchtop meter. Combined PRSTM-probes were eluted with a counterion solution (0.5 N HCl) for one hour followed by colorimetric analysis (NO_3^- -N, NH_4^+ -N) and ICP analysis (all other elements). (Western Ag 2010).

Data analysis: All response variables were analyzed as repeated measures using SAS PROC MIXED (version 9.3, SAS Institute Inc.). Fixed factors and effects were treatment (C, L, H), time (days since treatment), and treatment-x-time interaction. Two blocking factors were used for the Latin Square design at Site 1, and pre-treatment data were used as covariates in forest floor analyses at both sites. Normality of error terms was assessed for each variable using normal probability plot of residuals, and if violated, power transformations were used. Transformations were mainly needed for cumulative ion exposure data which consistently showed non-normal distributions. This was likely due to natural variation in background soil/site conditions and weather patterns, as well as variation in ATB chemistry and post-treatment responses. Compound symmetry was the best covariance structure found for probe data assessment and was used for all analyses. A combination of compound symmetry and

unstructured covariance structures were used for forest floor data analyses. Significance of model terms and multiple means comparisons for forest floor chemistry were assessed at $\alpha = 0.05$. However, means comparisons for cumulative ion exposure were assessed at $\alpha = 0.01$ to reduce the possibility of Type I error inflation from the large number of treatment combinations and significant interactions found during data analyses. Plots of means were generated using Minitab software version 17 (Minitab Inc.).

Due to differences in ATB chemical composition and application schedules, results from each trial site were treated as independent. Differences and similarities noted between trials are intended to highlight possible effects of ATB chemistry, soil conditions, and/or site variables on treatment responses.

RESULTS

Forest Floor Chemistry

Site 1: Significant treatment-x-time increases in forest floor total Ca ($p = 0.000$), pH ($p = 0.004$), and total Zn ($p = 0.007$) were found at Site 1 (Table 3.2 and Figure 3.5). This led to treatment versus control Ca ratios of 7.98 (high ATB rate) and 5.64 (low ATB rate) by the end of 2014 (Table 3.3). Similar end-of-trial ratios were 0.01 and 0.02 for H^+ , and 2.95 and 1.77 for Zn (Table 3.3). Associated with increased total Ca and pH was a near-significant decrease in exchangeable Al^{3+} (Table 3.2), with end-of-trial concentrations down by 74-86% in treated plots versus controls (Table 3.3). Although substantial, these decreases were not statistically significant due to high control plot variability and small sample size (Figure 3.5). Total forest floor Mn also showed considerable variability in treated plots, tending first to decrease then

increase over time versus the control (Table 3.3, Figure 3.6). All other forest floor element concentrations at Site 1 were not greatly affected by ATB treatments (Tables 3.2 and 3.3).

Site 2: Significant treatment increases in forest floor total Ca ($p = 0.002$) and pH ($p = 0.004$) were found at Site 2, as well as a marginally significant increase in total P ($p = 0.033$) (Table 3.2 and Figure 3.6). This resulted in end-of-trial treatment versus control Ca ratios of 6.84 (high rate) and 6.28 (low rate), H^+ ratios of 0.01 (high rate) and 0.02 (low rate), and P ratios of 1.16 (high rate) and 1.15 (low rate) (Table 3.3). In contrast, exchangeable Al^{3+} dropped significantly at Site 2 ($p = 0.004$, Table 3.2) resulting in a 92-93% decrease under both treatment levels by the end of 2014 (Table 3.3, Figure 3.5). Unlike Site 1, there was no significant increase in total Zn at Site 2, but there was a near-significant treatment-x-time response in total Mn ($p = 0.074$) (Table 3.2), with concentrations clearly decreasing, then increasing, over time (Figure 3.5). Total Mg also showed a near-significant treatment-x-time response ($p = 0.066$) with concentration ratios gradually increasing over time (Tables 3.2 and 3.3). All other forest floor element concentrations at Site 2 were not greatly affected by ATB treatments (Tables 3.2 and 3.3).

Ion Exposure

Site 1: Due to site conditions, Fe and Mn probe data from one control plot at Site 1 were removed prior to analyses. Site 1 was moderately well to imperfectly drained which led to occasional ponding in some micro-depressions after heavy rains and snow melt. Random probe placement coincided with these intermittently ponded areas in one plot which led to increased capture of mobile Fe^{2+} and Mn^{2+} under anaerobic conditions that was unrelated to ATB treatments.

Significant responses in cumulative exposure were found for all ions in treated plots at Site 1 except for NO_3^- -N, Cd^{2+} , and Pb^{2+} , with highly significant responses ($p < .001$) found for Ca^{2+} , K^+ , PO_4^{3-} -P, NH_4^+ -N, SO_4^{2-} -S, Al^{3+} , Fe^{3+} , and Mn^{2+} (Table 3.4). Exposure trends differed by ion (Figures 3.7 and 3.8, Table 3.5) with: (i) Ca^{2+} showing early and continuing increases in capture rate related to ATB rate, (ii) Mg^{2+} showing early increases in capture rate related to ATB rate followed by gradually increasing capture rate in high rate plots only, (iii) K^+ showing early increases in capture rate related to ATB rate followed by steady accumulation for all plots, (iv) PO_4^{3-} -P showing a delayed, but then sharp and continuous increase in capture rate over time related to ATB rate, (v) NH_4^+ -N showing increased accumulation in high rate plots followed by a gradual decrease in capture rate over time, but no response in low rate plots, (vi) SO_4^{2-} -S showing substantial early accumulation related to ATB rate followed by relatively steady capture rates for all plots, (vii) Al^{3+} showing an early increase in capture rate (especially in high rate plots) with a gradual decrease in capture rate over time, (viii) Fe^{3+} showing early increased capture in low rate plots, but decreased capture in high rate plots, followed by relatively steady capture rates for all plots, (ix) Mn^{2+} showing substantial accumulation early on at both ATB rates, but with more response in low rate plots, followed by decreasing capture rates over time, (x) Cu^{2+} showing an early pulse accumulation in high rate plots with gradual decline in capture rate, but no response in low rate plots, and (xi) Zn^{2+} showing a delayed and gradual decrease in capture rate for all treated plots over time, with a faster decrease in high rate plots.

Site 2: Significant responses in cumulative exposure were found for all ions in treated plots at Site 2 except for NO_3^- -N, Cd^{2+} , and Zn^{2+} , with highly significant responses ($p < .001$) found for Ca^{2+} , NH_4^+ -N, SO_4^{2-} -S, Al^{3+} , and Cu^{2+} (Table 3.4). As with Site 1, exposure trends differed by ion (Figures 3.9 and 3.10, Table 3.6) with: (i) Ca^{2+} showing early and continuing

increases in capture rate related to ATB rate, (ii) Mg^{2+} and K^+ showing early increases in capture rate related to ATB rate (especially for K^+) followed by relatively steady accumulation for all plots, (iii) PO_4^{3-} -P showing a delayed, but then continuous increase in capture rate over time related to ATB rate, (iv) NH_4^+ -N showing an early increase in capture rate for high rate plots followed by gradual decline, but no response in low rate plots, (v) SO_4^{2-} -S showing substantial early accumulation related to ATB rate followed by relatively steady capture rates for all plots, (vi) Al^{3+} showing an early increase in capture rate related to ATB rate with more gradual increases over time, (vii) Fe^{3+} showing early and almost equal increased accumulation at both ATB rates, followed by relatively steady accumulation by all plots, (viii) Mn^{2+} showing early increases in capture rate related to ATB rate with gradual decreases over time (more pronounced in high rate plots), (ix) Cu^{2+} showing step-like increases in capture rate related to ATB rate, and (x) Pb^{2+} showing an early pulse increase in high rate plots followed by minor increases in capture rate over time.

DISCUSSION

Forest Floor Chemistry

Treatment related responses in forest floor Ca, pH, and Al at both sites were in line with those reported for a young hardwood ATB trial in Maine, USA (which included similar and higher Ca application rates, Banaitis et al. 2009) and a Norway spruce (*Picea abies*) liming trial in southern Germany (Kreutzer 1995). In both these cases, amendment Ca loading and exchange led to higher Ca^{2+} concentrations, higher pH, and lower Al^{3+} concentrations in forest floor horizons after treatment. In addition, forest floor pH continued to increase in the Maine ATB trial for about two years before starting to stabilize or decline (Banaitis et al. 2009). In this study,

forest floor pH also started to show signs of leveling off after about two years at Site 1, but only with the low rate treatment (Figure 3.4). A more detailed comparison of pH responses between these trials is not possible, however, due to a lack of calcium carbonate equivalence data for the ATB batches used in this study.

Increases in forest floor Ca and pH at Site 2 did not show the time interaction found at Site 1 due to differences in treatment and assessment schedules between sites. Site 1 was treated in September, 2012 with a sampling range of 58 to 805 days. Site 2 was treated in June of 2013 with a sampling range of 147 to 518 days. The shorter assessment period at Site 2, together with later initial sampling and high variability in early measurements (Figure 3.6), probably masked the time interaction shown at Site 1.

Forest floor Ca in treated plots increased each year for the duration of study at both sites (Figures 3.4 and 3.6) indicating ongoing supply from amendment breakdown. No data are available on dissolution rates of surface applied ATB in forests, but in the Norway spruce liming trial described by Kreuzer (1995), it took six years for a 4 t ha⁻¹ application of dolomitic lime to completely dissolve. Although ATB used in this study had Ca contents less than that found in dolomitic limestone, application rates were higher, suggesting there will be a continuous release of Ca for several years at these sites, especially in high rate plots. It should be noted, however, that Ca values shown in Figure 3.4 are total (not exchangeable) measures, and even though care was taken to remove surface litter and vegetation prior to sampling of F and H horizons, it is possible that some of the Ca increases found may be from small particles of unreacted ATB in addition to increased exchangeable Ca²⁺ (Meiwes et al. 2002; Banaitis et al. 2009). This could also be the case for forest floor Zn, but the significant increases in total Zn found only at Site 1 were likely related to higher Zn content in the Site 1 ATB batch used (almost 3x that found in the

Site 2 batch) (Table 3.1). Increasing pH would also likely lead to greater Zn^{2+} retention over time in these organic horizons (Sims 1986).

In a similar fashion, variation in forest floor total Mn (Figure 3.5) can be attributed to initial Ca loading causing loss of exchangeable Mn^{2+} (early decreasing trend), followed by ATB breakdown and organic matter retention at higher pH values causing Mn accumulation (Sims 1986). This is also supported by trends in Mn^{2+} ion capture in surface mineral soils over time (discussed below). Marginally significant increases in forest floor total P at Site 2 were probably related to relatively high P inputs in the amendment used (Table 3.1) compared to background levels. Although not significant, total forest floor P also increased in treated plots at Site 1 (Table 3.3).

Ion Exposure

PRSTM-probes captured large quantities of ions at both sites over the duration of study, especially Ca^{2+} , K^+ , and SO_4^{2-} -S (Figures 3.7 and 3.9). This reflected the chemical make-up of the ATB batches used (Table 3.1), as well as the need for electro-neutrality in aqueous solutions, with SO_4^{2-} -S acting as the dominant companion anion to Ca^{2+} and K^+ . Significant Mg^{2+} exposure was also detected, but Mg^{2+} was the least responsive of the base cations due to lower ATB concentrations (Table 3.1) and greater affinity for exchange sites compared to K^+ . Due to variation in microtopography and related drainage patterns, use of the probes over winter, and the slightly off-vertical angle of insertion, it is likely that probes were subject to a range of diffusion and mass flow fluxes over the trial period. However, a comparison of maximum single sample measures recorded during this study with estimated single probe capacities suggest that

probe membranes were not saturated between sampling events and that most (if not all) Ca^{2+} , K^+ , SO_4^{2-} -S, and Mg^{2+} fluxes were captured (Table 3.7).

Increasing forest floor total Ca and Ca^{2+} capture in treated plots over time suggest a more gradual and continuous release of Ca^{2+} compared to K^+ which was more mobile after treatment. Indeed, the lack of any significant difference in forest floor total K between treated and control plots (Table 3.2) suggests an almost immediate loss of K^+ after treatment which would also have been promoted by the swamping of exchange sites with higher valence Ca^{2+} . These trends are generally supported by Banaitis et al. (2009) who reported significantly higher exchangeable Ca^{2+} concentrations, but lower exchangeable K^+ concentrations, in the forest floor horizons of similarly treated plots in their ATB trial. However, as indicated by significant increases found in white spruce foliage K concentrations in treated plots (Chapter 5), at least some of this available K^+ was taken up by vegetation before potentially leaving the rooting zone. In contrast, there were no significant increases in Mg concentrations found in either the forest floor (Table 3.2) or crop tree foliage (Chapter 5). This reflected low amendment input (Table 3.1), but also the competitive effects of increased Ca^{2+} and K^+ concentrations since plant uptake of Mg^{2+} is sensitive to cation competition (Ende and Evers 1997).

ATB treatments also led to delayed increases in available PO_4^{3-} -P in surface mineral soils at both sites (Figures 3.7 and 3.9). This was likely due to higher pH values and loss of Al^{3+} promoting increased P availability, as well as movement of colloidal P from forest floor horizons (Bol et al. 2016). With increased pH and Ca^{2+} concentrations, P immobilization can shift from insoluble Al/Fe phosphates to insoluble Ca phosphates, but based on forest floor data, pH did not rise to the near-neutral levels generally associated with enhanced Ca phosphate formation (Haynes 1982). When PRSTM-probes are in the ground for longer time periods, capture of

nutrient ions represent surplus availabilities rather than total supplies (Western Ag 2010), so the delay found in PO_4^{3-} -P capture may have been due to increased plant and microbial uptake soon after ATB applications. This was supported by post-treatment sampling in 2012 which showed significant increases in white spruce foliage P from high rate plots at Site 1 (Chapter 5).

With respect to N, data from both sites showed minimal ATB treatment effects on total and available N. Compared to Ca, total N in the ATB amendment used was low (Table 3.1) and previous studies, albeit under agricultural conditions, suggest only a small portion of this mineralizes and becomes available after application (Price et al. 2015). High ATB pH values (mean 9.7 to 10.9, Table 3.1) would also have promoted some volatilization loss of inorganic N during processing and immediately after surface application (Jones et al. 2013). There were some initial increases in NH_4^+ -N with high ATB rates (especially at Site 2), but the main trend was a general decrease in NH_4^+ -N exposure in high rate plots over time (Figures 3.7 and 3.9, Tables 3.5 and 3.6). This was presumably a result of increased nitrification following increases in forest floor pH. As with P, total inorganic N exposure in treated plots would be impacted by plant and microbial uptake, but data from tree and ground vegetation foliage sampling showed little increase in N concentrations at either site for the duration of study (Chapter 5), consistent with an overall lack of ATB treatment impact on available N. Although Kreutzer (1995) reported significant forest floor mineralization and NO_3^- production after liming a Norway spruce site, Matzner et al. (1985) and Ingerslev (1997) did not, concluding that surface applied lime had little impact on forest floor C and N stores. Little change to forest floor C and N in this study (Tables 3.2 and 3.3) are in line with these latter results. Variability in site response was also noted by Reid and Watmough (2014) in their review of lime and wood ash treatment effects in forests, suggesting amendment impacts on forest floor mineralization are related to more than just pH.

Patterns in Al^{3+} exposure at both sites (Figures 3.8 and 3.10) were in keeping with those found in forest floor horizons (Figure 3.5). The greatest Al^{3+} exposure at Site 1 was associated with high rate plots shortly after treatment, whereas both treatment rates led to significant increases in Al^{3+} exposure at Site 2. In a similar fashion, Mn^{2+} exposure increased immediately after treatment at both sites, but started to decline due to increased retention associated with increasing forest floor pH. This pattern was most evident at Site 1 (Figure 3.8) where cumulative exposure in control plots was approaching that of high rate plots by the end of the trial. A similar, though less pronounced, pattern was also evident at Site 2 (Figure 3.10). This same pH influence on metal ion retention can be seen at Site 1 where cumulative Zn^{2+} exposures started to decrease in treated plots after about 400 days, with high rate plots decreasing faster than low rate plots (Figure 3.8).

Trends in Fe^{3+} exposure were less clear than for Mn^{2+} and Zn^{2+} . Cumulative exposures were equally high for all treated plots at Site 2, but with no time interaction (Figure 3.10, Table 3.5). This suggests an initial Fe pulse after treatment followed by relatively steady exposure levels not influenced by changing forest floor pH (Figure 3.10). At Site 1, low rate plots showed increased Fe^{3+} exposure related to controls, but high rate plots showed less (Figure 3.8). Overall exposure levels were also greater at Site 1 compared to Site 2 (about 4x), despite similar Fe concentrations in ATB batches (Table 3.1). These opposing results may have been due, in part, to differences in surface soils between sites. A-horizon thickness was highly variable at Site 1 (ranging from about 0-10 cm), so PRSTM-probes were more likely to have been in contact with Fe-enriched B-horizons than would have been the case at Site 2 where leached Ae horizons were generally thicker. Variable drainage conditions at Site 1 may have also led to increased Fe^{2+} capture even with the removal of obviously biased data.

One of the concerns with land application of biosolids is possible metal contamination of soils and surface waters, although this is less of a risk when using high quality (Class A) biosolids (Lu et al. 2012). With infrequent applications of ATB in a forest setting, the main concern is more likely to be possible contamination of surface or subsurface waters after application rather than soil contamination (e.g., Luo and Christie 2001). Trace or heavy metals are generally less soluble at higher pH values and would therefore be less of a problem with ATB versus regular biosolids (Basta et al. 2005; Gagnon et al. 2013). However, liming has been shown to increase the leaching potential of organically complexed metals (especially Cu and Pb) when forest floor mineralization was stimulated by increased pH (Kreutzer 1995), suggesting metal leaching is still a possibility when using high pH amendments. In this study, there was some Cu^{2+} and Pb^{2+} leaching from the forest floor after treatment (mainly associated with high rate treatments), but overall exposures were low. Cumulative Cu^{2+} exposure in high rate plots was only $3.2 \mu\text{g } 10\text{cm}^{-2}$ after 808 days at Site 1 and less than $1 \mu\text{g } 10\text{cm}^{-2}$ after 533 days at Site 2 (Figures 3.8 and 3.10). For Pb^{2+} , cumulative exposure at Site 1 was $5.5 \mu\text{g } 10\text{cm}^{-2}$ in high rate plots after 808 days, but was not significantly greater than control plot exposure ($3.5 \mu\text{g } 10\text{cm}^{-2}$). At Site 2, cumulative exposure was less than $1 \mu\text{g } 10\text{cm}^{-2}$ in high rate plots after 533 days (Figures 3.10). These low Cu^{2+} and Pb^{2+} exposures reflect low concentrations in the amendments used (Table 3.1) and a lack of forest floor mineralization as discussed above. There was also no treatment related response in Cd^{2+} exposure at either site (Table 3.5).

CONCLUSIONS

Alkaline-treated biosolids (ATB) were surface applied at 7.5 t ha^{-1} (low rate) and 15 t ha^{-1} (high rate) under two 10-15-year old white spruce plantations in Nova Scotia, Canada. At the rates

applied, ATB treatments led to (i) significant increases in total and available Ca within the forest floor and surface mineral soil, (ii) significant increases in forest floor pH, (iii) significant or near-significant declines in exchangeable Al^{3+} concentrations, (iv) relatively little impact on total and available N, and (v) negligible leaching of metals (Cu, Cd, Pb, Zn) for the duration of study. P availability was also slightly enhanced after an initial delay period. However, despite relatively high K concentrations in the ATB product used, there were no significant increases in forest floor K concentration or surface mineral ion exposures after the first year of treatment, suggesting a relatively rapid release and movement of K^+ to deeper soil layers compared to more steady release and movement of Ca^{2+} . There were also no significant increases in forest floor Mg due to low amendment content and displacement by Ca. Furthermore, these trends were consistent between two sites with contrasting soils.

Overall results suggest that ATB could be a good source of Ca in Ca-limited sites, but due to the high concentration of Ca versus other nutrients in ATB, nutrient imbalances may be a problem on sites where N is also limiting or where extensive K and Mg depletion has also occurred. This may be particularly important for Mg if the alkaline product used in ATB production is low in this nutrient, as was the case in this study. It is recommended that forest soil nutrient levels be assessed prior to ATB use so that mitigation of Ca deficiencies does not cause other nutrient management problems. In addition, although ATB treatments in this study did not result in any significant increase in forest floor NO_3^- leaching, this type of response is still possible on other sites (especially N-rich sites), and should be monitored if ATB use on forest sites is considered.

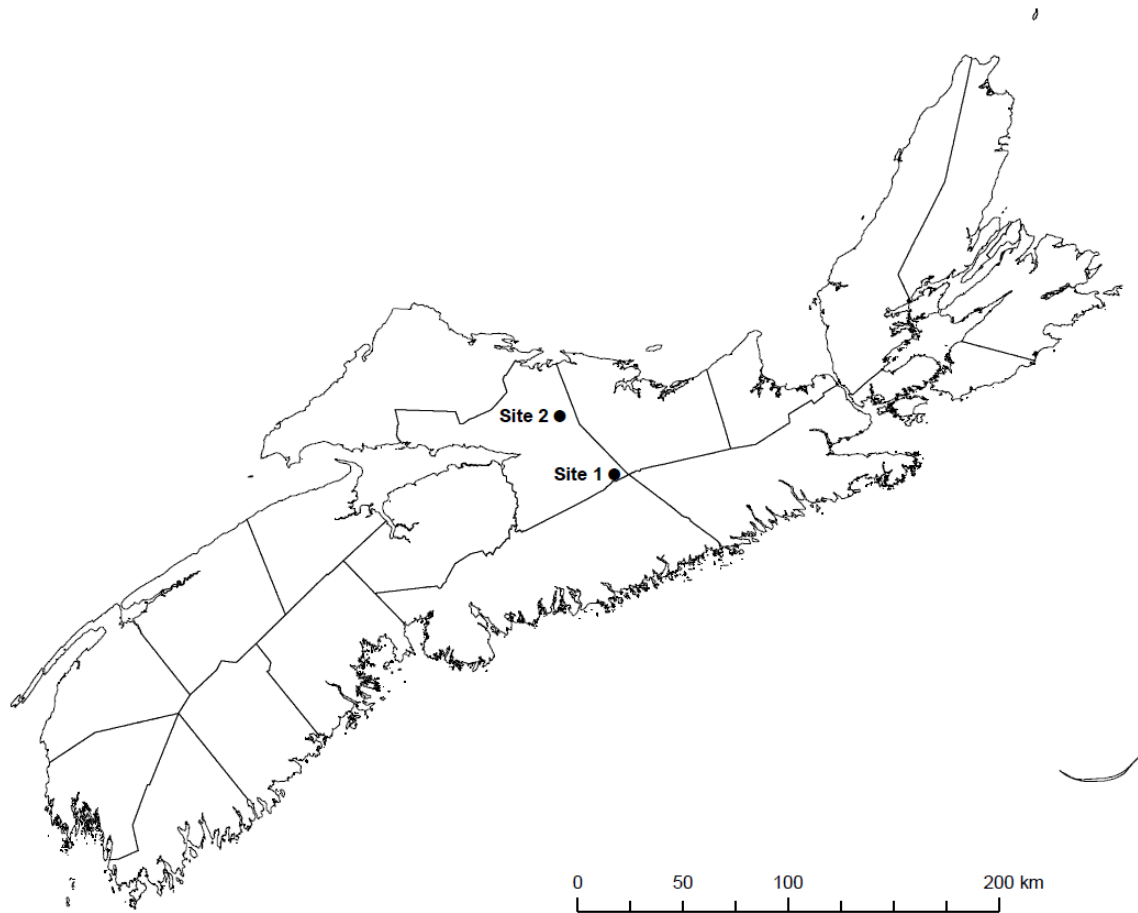


Figure 3.1. Location of ATB field trial sites in central Nova Scotia, Canada.

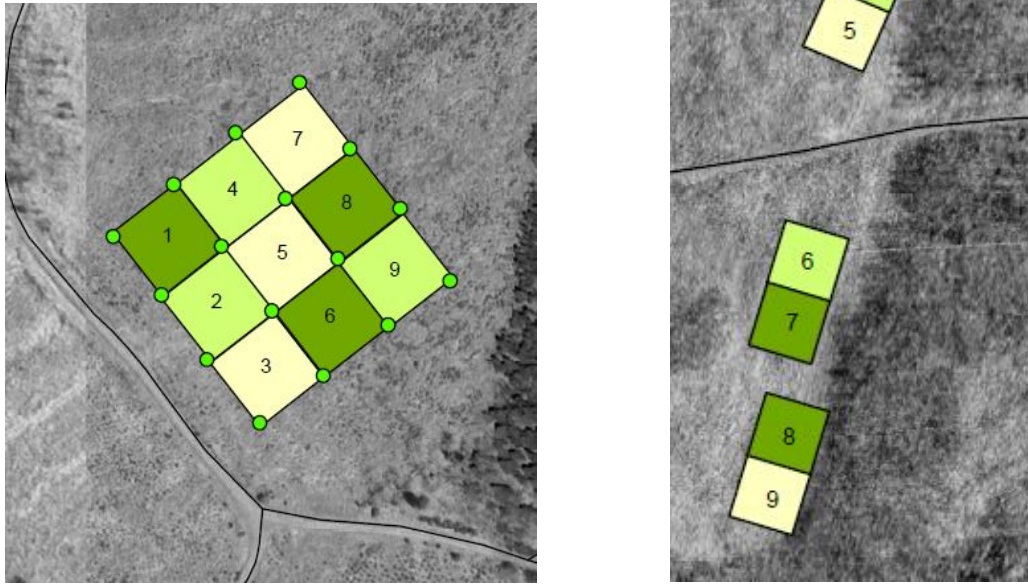


Figure 3.2. Plot layout at Site 1 (left) and Site 2 (right). Pale yellow = control, pale green = low rate treatment (7.5 t ha^{-1}), dark green = high rate treatment (15 t ha^{-1}). The Latin square design at Site 1 followed a northeast ground vegetation gradient from the road and a northwest ingrowth gradient from the treed area to the east. Plots at Site 2 were not as close to the stand boundary as suggested by the image. Treatments were randomly assigned within the constraints of each layout design. To ensure adequate treatment dispersion at Site 2, random patterns were generated until each treatment type was found on both sides of the road.

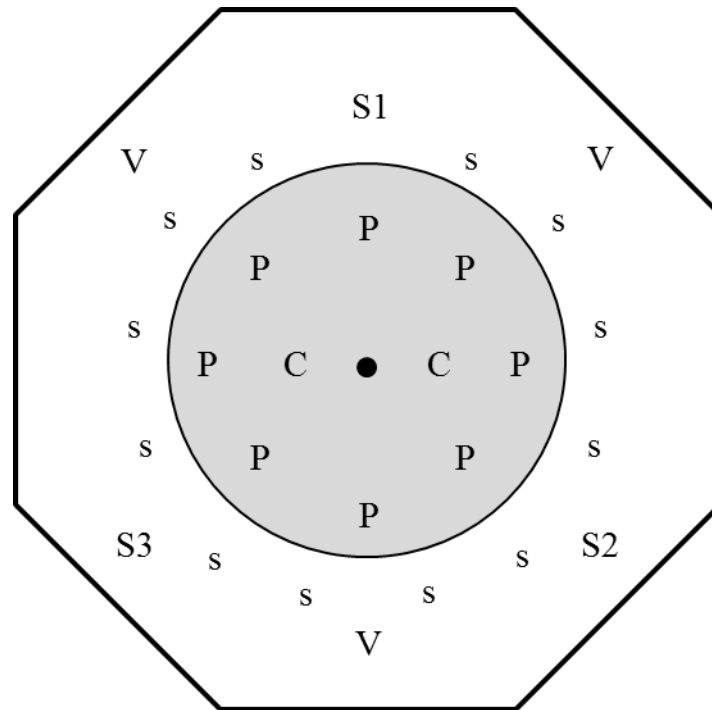


Figure 3.3. Schematic of a single field plot with systematic sample points based on a random initial bearing for soil pit 1 (S1). Octagon: ATB treatment area within 40 m x 40 m plot. Grey circle: tree growth assessment area (10.3 m radius). C = porous cup solution samplers (4 m from centre). P = paired PRSTM-probes (8 m from centre). S1-S3 = pre-treatment soil pit sampling sites (13 m from centre). s = post-treatment soil sampling sites (12 m from centre). V = ground vegetation monitoring sub-plots (13-14 m from centre).

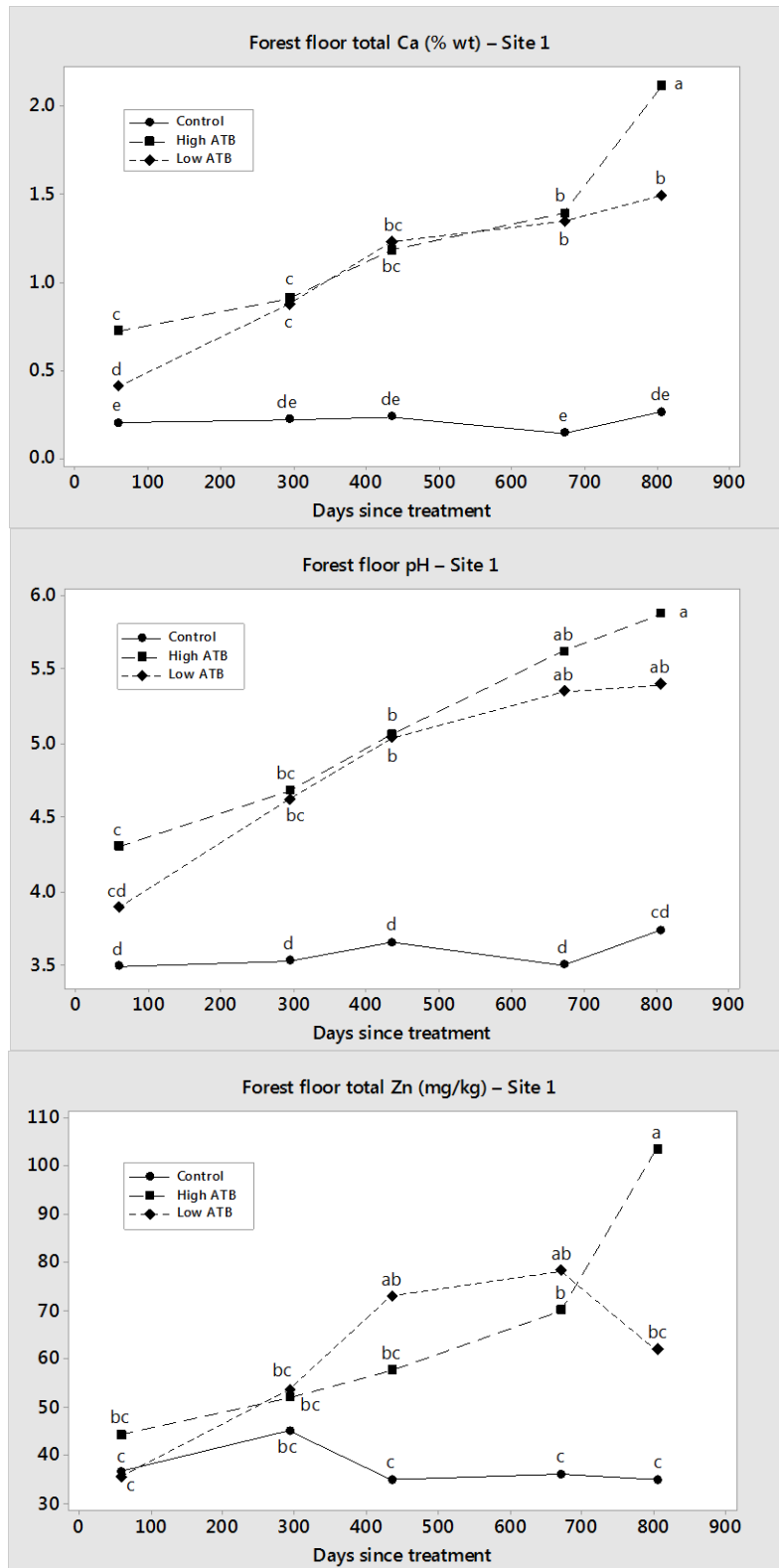


Figure 3.4. Mean forest floor total Ca, pH, and total Zn by treatment and time for Site 1. Values with different letters are statistically different at $p = 0.05$.

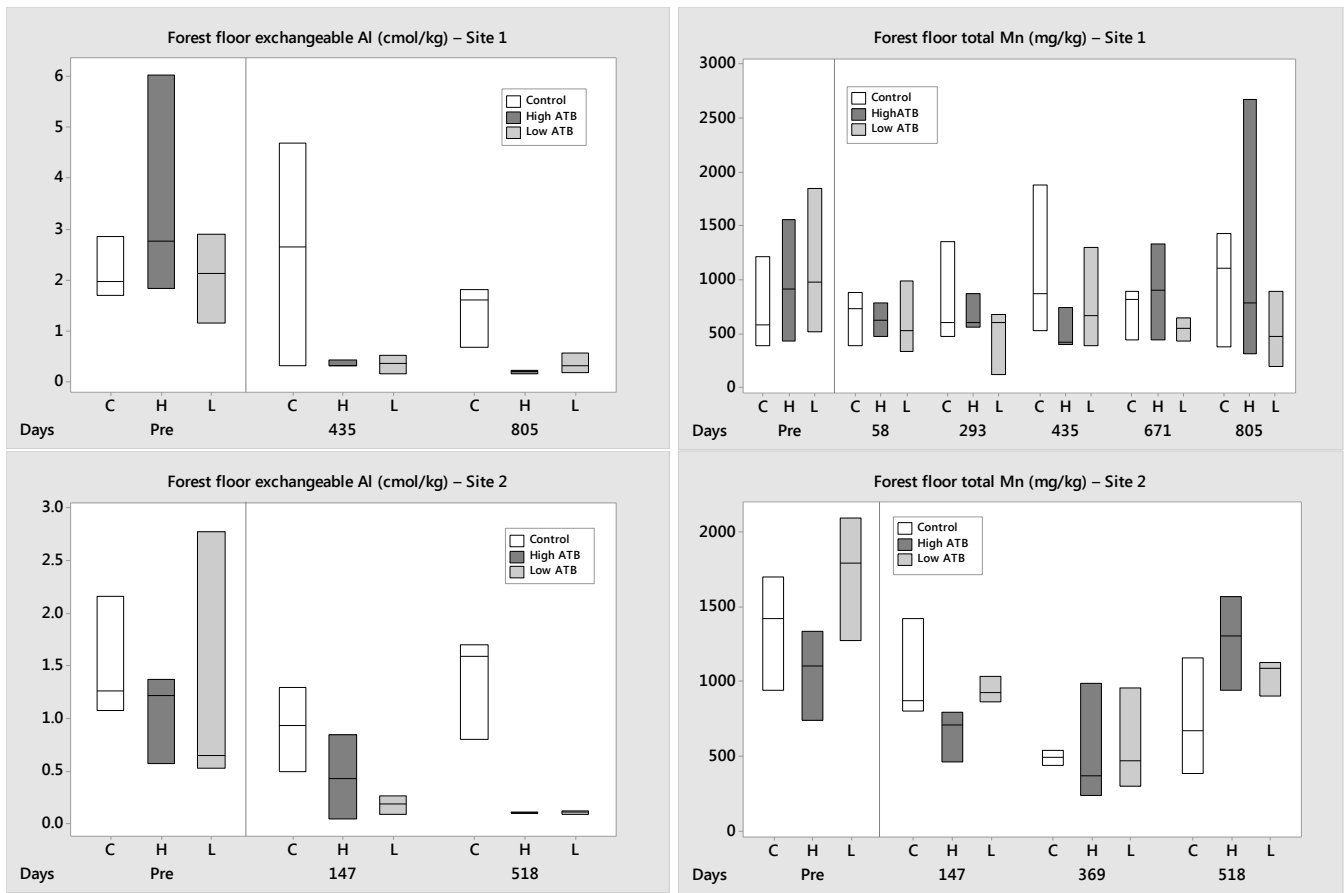


Figure 3.5. Pre-treatment (left of line) and post-treatment (right of line) forest floor exchangeable Al^{3+} and total Mn concentrations at Site 1 and Site 2 by treatment and days since treatment.

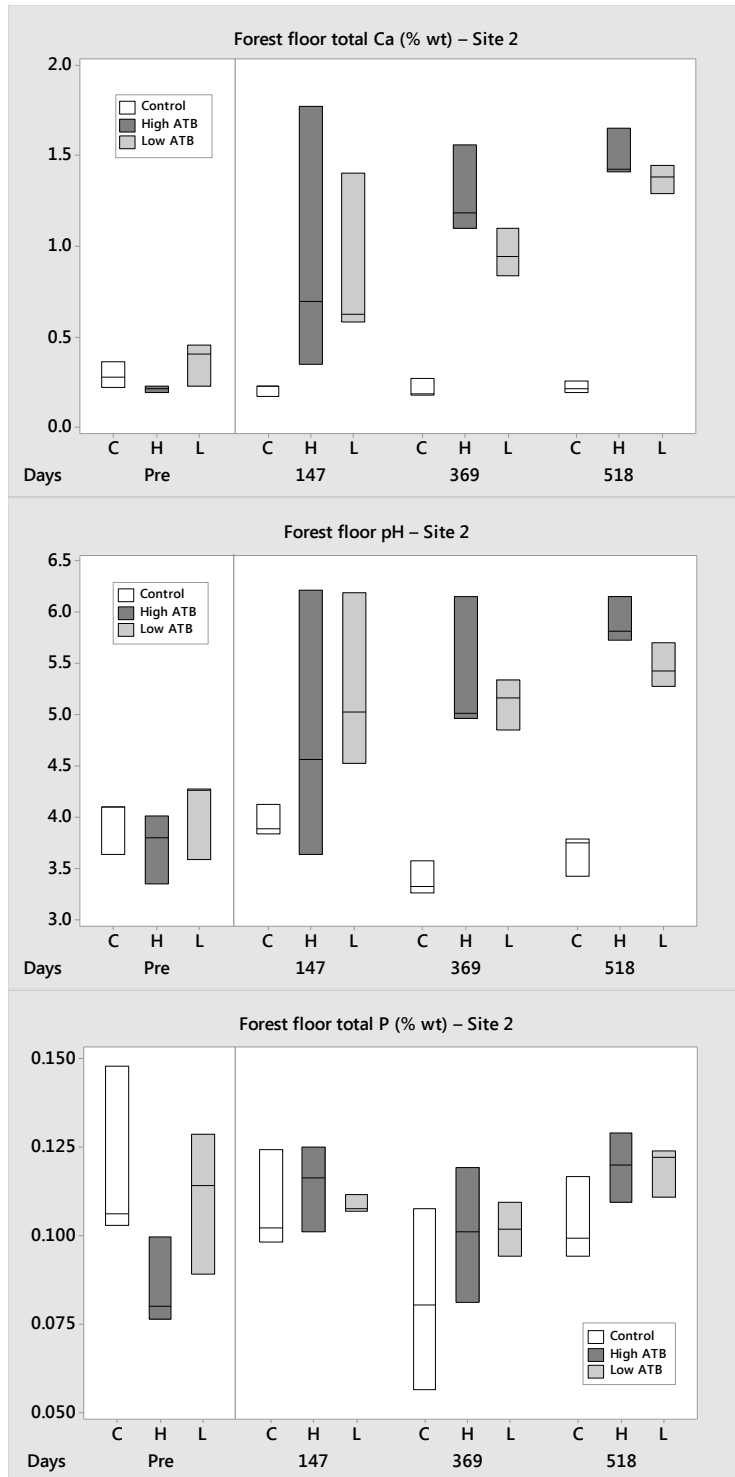


Figure 3.6. Pre-treatment (left of line) and post-treatment (right of line) forest floor total Ca, pH, and total P at Site 2 by treatment and days since treatment.

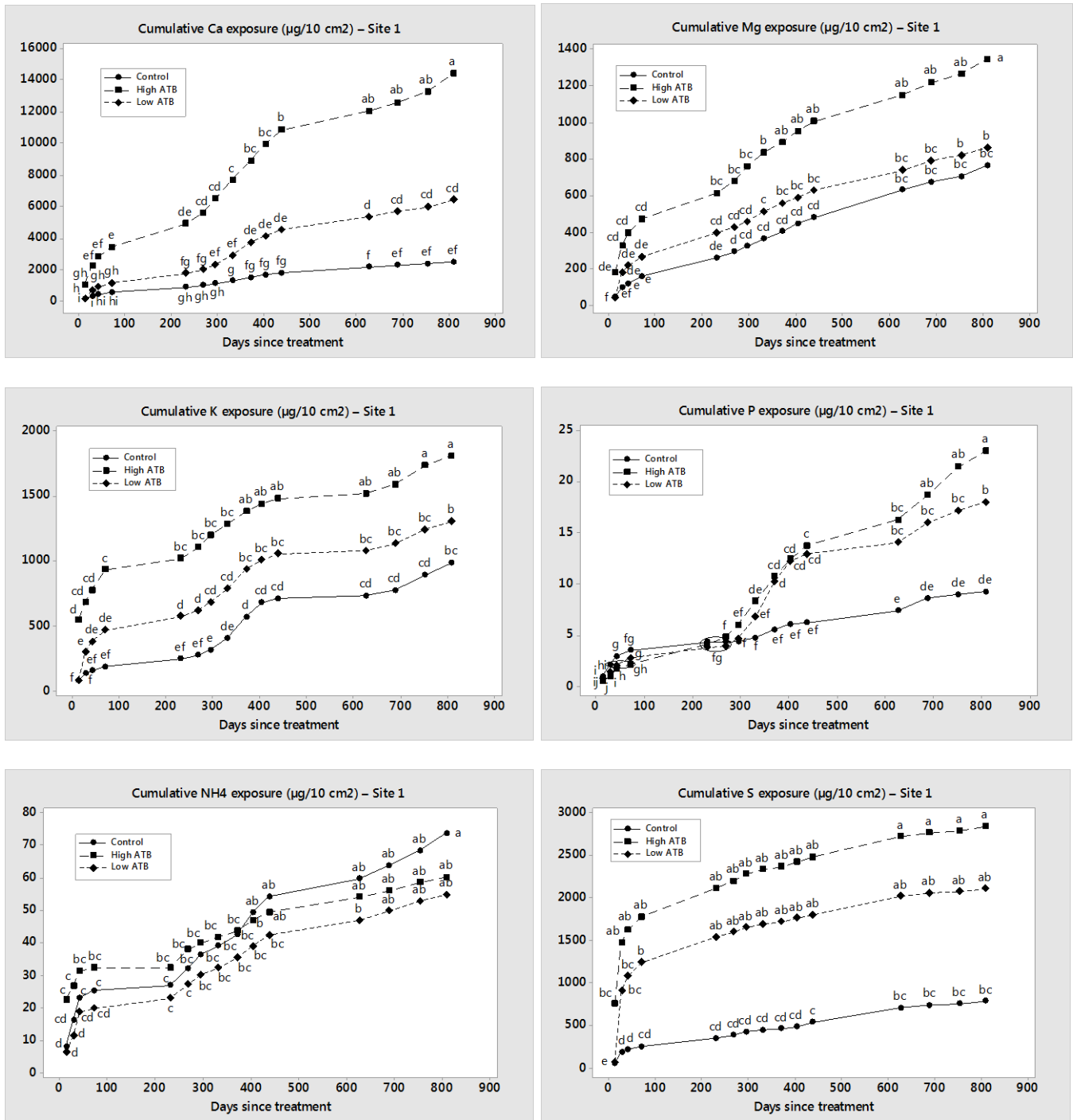


Figure 3.7. Cumulative mean Ca^{2+} , Mg^{2+} , K^+ , $\text{PO}_4^{3-}\text{-P}$, $\text{NH}_4^+\text{-N}$, and $\text{SO}_4^{2-}\text{-S}$ ion exposures by treatment and time for Site 1. Values with different letters in each graph are statistically different at $p = 0.01$.

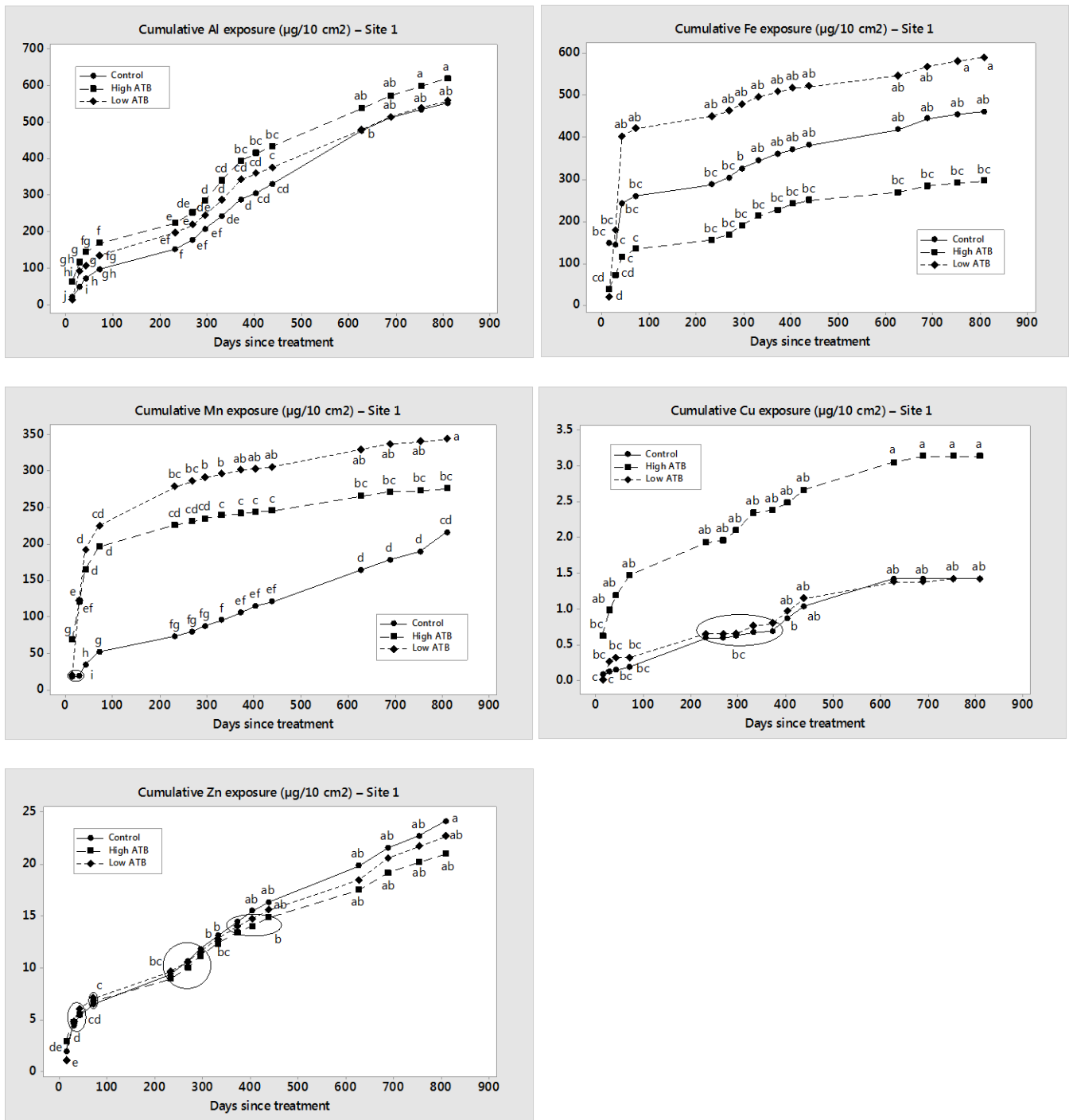


Figure 3.8. Cumulative mean Al^{3+} , Fe^{3+} , Mn^{2+} , Cu^{2+} , and Zn^{2+} ion exposures by treatment and time for Site 1. Values with different letters in each graph are statistically different at $p = 0.01$.

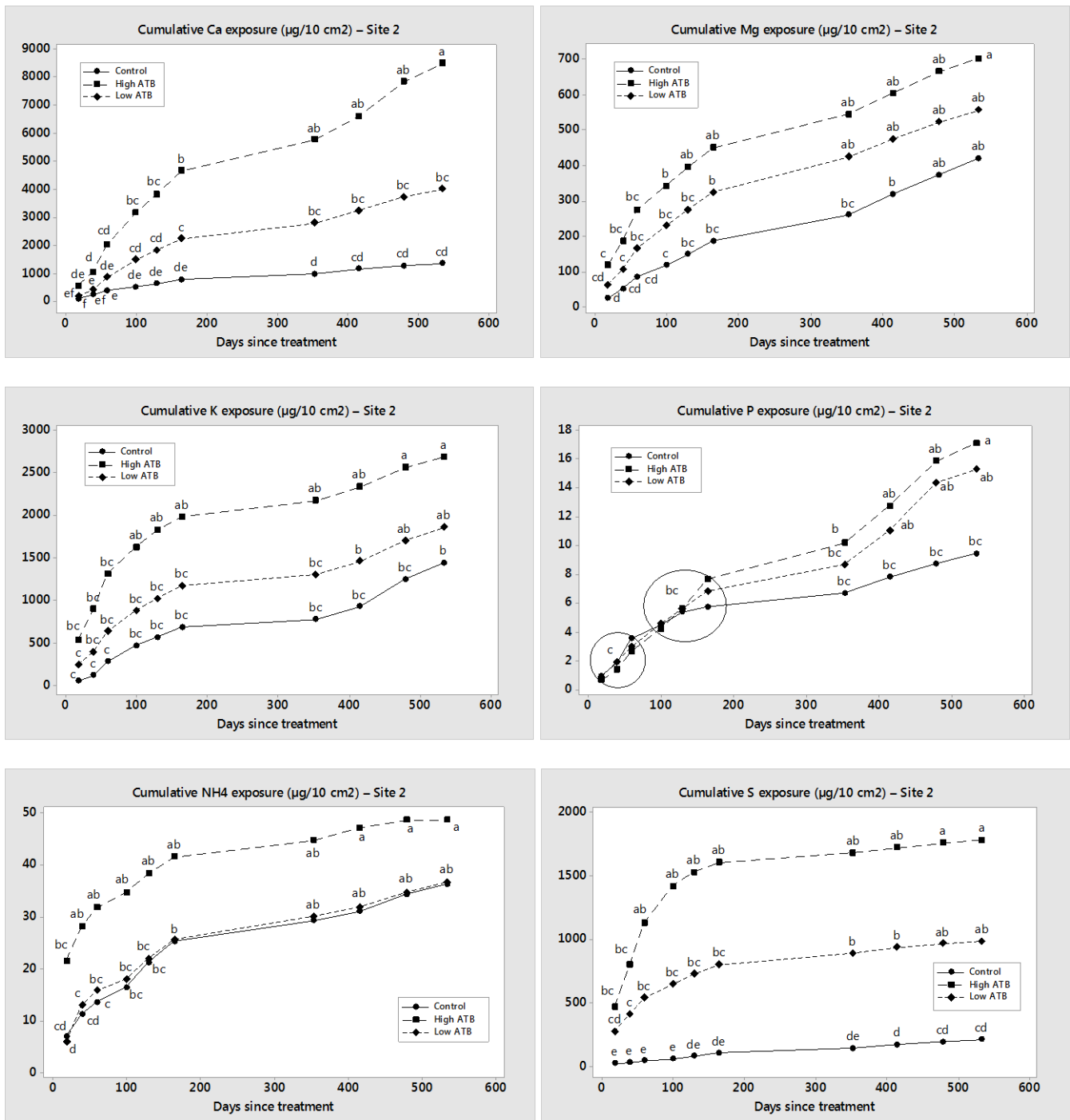


Figure 3.9. Cumulative mean Ca^{2+} , Mg^{2+} , K^+ , $\text{PO}_4^{3-}\text{-P}$, $\text{NH}_4^+\text{-N}$, and $\text{SO}_4^{2-}\text{-S}$ exposures by treatment and time for Site 2. Values with different letters in each graph are statistically different at $p = 0.01$.

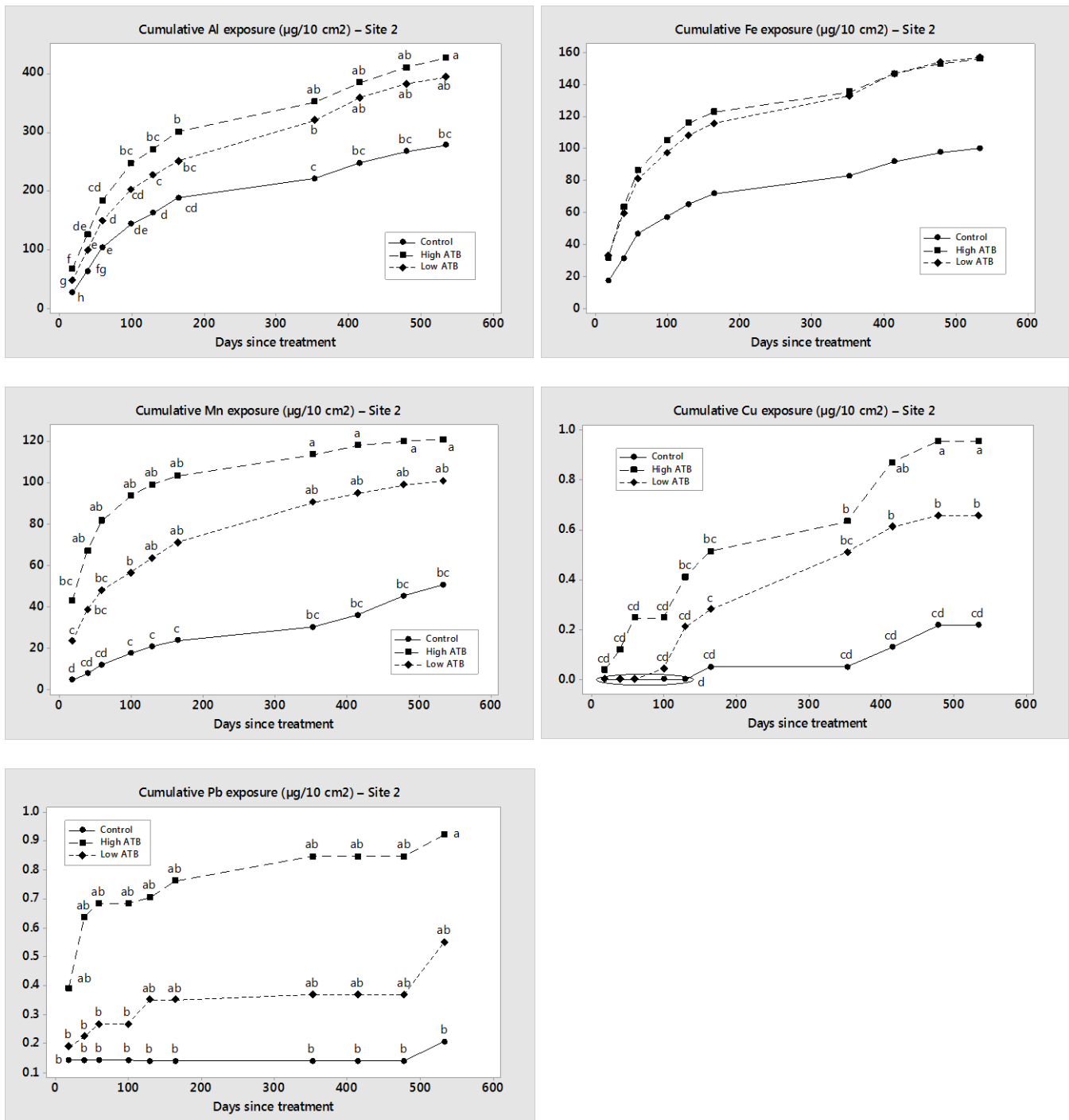


Figure 3.10. Cumulative mean Al^{3+} , Fe^{3+} , Mn^{2+} , Cu^{2+} , and Pb^{2+} ion exposures by treatment and time for Site 2. Values with different letters in each graph are statistically different at $p = 0.01$.

Table 3.1. Summary of ATB chemistry and estimated application rates for Site 1 and Site 2. All values are from on-site samples except Cd and Pb values which came from quarterly analyses conducted by N-Viro Systems Canada for 2012 and 2013.

Site 1															
Statistic	pH	EC	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn
(n=6)		(mS) (% wt) (mg kg ⁻¹)							
Mean	10.9	4.65	20.6	0.41	1.72	0.85	0.13	0.52	7,571	218	18,358	107	<0.1	79	494
Stdev	0.5	0.25	1.16	0.03	0.04	0.17	0.06	0.04	416	15	1,135	5	nd	nd	58
% CV	5	5	6	7	2	20	44	7	5	7	6	5	nd	nd	12
Estimated element application (kg ha⁻¹)															
	Trmt	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn	
	High	1,914	38	160	79	12	49	70	2.0	171	1.00	<0.001	0.74	4.6	
	Low	957	19	80	39	6	24	35	1.0	85	0.50	<0.001	0.37	2.3	

Site 2															
Statistic	pH	EC	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn
(n=4)		(mS) (% wt) (mg kg ⁻¹)							
Mean	9.7	5.47	20.1	0.34	1.65	0.87	0.08	0.58	7,550	226	20,765	101	<0.1	117	180
Stdev	1.3	1.18	2.38	0.04	0.32	0.04	0.01	0.05	767	38	2,240	9	nd	nd	14
% CV	13	22	12	10	19	5	13	9	10	17	11	8	nd	nd	8
Estimated element application (kg ha⁻¹)															
	Trmt	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn	
	High	1,870	32	153	81	7	54	70	2.1	193	0.94	<0.001	1.09	1.7	
	Low	935	16	77	41	4	27	35	1.1	97	0.47	<0.001	0.54	0.8	

Table 3.2. Repeated measures analysis results for forest floor chemistry at Site 1 and Site 2. Significant treatment and treatment-x-time interactions have been shaded (alpha = 0.05).

p values - Site 1												
Effect	Ca	Mg	K	P	N	S	C	Al	Fe	Mn	Zn	pH
Block 1	0.406	0.924	0.713	0.257	0.009	0.291	0.884	0.354	0.708	0.452	0.694	0.425
Block 2	0.581	0.819	0.128	0.113	0.014	0.297	0.510	0.511	0.792	0.941	0.664	0.488
Pre-trmt Value	0.835	0.694	0.142	0.218	0.025	0.742	0.627	0.257	0.655	0.714	0.683	0.584
Treatment	0.079	0.789	0.393	0.134	0.093	0.175	0.712	0.101	0.595	0.750	0.345	0.137
Days	<.0001	0.493	0.001	0.000	<.0001	<.0001	<.0001	0.045	0.014	0.770	0.004	<.0001
Trmt x Days	0.000	0.496	0.532	0.946	0.574	0.420	0.592	0.105	0.719	0.498	0.007	0.004

p values - Site 2												
Effect	Ca	Mg	K	P	N	S	C	Al	Fe	Mn	Zn	pH
Pre-trmt Value	0.812	0.007	0.170	0.025	0.279	0.876	0.171	0.172	0.528	0.703	0.061	0.422
Treatment	0.002	0.773	0.424	0.033	0.696	0.786	0.471	0.003	0.921	0.908	0.533	0.004
Days	0.041	0.011	0.077	0.000	0.578	0.067	0.112	0.605	0.004	0.001	0.537	0.025
Trmt x Days	0.385	0.066	0.510	0.300	0.385	0.649	0.742	0.194	0.161	0.074	0.863	0.475

Table 3.3. Ratios of mean forest floor chemistry values in ATB treated plots versus control plots at Site 1 and Site 2 by year and days since treatment (pre = pre-treatment, H = high ATB rate, L = low ATB rate). pH was converted to H⁺ for ratio calculations.

Site 1														
Year	Days	Trmt	Ca	Mg	K	P	N	S	C	Al ³⁺	Fe	Mn	Zn	H ⁺
2012	Pre	H	1.04	0.75	1.21	1.09	1.14	1.10	1.07	1.63	0.75	1.33	0.82	0.76
		L	1.25	0.92	0.95	0.92	1.07	1.08	1.11	0.94	0.76	1.54	0.93	1.02
	58	H	3.60	0.81	0.93	1.05	1.05	0.99	1.10	nd	0.78	0.95	1.21	0.16
		L	2.03	0.87	0.95	0.98	0.96	0.94	1.03	nd	0.95	0.93	0.97	0.40
2013	293	H	4.08	1.07	1.20	1.18	1.00	0.86	1.02	nd	0.93	0.84	1.15	0.07
		L	3.92	0.80	0.97	1.10	1.01	0.90	1.08	nd	0.77	0.57	1.19	0.08
	435	H	5.03	1.01	1.10	1.16	0.90	0.80	0.93	0.14	1.13	0.48	1.65	0.04
		L	5.22	1.03	1.07	1.06	0.90	0.80	0.97	0.14	0.98	0.71	2.08	0.04
2014	671	H	9.54	1.26	1.13	1.20	0.99	0.91	0.91	nd	1.02	1.24	1.94	0.01
		L	9.21	1.22	1.09	1.15	1.03	0.99	0.98	nd	0.95	0.75	2.17	0.01
	805	H	7.98	1.27	1.52	1.51	0.88	0.77	0.92	0.14	1.21	1.30	2.95	0.01
		L	5.64	0.82	1.15	1.18	1.00	0.98	1.01	0.26	0.86	0.53	1.77	0.02
Site 2														
Year	Days	Trmt	Ca	Mg	K	P	N	S	C	Al ³⁺	Fe	Mn	Zn	H ⁺
2012	Pre	H	0.74	0.47	0.89	0.72	0.94	1.01	0.91	0.70	0.66	0.78	0.55	1.69
		L	1.27	0.51	1.20	0.93	1.04	0.91	1.05	0.88	0.78	1.27	0.99	0.81
2013	147	H	4.48	0.78	1.03	1.06	1.13	1.08	1.18	0.48	0.48	0.64	0.95	0.14
		L	4.16	0.83	1.03	1.01	0.88	0.79	0.91	0.20	0.64	0.91	1.04	0.05
2014	369	H	6.08	0.68	1.17	1.23	1.02	0.94	0.98	nd	0.79	1.09	0.98	0.01
		L	4.56	0.71	1.23	1.25	1.03	0.98	0.91	nd	0.82	1.18	0.89	0.02
	518	H	6.84	0.97	1.10	1.16	0.95	0.85	1.02	0.07	0.89	1.72	0.98	0.01
		L	6.28	0.97	1.03	1.15	1.07	0.93	1.07	0.08	0.83	1.41	1.11	0.02

Table 3.4. Repeated measures analysis results for surface mineral soil ion exposures at Site 1 and Site 2. Significant treatment and treatment-x-time interactions have been shaded (alpha = 0.05). Ion charges have been omitted for ease of presentation.

Site 1														
Effect	Ca	Mg	K	PO ₄ - P	NH ₄ - N	NO ₃ - N	SO ₄ - S	Al	Fe	Mn	Cu	Cd	Pb	Zn
Block 1	0.638	0.174	0.237	0.263	0.857	0.954	0.488	0.026	0.175	0.122	0.570	0.766	0.049	0.115
Block 2	0.144	0.083	0.795	0.099	0.245	0.777	0.200	0.108	0.168	0.195	0.463	0.911	0.062	0.162
Treatment	0.014	0.076	0.031	0.055	0.387	0.913	0.046	0.055	0.223	0.161	0.187	0.738	0.090	0.964
Days	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Trmt x Days	<.0001	0.029	<.0001	<.0001	<.0001	0.831	<.0001	<.0001	<.0001	<.0001	0.025	0.928	0.783	0.028

Site 2														
Effect	Ca	Mg	K	PO ₄ - P	NH ₄ - N	NO ₃ - N	SO ₄ - S	Al	Fe	Mn	Cu	Cd	Pb	Zn
Treatment	0.001	0.072	0.062	0.498	0.040	0.439	0.000	0.005	0.026	0.005	0.001	0.156	0.032	0.384
Days	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Trmt x Days	<.0001	0.024	0.008	0.009	<.0001	0.177	<.0001	<.0001	0.320	0.003	<.0001	0.415	0.032	0.179

Table 3.5. Ratios of mean cumulative ion exposures in ATB treated plots versus control plots at Site 1 by year and days since treatment (H = high ATB rate, L = low ATB rate). Ion charges have been omitted for ease of presentation.

Year	Day	Trmt	Ca	Mg	K	PO4-P	NH4-N	NO3-N	SO4-S	Al	Fe	Mn	Cu	Cd	Pb	Zn
2012	14	H	5.63	3.82	6.86	0.52	2.90	1.35	12.63	2.97	0.26	3.85	8.28	1.40	5.44	1.41
	14	L	0.79	1.05	1.28	0.81	0.76	0.28	1.13	0.69	0.12	1.15	0.00	0.48	7.08	0.55
	29	H	6.77	3.35	5.25	0.47	1.62	0.99	7.77	2.39	0.50	6.37	8.50	1.40	1.60	0.91
	29	L	2.12	1.75	2.27	0.69	0.66	0.55	4.80	1.90	1.26	6.50	2.25	0.63	1.63	0.90
	42	H	6.50	3.34	5.15	0.58	1.33	0.95	7.40	1.98	0.48	4.80	8.16	1.45	1.06	0.85
	42	L	2.07	1.69	2.49	0.70	0.77	0.57	4.92	1.47	1.66	5.58	2.20	0.59	1.09	0.93
	71	H	5.97	3.13	5.06	0.61	1.23	1.52	7.09	1.74	0.52	3.78	7.88	1.33	1.05	0.87
	71	L	2.02	1.60	2.55	0.78	0.74	0.74	4.95	1.38	1.62	4.33	1.70	0.64	1.47	0.90
2013	231	H	5.42	2.50	4.16	0.94	1.17	1.39	6.00	1.47	0.54	3.08	3.25	1.39	1.00	0.82
	231	L	1.96	1.46	2.35	0.87	0.81	1.06	4.37	1.29	1.56	3.78	1.10	0.66	1.11	0.87
	268	H	5.40	2.48	4.01	1.10	1.17	1.26	5.69	1.42	0.55	2.90	3.30	1.20	2.58	0.83
	268	L	1.94	1.39	2.27	0.91	0.82	0.98	4.14	1.23	1.52	3.59	1.10	0.71	3.62	0.86
	295	H	5.67	2.54	3.80	1.37	1.08	1.23	5.39	1.36	0.58	2.69	3.36	1.18	1.39	0.84
	295	L	2.02	1.36	2.19	1.06	0.81	1.00	3.91	1.18	1.47	3.34	1.04	0.70	1.51	0.86
	331	H	5.81	2.51	3.15	1.74	1.06	1.35	5.21	1.40	0.62	2.51	3.49	1.23	1.13	0.86
	331	L	2.19	1.37	1.94	1.42	0.81	1.02	3.77	1.18	1.44	3.11	1.13	0.71	1.17	0.86
	371	H	5.82	2.44	2.42	1.94	1.02	1.37	5.12	1.37	0.63	2.29	3.46	0.92	1.02	0.86
	371	L	2.44	1.35	1.65	1.85	0.81	1.06	3.71	1.19	1.41	2.84	1.15	0.82	1.26	0.87
	403	H	5.85	2.37	2.10	2.04	0.94	1.34	4.98	1.36	0.65	2.12	2.85	0.90	1.00	0.85
	403	L	2.43	1.30	1.47	2.01	0.78	1.04	3.61	1.17	1.39	2.64	1.11	0.81	1.05	0.87
	438	H	5.98	2.34	2.07	2.20	0.91	1.35	4.62	1.31	0.65	2.03	2.58	0.89	5.70	0.86
	438	L	2.51	1.30	1.48	2.07	0.77	1.05	3.35	1.14	1.37	2.53	1.11	0.79	9.70	0.87

Table 3.5. Continued...

Year	Day	Trmt	Ca	Mg	K	PO4- P	NH4- N	NO3- N	SO4- S	Al	Fe	Mn	Cu	Cd	Pb	Zn
2014	626	H	5.51	2.05	2.06	2.20	0.90	1.30	3.85	1.13	0.64	1.62	2.15	0.86	1.37	0.82
	626	L	2.45	1.15	1.47	1.90	0.77	1.15	2.86	1.00	1.30	2.01	0.97	0.78	1.47	0.85
	688	H	5.48	2.06	2.03	2.17	0.88	1.34	3.75	1.12	0.64	1.52	2.21	0.85	1.03	0.83
	688	L	2.48	1.16	1.46	1.85	0.77	1.13	2.79	1.00	1.28	1.89	0.97	0.77	1.05	0.88
	752	H	5.58	2.07	1.93	2.39	0.86	1.34	3.68	1.12	0.64	1.44	2.21	0.90	1.00	0.84
	752	L	2.51	1.15	1.39	1.91	0.76	1.14	2.74	1.01	1.28	1.80	1.00	0.77	1.26	0.88
	808	H	5.77	2.05	1.83	2.48	0.82	1.33	3.61	1.12	0.64	1.28	2.21	0.87	1.00	0.82
	808	L	2.58	1.12	1.32	1.94	0.73	1.12	2.67	1.01	1.28	1.59	1.00	0.77	1.11	0.87

Table 3.6. Ratios of mean cumulative ion exposures in ATB treated plots versus control plots at Site 2 by year and days since treatment (H = high ATB rate, L = low ATB rate, na = not applicable due to zero values for control plots). Ion charges have been omitted for ease of presentation.

Year	Day	Trmt	Ca	Mg	K	PO4-P	NH4-N	NO3-N	SO4-S	Al	Fe	Mn	Cu	Cd	Pb	Zn
2013	18	H	5.33	4.81	9.77	0.70	3.06	3.70	25.14	2.54	1.85	9.25	na	1.50	2.75	2.33
	18	L	2.12	2.89	4.51	0.95	0.85	3.05	15.13	1.80	1.92	5.56	na	0.50	1.33	2.58
	39	H	4.35	3.59	7.35	0.72	2.50	3.02	28.82	2.00	1.99	8.64	na	1.96	4.46	2.22
	39	L	1.90	2.29	3.26	1.02	1.16	2.48	15.81	1.59	1.91	5.60	na	1.03	1.57	2.65
	59	H	5.17	3.22	4.62	0.75	2.34	2.63	26.68	1.78	1.83	6.83	na	1.88	4.80	1.93
	59	L	2.42	2.14	2.25	0.84	1.17	2.39	14.07	1.46	1.75	4.42	na	1.22	1.87	2.18
	99	H	6.05	2.89	3.43	0.94	2.11	2.39	25.93	1.72	1.82	5.34	na	1.89	4.80	1.66
	99	L	3.09	2.12	1.87	1.02	1.09	2.17	12.86	1.42	1.72	3.45	na	1.47	1.87	1.92
	129	H	5.90	2.62	3.21	1.04	1.81	2.37	19.53	1.67	1.76	4.75	na	1.84	5.05	1.54
	129	L	3.09	1.99	1.79	1.05	1.03	2.08	10.10	1.41	1.68	3.23	na	1.50	2.51	1.81
	164	H	5.86	2.39	2.88	1.33	1.65	2.17	15.65	1.60	1.70	4.34	10.20	1.48	5.45	1.44
	164	L	3.03	1.85	1.70	1.19	1.01	1.85	8.44	1.34	1.63	3.14	5.53	1.14	2.51	1.69
2014	352	H	5.80	2.08	2.79	1.52	1.53	1.58	11.96	1.60	1.63	3.73	12.63	1.44	6.06	1.40
	352	L	2.99	1.70	1.67	1.30	1.03	1.43	6.77	1.47	1.62	3.09	10.23	1.14	2.64	1.76
	414	H	5.64	1.88	2.51	1.63	1.52	1.71	10.16	1.56	1.59	3.27	6.77	1.34	6.06	1.32
	414	L	2.93	1.55	1.57	1.41	1.03	1.49	5.86	1.47	1.62	2.74	4.75	1.07	2.64	1.68
	478	H	6.07	1.78	2.05	1.81	1.41	1.76	9.27	1.54	1.56	2.64	4.42	1.36	6.06	1.24
	478	L	3.09	1.48	1.36	1.64	1.01	1.53	5.39	1.45	1.60	2.26	3.05	1.09	2.64	1.57
	533	H	6.19	1.67	1.86	1.81	1.34	1.69	8.53	1.54	1.55	2.37	4.42	1.35	4.48	1.21
	533	L	3.15	1.41	1.29	1.62	1.01	1.46	4.98	1.44	1.59	2.05	3.05	1.10	2.67	1.53

Table 3.7. Maximum single sampling PRS™-probe measurements ($\mu\text{g } 10\text{cm}^{-2}$) found during field trials (both sites) and maximum single ion capacities reported by Western Ag (2010).

Ion	Ca²⁺	Mg²⁺	K⁺	PO₄³⁻-P	NH₄⁺-N	NO₃⁻-N	SO₄²⁻-S	Al³⁺	Fe³⁺	Mn²⁺	Cu²⁺	Zn²⁺
Field Maximum	2021	744	835	6	41	83	1587	377	718	297	3	19
Probe Capacity	4753	2883	9273	4620	3320	2088	4782	4131	8552	8412	9731	10012

CHAPTER 4

MINERAL SOIL AND LEACHATE CHEMISTRY FOLLOWING SURFACE APPLICATION OF ALKALINE-TREATED BIOSOLIDS UNDER TWO WHITE SPRUCE (*Picea glauca*) PLANTATIONS IN NOVA SCOTIA, CANADA

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ABSTRACT

Two field trials were established to evaluate the use of alkaline-treated biosolids (ATB) to offset current or predicted Ca deficits in Nova Scotia forest soils under juvenile white spruce (*Picea glauca*) plantations. Analysis of B- and BC-horizon soils after application of 7.5 and 15 t ha⁻¹ ATB (wet weight) showed increases in total base cation / Al ratios related to application rate at Site 1 (underlain by imperfectly drained loamy soil), but no such increases at Site 2 (underlain by well drained coarse soil). In addition, there were no significant treatment related changes in soil pH, soil microbial biomass, or trace metal concentrations (Cd, Cu, Pb, Zn) at either site. Representative leachate sampling showed significant increases in base cation (Ca²⁺, Mg²⁺, K⁺) and SO₄²⁻ leaching associated with ATB treatments, but no increases in NO₃⁻ leaching. Results

from this study, and a related assessment of forest floor and surface ion flux chemistry, suggest that ATB could be a good source of Ca in Ca-limited sites, but that site and soil conditions need to be considered when developing treatment regimes.

KEYWORDS

Alkaline-Treated Biosolids, Spruce Plantations, Soil Chemistry, Microbial Biomass, Leachate Chemistry, Base Cations

INTRODUCTION

To aid sustainable forest management planning in Nova Scotia, Canada, a geospatial nutrient budget model was developed that can estimate the sustainable mean annual increment (SusMAI) of a given site based on soil, substrate, and atmospheric deposition data (Keys et al. 2016). With this model, site-specific nutrient inputs can be compared to estimated nutrient outputs from harvesting and acid leaching to assess the long-term sustainability of various harvest scenarios. Initial use of this model has shown that calcium (Ca) is a potential limiting nutrient on many sites with slowly weathering soils (Chapter 2) due, in part, to base cation depletion from decades of acid deposition in the region (e.g., Watmough and Ouimet 2005; Lawrence et al. 2012).

Although a range of conditions can be found, the potential for Ca deficits is expected to be greater for areas under plantation management where the primary objective is to increase fibre yields over time through a combination of silviculture treatments and shorter rotation lengths. Predicted deficits may, however, be offset through judicious use of soil amendments such as lime or wood ash (e.g., Tomlinson 1990; Nilsson et al. 2001; Levin and Eriksson 2010). Alkaline-

treated biosolids (ATB) – often used in agriculture – may also be a good liming amendment (e.g., Banaitis et al. 2009). To evaluate the potential for ATB to offset current or predicted Ca deficits in Nova Scotia forest soils, two field trials were established to measure effects of surface applied ATB on white spruce (*Picea glauca*) plantation soils and vegetation.

ATB treatment effects on forest floor and near-surface ion fluxes have already been discussed in Chapter 3. Results showed (i) significant and persistent increases in total and available Ca, (ii) reduced aluminum (Al^{3+}) concentrations, (iii) increased movement of potassium (K^+) and magnesium (Mg^{2+}), (iv) slightly enhanced phosphorous (P) availability, (v) minimal effect on total or available nitrogen (N), and (vi) negligible leaching of trace metals (Cu^{2+} , Cd^{2+} , Pb^{2+} , Zn^{2+}). This chapter discusses the effects of ATB treatments on deeper B- and BC-horizon chemistry, soil microbial biomass, and rooting zone leachate chemistry.

METHODS

A full description of experimental design is found in Chapter 3. A brief summary is presented here for context, along with details related to mineral soil and leachate analyses.

Two 10-15-year old upland white spruce plantations in central Nova Scotia, Canada were selected for independent field trials. Site 1 was moderately well to imperfectly drained and underlain by a shaly loam soil derived from slate till. Site 2 was well to rapidly drained and underlain by a gravelly/cobbly sandy loam soil derived from granitic till. Soils at both sites were field-classified as Humo-Ferric Podzols (SCWG 1998) or Typic Haplorthods (USDA 1999) (Figure 4.1).

An ATB product manufactured by N-Viro Systems Canada and trademarked as Halifax Soil Amendment™ was used in this study. This product is categorized as a Class A biosolid

under Nova Scotia Department of Environment regulations (NSE 2010) and is sold commercially under an approved fertilizer label. Target application rates were 1,000 kg ha⁻¹ Ca (low rate) and 2,000 kg ha⁻¹ Ca (high rate) with default concentrations used for other elements. Actual Ca application rates were determined to be 957 kg ha⁻¹ and 1,914 kg ha⁻¹ (Site 1: low and high rates), and 935 kg ha⁻¹ and 1,870 kg ha⁻¹ (Site 2: low and high rates) (Table 4.1). Site 1 – a Latin square design – was treated in September, 2012. Site 2 – a random plot design – was treated in June, 2013. Post-treatment measurements at both trial sites were taken to November, 2014.

A systematic sampling scheme was developed to assess soil and vegetation parameters at each plot (Figure 4.2). Three large soil pits per plot were excavated prior to treatment to assess soil/site variability, collect bulk density (Db) samples, and to collect pooled samples for chemical and texture analyses (Table 4.2). Pits were located 12 m from plot centre with Pit 1 run on a random bearing and Pits 2 and 3 each offset by 120°. Samples were taken from the upper B-horizon (referred to as upper soil) and BC-horizon at approximately rooting zone depth (referred to as lower soil). Post-treatment soil samples were collected in early summer and late fall each year following treatment and consisted of pooled samples from three systematically located points based off the original random bearing used to locate Pit 1 (Figure 4.2). Post-treatment samples were collected using a soil auger (Site 1) and small excavations (Site 2) with soil colour and texture indicators identified during pre-treatment assessment used to confirm appropriate sampling depths. Soils were analyzed for: pH; exchangeable Ca²⁺, Mg²⁺, K⁺, and ammonium (NH₄⁺); available nitrate (NO₃⁻) and phosphorous (PO₄³⁻-P); exchangeable Al³⁺; total carbon, nitrogen and sulphur (CNS); and microbial biomass carbon (MBC). In addition, end-of-trial samples were analyzed for total cadmium (Cd), copper (Cu), lead (Pb), and zinc (Zn).

To assess nutrient and metal fluxes leaving the rooting zone, two ceramic cup samplers attached to 4.8 cm diameter PVC pipe (model 1900 Soilmoisture Equipment Corp.) were used to sample leachate from each plot at 35 to 40 cm (Site 1) and 50 to 55 cm (Site 2) which coincided with average rooting depths at each location (Figure 4.2). All 18 cups at Site 1 were installed in August, 2012, about a month before the start of sampling. A bucket auger was used to excavate holes to the required depth, and a powdered silica slurry used to ensure continuity between cups and soil. A layer of bentonite was also used to seal each sampler from preferential pipe flow before backfilling with soil. Nine cups at Site 2 were installed in October, 2012 with the other half installed in May, 2013 about a month before the start of sampling. Installation at Site 2 was the same as Site 1, except holes had to be dug by hand due to high coarse fragment content. Additional soil from nearby soil pits was also used for backfilling after sieving out larger gravel and cobbles.

Leachate sampling took place approximately bi-weekly for the first six weeks after ATB applications, then monthly for the remaining study period (when soils were not frozen or snow covered). Cups were placed under vacuum with a hand pump to 60 kPa three to four days prior to sampling, with samples collected in rinsed plastic bottles, kept in a cooler while in the field, then frozen until analysis at the end of each field season. Solutions were analyzed for pH, Ca^{2+} , Mg^{2+} , K^+ , NH_4^+ , NO_3^- , PO_4^{3-} , SO_4^{2-} , Fe^{n+} , Al^{3+} , Mn^{2+} , Cu^{2+} , and Zn^{2+} . Due to potential interaction with cup material (e.g., contamination or sorption) and/or precipitation caused by changes in solution pH or oxygen content with the sampler, ceramic cup samplers are not particularly well suited for PO_4^{3-} , Al^{3+} , Fe^{3+} , Mn^{2+} , Cu^{2+} , and Zn^{2+} assessment (e.g., Nagpal 1982; Raulund-Rasmussen 1989; Wenzel et al. 1997; Weihermüller et al. 2007). However, these analyses were included here to match variables measured in other study components.

Sample Analysis and Statistical Procedures

Sample analysis: Amendment Ca, Mg, K, P, and metals were determined through nitric acid digestion using a Microwave Accelerated Reaction System (MARS) followed by AAS analysis. The same method was used to measure metals in end-of-trial soil samples. Amendment pH and electrical conductivity (5:1 water) were measured using an ExStik EC500 meter (Extech Instruments). Soil pH was measured using 0.01 CaCl₂ solution (1:1 ratio) and a benchtop meter. Amendment and soil CNS were measured using a LECO induction furnace. Soil exchangeable bases (Ca²⁺, Mg²⁺, K⁺) were determined through 1M NH₄OAc extraction (adjusted to pH 7) followed by AAS analysis. Available PO₄³⁻-P was determined through colorimetric analysis after extraction with 0.5M NaHCO₃ solution. Exchangeable NH₄⁺ and available NO₃⁻ were determined through 2M KCl extraction followed by colorimetric analysis. Exchangeable Al³⁺ was determined through 1M KCl extraction followed by AAS analysis. A separate 1M KCl extraction was also conducted on pre-treatment soil samples to estimate the proportion of exchangeable acidity comprised of exchangeable Al³⁺ (standard titration method described by Sims 1996). MBC was assessed through fumigation direct extraction and colorimetric analysis (Voroney et al. 1993). For leachate samples, NH₄⁺ and NO₃⁻ were measured using colorimetric analysis with SO₄²⁻ assessed by ion chromatography (Waters Corp.). All other elements (ions) were determined through ICP or AAS analysis.

Data analysis: All response variables, except one-time soil metals analysis, were analyzed as repeated measures using SAS PROC MIXED (version 9.3, SAS Institute Inc.). Fixed factors and effects were treatment (control, low ATB rate, high ATB rate), time (days since treatment), and treatment-x-time interaction. Two blocking factors were used for the Latin square design at Site 1, and pre-treatment data were used as covariates in mineral soil analyses at both

sites. Normality of error terms was assessed for each variable using normal probability plot of residuals, and if violated, power transformations were used. Compound symmetry was the best covariance structure found and was used for all analyses. Significance of model terms and multiple means comparisons were generally assessed at $\alpha = 0.05$. However, means comparisons for leachate with significant treatment-x-time interactions were assessed at $\alpha = 0.01$ to reduce the possibility of Type I error inflation from the large number of treatment combinations associated with this study. Plots of means were generated using Minitab software version 17 (Minitab Inc.). End-of-trial soil metals data were assessed using Welch's ANOVA with Games-Howell comparison of means ($\alpha = 0.05$).

Due to differences in ATB chemical composition and application schedules, results from each trial site were treated as independent. Differences and similarities noted between trials are intended to highlight possible effects of ATB chemistry, soil conditions, and/or site variables on treatment responses.

RESULTS and DISCUSSION

Mineral Soil Chemistry

Ca and K were the only elements that showed a significant response to ATB treatment at Site 1, but only in the upper soil of high rate plots (Table 4.3). Overall, mean exchangeable Ca^{2+} increased by a factor of 4.6 in high rate plots versus the control, with concentrations also increasing over time (Figure 4.3). Low rate plots also showed consistent Ca increases, but these were not statistically significant (Figure 4.3). Mean exchangeable K^+ increased by a factor of 1.4 overall in high rate plots, but no time interaction was found (Figure 4.3). Although not statistically significant, exchangeable Mg^{2+} in high rate plots followed a similar trend to that of

K⁺ (Figure 4.3). Despite these increases in base cation concentration, upper soil pH was little affected by ATB treatment at Site 1 (Figure 4.3).

Ca and K were also the only elements to show a significant treatment response at Site 2 (Table 4.4), but overall trends were different than Site 1. Mean upper soil exchangeable Ca²⁺ was significantly greater with both application rates versus the control, but only in the first assessment period after treatment. Concentrations increased by a factor of 3.4 in high rate plots and 1.8 in low rate plots in fall, 2013, but were no different than control plots in 2014 (Figure 4.4). Lower soil exchangeable Ca²⁺ also showed an apparent response to treatment (Table 4.4), but this was due to decreases in control plot concentrations rather than treatment related increases in Ca, making this result suspect (Figure 4.5). Control plot data were also responsible for the near-significant response found in lower soil pH (Table 4.4, Figure 4.5), again suggesting soil/site variability rather than ATB treatment as the cause. With respect to K⁺, mean upper soil concentration increased by a factor of 1.4 overall in high rate plots versus the control (similar to Site 1), but results from low rate plots were more variable (Figure 4.4). Treatment related trends in mean upper soil exchangeable Mg²⁺ and pH were also variable at Site 2 (Figure 4.4).

Unlike forest floor horizons where ATB treatments led to immediate and significant increases in pH (Chapter 3), there were no significant increases in B- and BC-horizon mineral soil pH at either site over the study period. Price et al. (2015) reported early and significant increases in mineral soil pH with a single ATB application at similar rates used here, but this was in an agricultural soil where ATB was directly incorporated into surface mineral soil and pre-treatment pH was already above 5.0. In contrast, forest liming studies have shown that it can take years for surface lime applications to cause changes in mineral soil pH (e.g., Kreutzer 1995; Long et al. 2015). It is also conceivable that no significant pH changes will be detected in these

mineral soils after just one ATB treatment. This could be due, in part, to high forest floor buffering capacity (e.g., Meiwes et al. 2002), but also to the complex and often confounding relationships found between pH, Al speciation, and organic matter content in acidic forest soils compared to agricultural soils (Ross et al. 2008). For example, Brunner et al. (1999) reported base saturation ($\text{Ca}^{2+} + \text{Mg}^{2+} + \text{K}^+$) values of 11%-99% across a range of Norway spruce (*Picea abies*) stands in Switzerland, all with soil pH values below 4.75. This suggests pH is not a reliable indicator of potential base cation status in acidic forest soils.

The main purpose of applying ATB to spruce plantations in Nova Scotia is not to increase soil pH, but to offset current or predicted deficiencies in Ca due to acid leaching and harvest removals. This may best be assessed by looking at post-treatment changes in base cation concentrations and/or mass (Ross et al. 2008), or by changes in base cation / Al ratios since Al^{3+} dominates exchangeable soil acidity at both study sites (Table 4.5).

Looking only at pre-treatment and end-of-trial data, there was a clear improvement in mineral soil Ca content and base cation / Al ratios from ATB treatments at Site 1, but not at Site 2 (Table 4.6). Indeed, after adjustment for Al acidity fraction (Table 4.5), estimated % BS in high rate plots at Site 1 was in the 20% range thought to be critical for soil health (Driscoll et al. 2001). Several factors may have contributed to the higher rates of base cation exchange and retention found at Site 1 versus Site 2:

- Low clay content at Site 2 (6-8%) meant that almost all CEC was associated with soil organic matter (Johnson 2002) which was also dominated by strongly held and/or complexed Al^{3+} . In contrast, Site 1 had double the clay content and a larger percentage of more easily exchangeable H^+ ions (Tables 4.2 and 4.5).

- Site 1 is moderately well to imperfectly drained with a shallow, massive subsoil that likely led to higher soil solution concentrations per unit depth, more uniform distribution, and longer residence times compared to the deeper, well to rapidly drained soils at Site 2.
- Site 1 had a longer study period (and therefore ATB dissolution time) than Site 2 which, combined with more favourable soil/site conditions, likely led to increased cation exchange.

The short-term increase in exchangeable Ca^{2+} found in Site 2 upper soils (Figure 4.4) was somewhat surprising, but may again be related to site-specific soil and drainage conditions. Analysis of forest floor chemistry and surface ion fluxes at Site 2 showed substantial movement of Ca^{2+} related to treatment rate (Chapter 3) that likely led to the increase in upper soil exchangeable Ca^{2+} found during initial sampling. However, this early pulse may have been offset by ongoing movement of Al^{3+} from forest floor horizons where exchangeable Al^{3+} decreased over 90% by the end of 2014 (Chapter 3). If this was the case, a second ATB application could result in greater and/or longer Ca retention in Site 2 mineral soil.

Although % BS was improved in the high rate plots at Site 1, data also showed that Mg^{2+} concentrations were low prior to treatment and not greatly improved by ATB applications (Table 4.6). This was mainly due to the lack of Mg in the ATB product used (Table 4.1). Mg uptake by fine roots and mycorrhizae is sensitive to cation competition, and possible reduction of Mg uptake must be considered when acidic forest soils are limed (Ende and Evers 1997).

Soil Microbial Biomass

Soil microbial biomass (SMB) represents the living fraction of soil organic matter responsible for decomposition and nutrient cycling. In general terms, an increase in SMB is

viewed as beneficial, while a decrease can be detrimental if it leads to a decrease in biological function (Gonzalez-Quiñones et al. 2011). MBC measured at both trial sites showed expected depth trends (related to C and N content) and seasonal trends (related to available moisture), but no obvious treatment response (Figure 4.6, Tables 4.3 and 4.4). This is not surprising given the lack of significant response in mineral soil pH, C, N, and available P which, along with moisture content, typically drive SMB dynamics (e.g., He et al. 1997; Lupwayi et al. 2009; Gonzalez-Quiñones et al. 2011). Anderson (1998) found significant increases in MBC in forest floor and surface mineral horizons in a limed Norway spruce stand, but this was attributed to an increase in soil pH, not soil Ca. Although MBC measures were highly variable at Site 1, treatment related increases in Ca did not appear to affect SMB in the absence of similar changes in soil pH.

Trace Metals

End-of-trial assessments showed no statistically significant increases in mineral soil Cd^{2+} , Cu^{2+} , Pb^{2+} , or Zn^{2+} concentrations with ATB treatments at either site, although there was some trend toward increased Pb^{2+} in high rate plots (Table 4.7). These results are consistent with low metal ion fluxes measured below the forest floor at both sites (Chapter 3), as well as results reported by Banaitis (2009) after application of ATB at similar and higher rates in a young hardwood stand in Maine, USA. Results also reflect low metal contents in the Class A biosolids product used in this study (Table 4.1).

Soil metal concentrations in treated plots were well below Canadian standards for various land uses including agriculture (Table 4.7) and suggest several ATB applications could be accommodated on typical spruce plantation sites in Nova Scotia. Given that a single treatment has already resulted in significant and meaningful increases in mineral soil Ca at Site 1, further

use of ATB would likely lead to adequate replenishment of Ca long before metal contamination would be of concern.

Porous Cup Samplers

There were clear differences found in control plot leachate concentrations between sample years at Site 1, with values in fall, 2012, being much higher than those found in 2013 and 2014 for many ions. This suggested an installation effect on sample chemistry which was confirmed in 2013 when one sampler had to be re-installed after failing to consistently capture leachate samples. Data from that sampler were much higher after re-installation, mirroring trends found in 2012. Schreffler and Sharpe (2003) had similar results when using porous cup samplers in a forest amendment trial in Pennsylvania, USA, but they did not discuss a possible cause.

Since all porous cup samplers were installed at the same time at Site 1, it is possible that relative differences found between treatment and control plots are valid. However, absolute concentrations for 2012 are suspect, so assessment of treatment-x-time interactions are not valid unless 2012 data are separated from the rest. Consequently, leachate chemistry data for Site 1 are reported in two periods: 2012 and 2013-2014. Additionally, only ions that showed statistical differences in 2013-2014 were considered.

There was a significant and consistent increase in leachate Ca^{2+} in treated plots at Site 1 in 2013-2014, but no difference between treatment rates (Figure 4.7). These increases (also noted in 2012 data) were still evident at the end of 2014. K^+ showed a similar response to Ca^{2+} , although less pronounced (Figure 4.7). In contrast, Mg^{2+} showed a more immediate treatment response that was somewhat rate dependent, but with significant differences that only lasted until the end of 2013 (Figure 4.7). SO_4^{2-} concentrations were much higher than cation concentrations,

and significantly greater in treated plots versus controls in 2013, but not in 2014 (Figure 4.8).

Also of interest was the significant decrease in leachate pH found in high rate plots (Figure 4.8).

Leachate results from Site 1 are consistent with previously discussed results for mineral soils (this Chapter) and surface ion fluxes (Chapter 3). Greater retention of Ca^{2+} by soils in high rate plots reduced potential Ca^{2+} loss resulting in similar leaching of excess Ca^{2+} under both application rates. High inputs of Ca and K resulted in early displacement and loss of Mg^{2+} that was not replenished by amendment inputs. High SO_4^{2-} concentrations reflected the dominance of this companion anion in soil solution. Low leachate pH found in high rate plots reflected the displacement of H^+ ions and related increase found in % BS (Table 4.6).

A concern with applying high pH amendments on acidic forest soils is the potential for increased NO_3^- leaching from enhanced mineralization and/or nitrification (e.g., Kreutzer 1995; Pitman 2006). This is a concern for both water quality and further base cation loss. However, no significant increases in NO_3^- or NH_4^+ were found in Site 1 mineral soils or leachate, consistent with the lack of significant N input, mineralization, and nitrification found in forest floor horizons at this site (Chapter 3).

With respect to leaching of metals, all results for Cu^{2+} were below detection, as were the majority of results for Zn^{2+} , Al^{3+} , Mn^{2+} , and Fe^{n+} (data not shown). All PO_4^{3-} results were also below detection. It is unclear whether these results reflected actual soil solution chemistry, interference of porous cup material on sample chemistry, or a combination of both. However, based on ion flux data collected below the forest floor (Chapter 3), significant leaching of Cu^{2+} or Zn^{2+} would be unlikely at Site 1, while some increase in soil solution Al^{3+} (and perhaps Mn^{2+}) would have been expected.

Half of the porous cup samplers at Site 2 were installed in fall, 2012, with the remainder installed in spring, 2013. The installation effect noted at Site 1 was also found at Site 2 which made it necessary to separate new and old installation data for 2013, whereas all data could be combined for 2014. No treatment related responses or trends in leachate chemistry were found at Site 2, as exemplified by Ca^{2+} results (Figure 4.9). Indeed, although highly variable, control plots often had higher leachate Ca^{2+} concentrations than treated plots in 2013.

One of the reasons ATB treatments at Site 2 were delayed until summer, 2013, was the perceived risk of extensive leaching losses with heavy fall rains at this well to rapidly drained site. Ion fluxes measured in near-surface mineral soils at both study sites clearly showed similar inputs after ATB applications (Chapter 3). This, combined with the lack of long-term changes in mineral soil base cation concentrations, suggests that leaching losses must have been significant at Site 2 even though leachate data did not support this finding. A possible explanation is that porous cup samplers at Site 2 were too small and/or too few to representatively sample the fast, preferential flow that is often associated with coarse, gravelly soils (e.g., Curley et al. 2011; Wang et al. 2012). Systematic rather than event sampling could have also allowed significant base cation losses to go unnoticed. In contrast, more uniform moisture distribution and restricted drainage at Site 1 probably allowed for more representative leachate sampling, even with a systematic sampling regime.

CONCLUSIONS

Alkaline-treated biosolids (ATB) were surface applied at 7.5 t ha^{-1} and 15 t ha^{-1} (wet weight) under two 10-15-year old white spruce plantations in Nova Scotia, Canada. Analysis showed: (i) increases in end-of-trial Ca and base cation / Al ratios in imperfectly drained loamy

soils (Site 1), but no increases in these parameters in well drained coarse soils (Site 2), (ii) no treatment related changes in mineral soil pH, (iii) no treatment related changes in mineral soil microbial biomass, (iv) no treatment related increases in NO_3^- leaching, and (v) no significant increases in end-of-trial mineral soil Cd, Cu, Pb, and Zn concentrations. Soil and site conditions at Site 1 (higher clay content, restricted drainage, and lower Al fraction of exchangeable acidity) probably allowed for greater base cation responses than conditions at Site 2 (lower clay content, unrestricted drainage, and higher Al fraction of exchangeable acidity).

Porous cup sampler data showed significant increases in soil solution Ca^{2+} and K^+ at Site 1 that was ongoing and unrelated to application rate, as well as significant increases in Mg^{2+} that was rate and time dependent. It is also suspected that base cation leaching loss was extensive at Site 2 despite the lack of supporting porous cup sampler data.

Results from this study, and a related assessment of forest floor and surface ion flux chemistry (Chapter 3), suggest that ATB could be a good source of Ca in Ca-limited sites, but that soil and site conditions need to be considered when developing treatment regimes. Application rates of 7.5 t ha^{-1} and 15 t ha^{-1} resulted in similar changes in forest floor chemistry, but not deeper mineral soil chemistry, on two contrasting sites. The following general conclusions can be made:

- Sites with loamy soils (clay $> 10\%$) and an exchangeable H^+ acidity of $\geq 20\%$ can probably benefit from a single, high rate ATB application (e.g., 15 t ha^{-1}).
- Sites with coarse soils (clay $\leq 10\%$), an exchangeable H^+ acidity of $< 20\%$, and well to rapid drainage should be initially treated with a lower ATB rate to avoid excessive loss of base cations. In the case of newly established spruce plantations, it should be possible to

treat sites twice within a 5-year period before potential seedling damage prevents further machine access.

- Soils/sites with low Mg levels may not fully benefit from ATB applications due to enhanced displacement, leaching loss, and lack of amendment input. An additional Mg fertilizer, or possibly a mixture of dolomitic limestone and ATB, may be required on sites with low Mg and Ca levels.
- Neither site treated in this study showed a significant increase in N mineralization, nitrification, or NO_3^- leaching from increased forest floor pH, but this possibility still exists on other site types and should be monitored, especially on relatively N-rich sites.
- Low inputs of heavy metals from Class A biosolids, combined with enhanced forest floor retention at higher pH values, suggests that ATB can be safely applied to spruce plantation ecosystems at the expected rates and frequencies needed to offset Ca deficits.



Figure 4.1. Sample profiles for a shaly loam soil derived from slate till (Site 1 - left) and a gravelly/cobbly sandy loam soil derived from granitic till (Site 2 – right). Both soils are classed as Orthic Humo-Ferric Podzols (SCWG 1998) or Typic Haplorthods (USDA 1999).

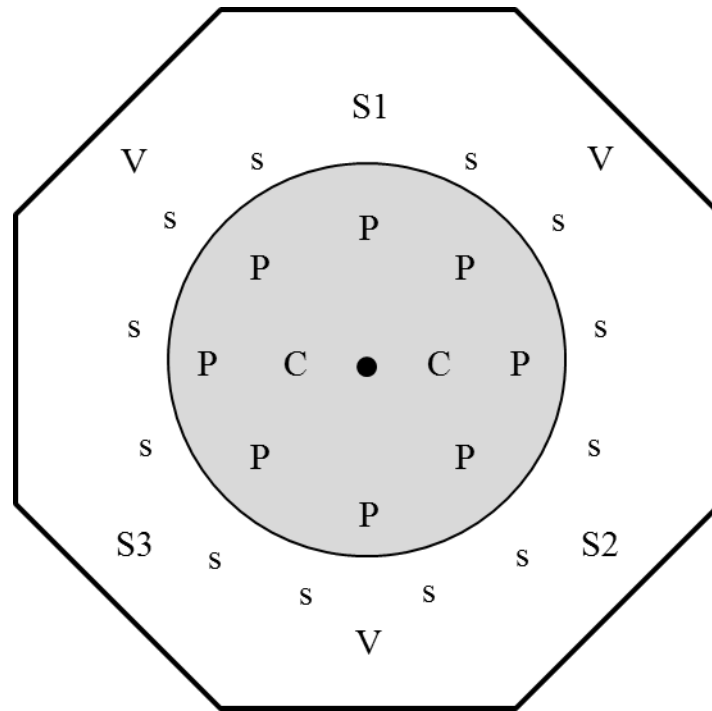


Figure 4.2. Schematic of a single field plot with systematic sample points based on a random initial bearing for soil pit 1 (S1). Octagon: ATB treatment area within 40 m x 40 m plot. Grey circle: tree growth assessment area (10.3 m radius). C = porous cup solution samplers (4 m from centre). P = paired PRSTM-probes (8 m from centre). S1-S3 = pre-treatment soil pit sampling sites (13 m from centre). s = post-treatment soil sampling sites (12 m from centre). V = ground vegetation monitoring sub-plots (13-14 m from centre).

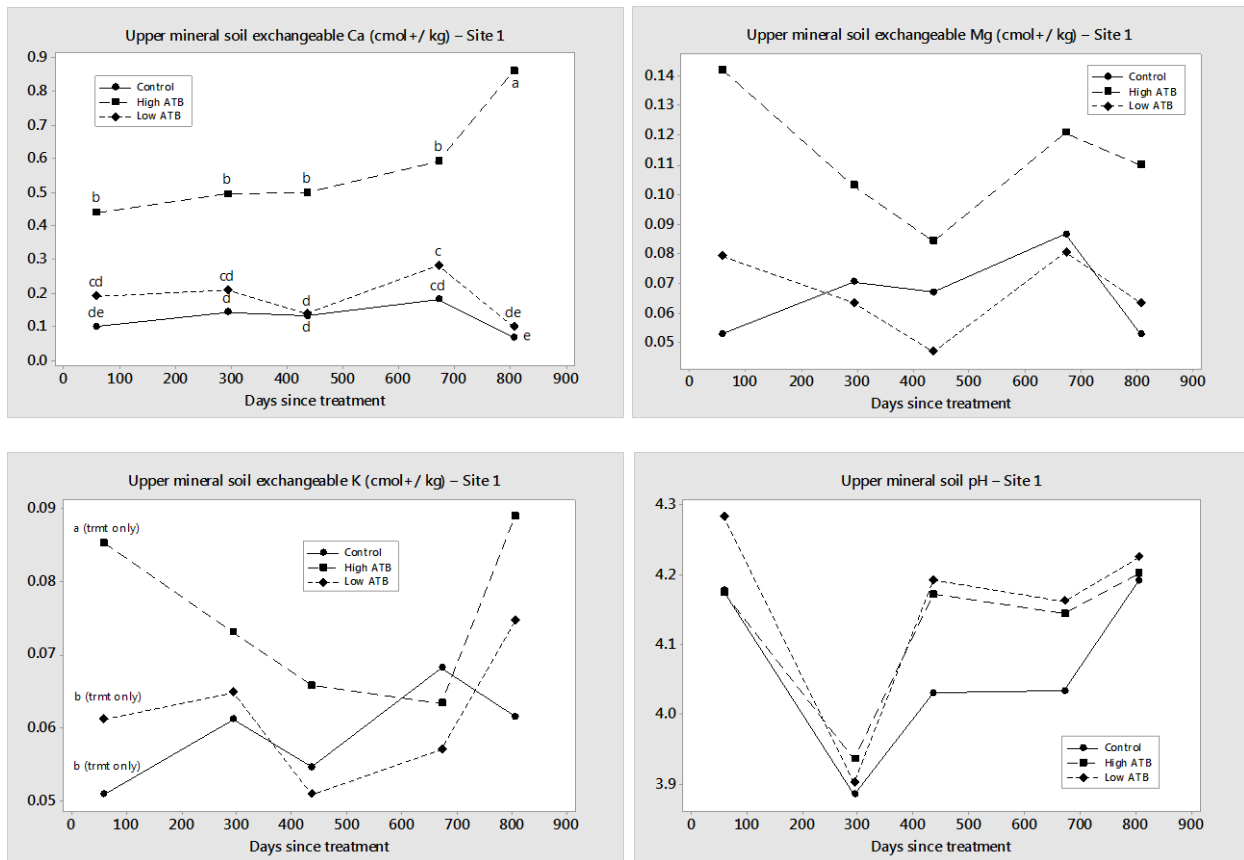


Figure 4.3 Mean upper mineral soil exchangeable Ca^{2+} , Mg^{2+} , K^{+} , and pH by treatment and time for Site 1. Values with different letters are statistically different at $p = 0.05$.

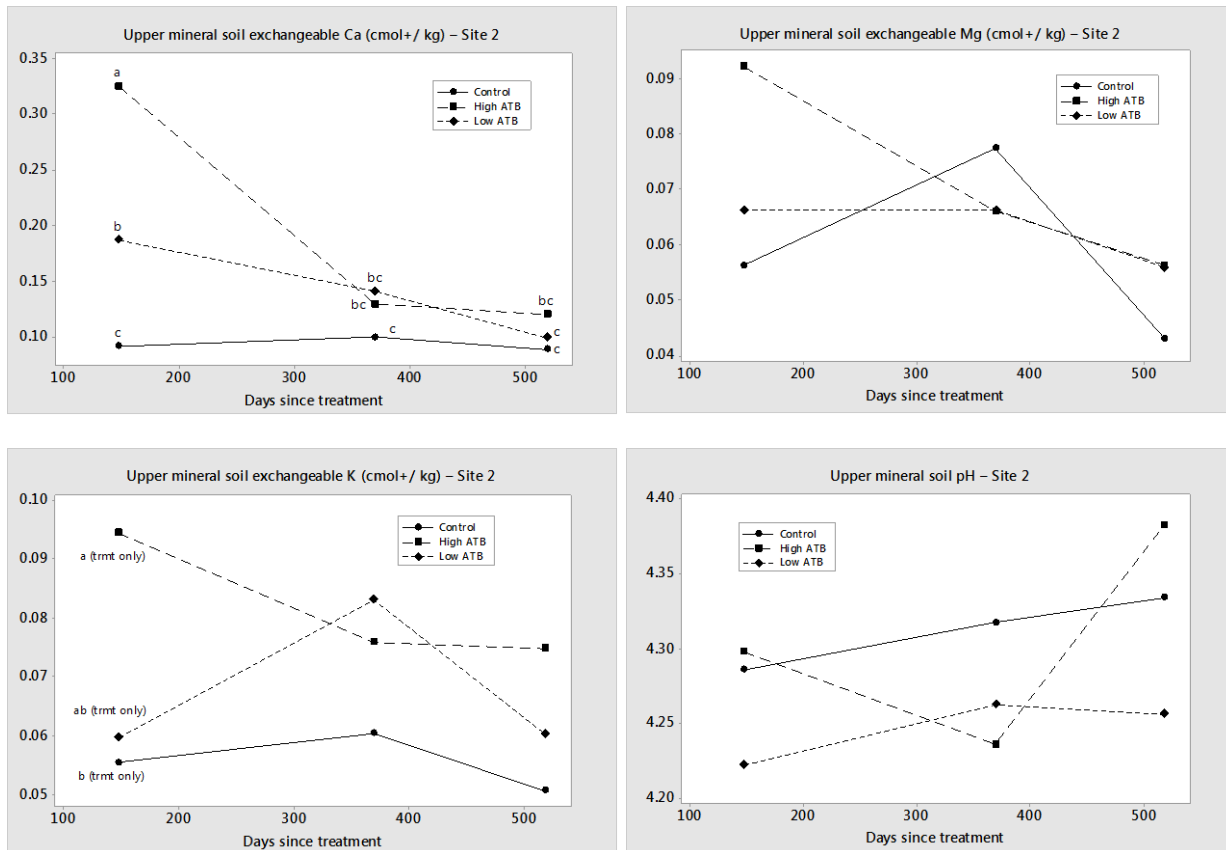


Figure 4.4. Mean upper mineral soil exchangeable Ca^{2+} , Mg^{2+} , K^+ , and pH by treatment and time for Site 2. Values with different letters are statistically different at $p = 0.05$.

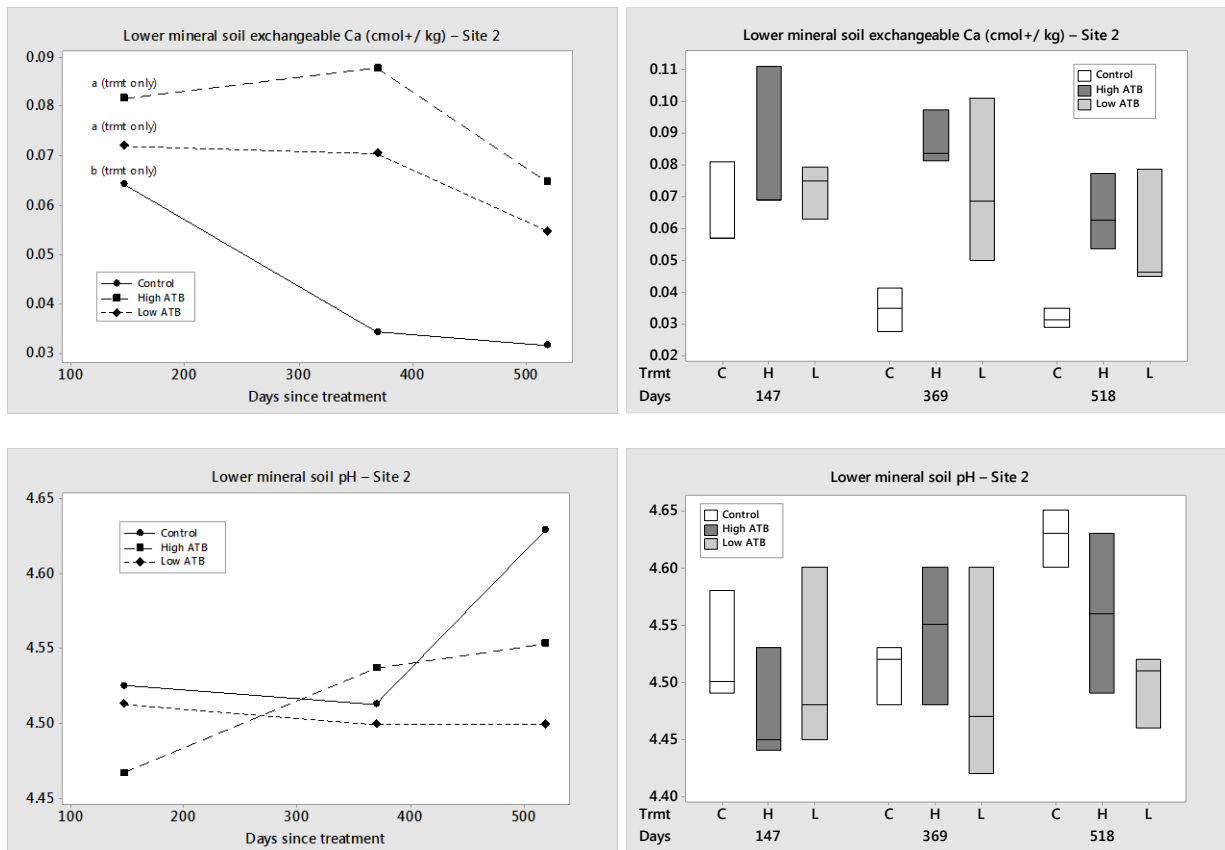


Figure 4.5. Mean lower mineral soil exchangeable Ca^{2+} and pH (left) and data range (right) by treatment and time for Site 2. Values with different letters are statistically different at $p = 0.05$.

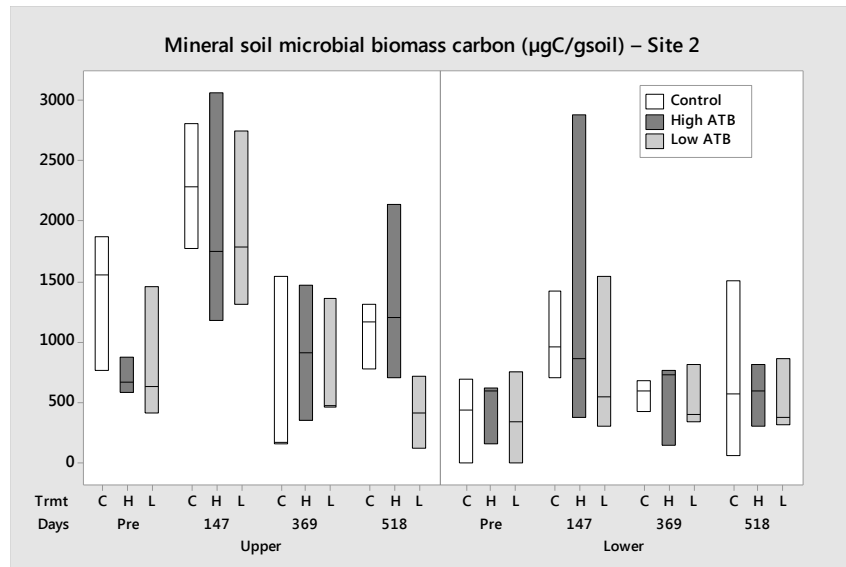
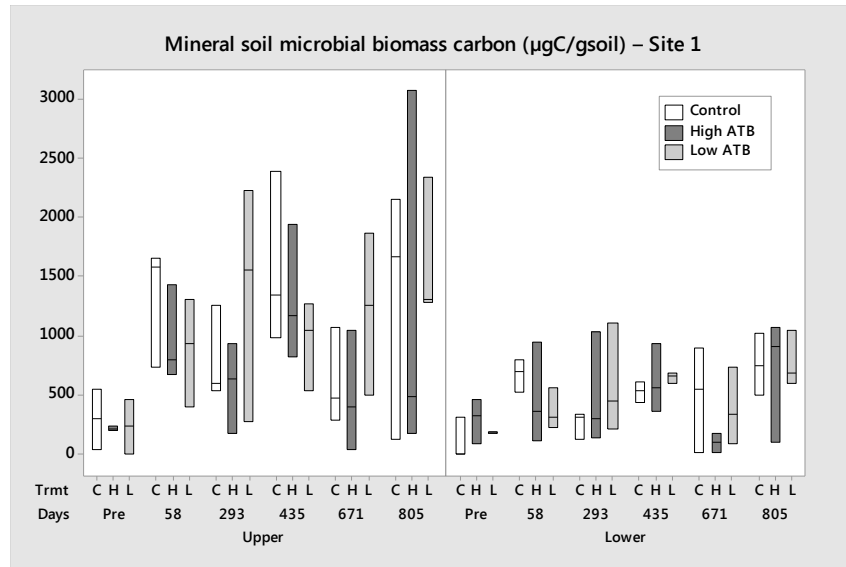


Figure 4.6. Upper and lower mineral soil microbial biomass carbon at Site 1 (top) and Site 2 (bottom) by treatment and time (Pre = pre-treatment measures).

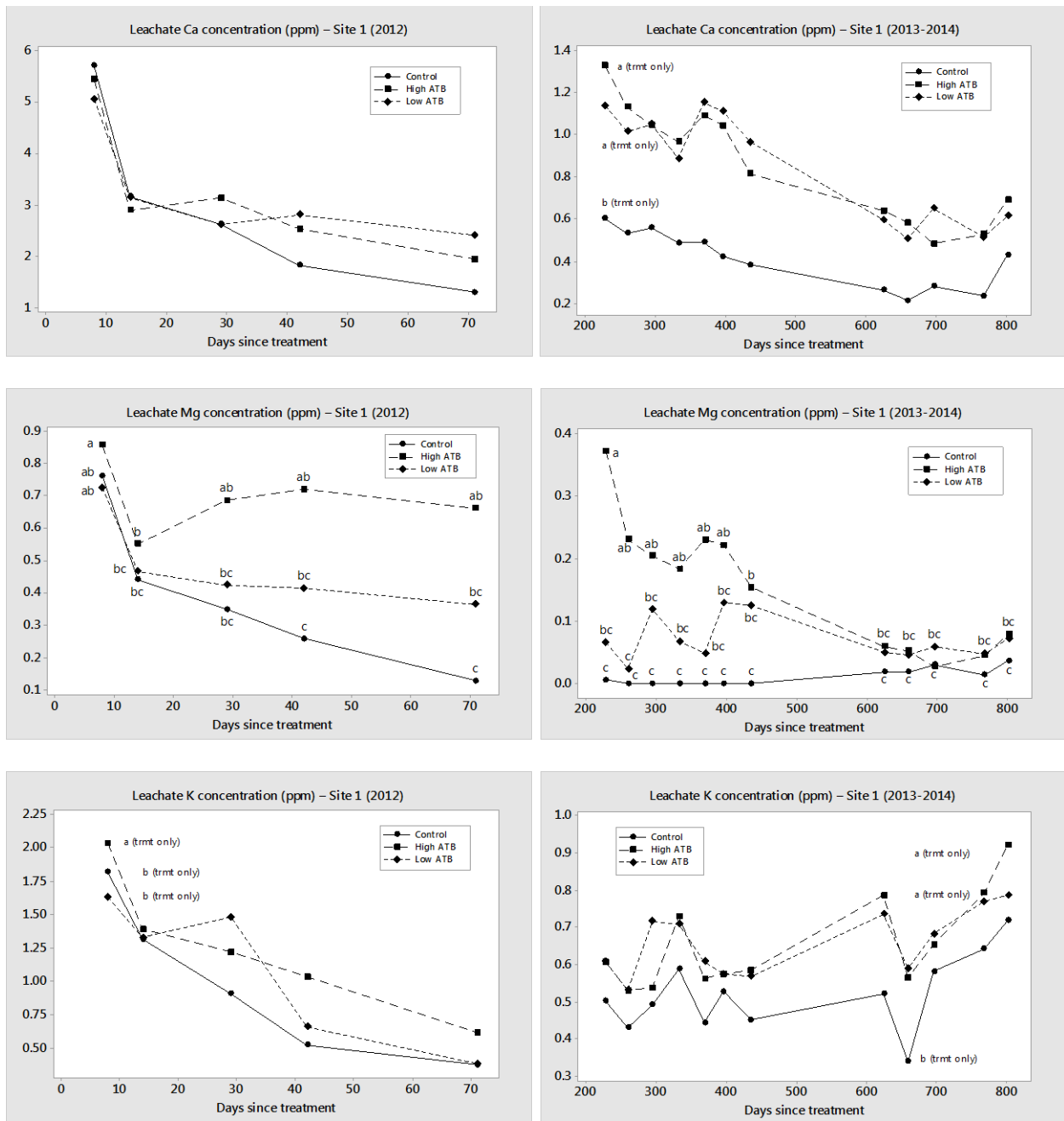


Figure 4.7. Porous cup sampler Ca^{2+} , Mg^{2+} , and K^{+} concentrations from Site 1 by treatment and time. 2012 data (left) are separate from 2013-2014 data (right) due to a cup installation impact on leachate chemistry. Values with different letters are statistically different at $p = 0.05$ (trmt only) or $p = 0.01$ (trmt x time).

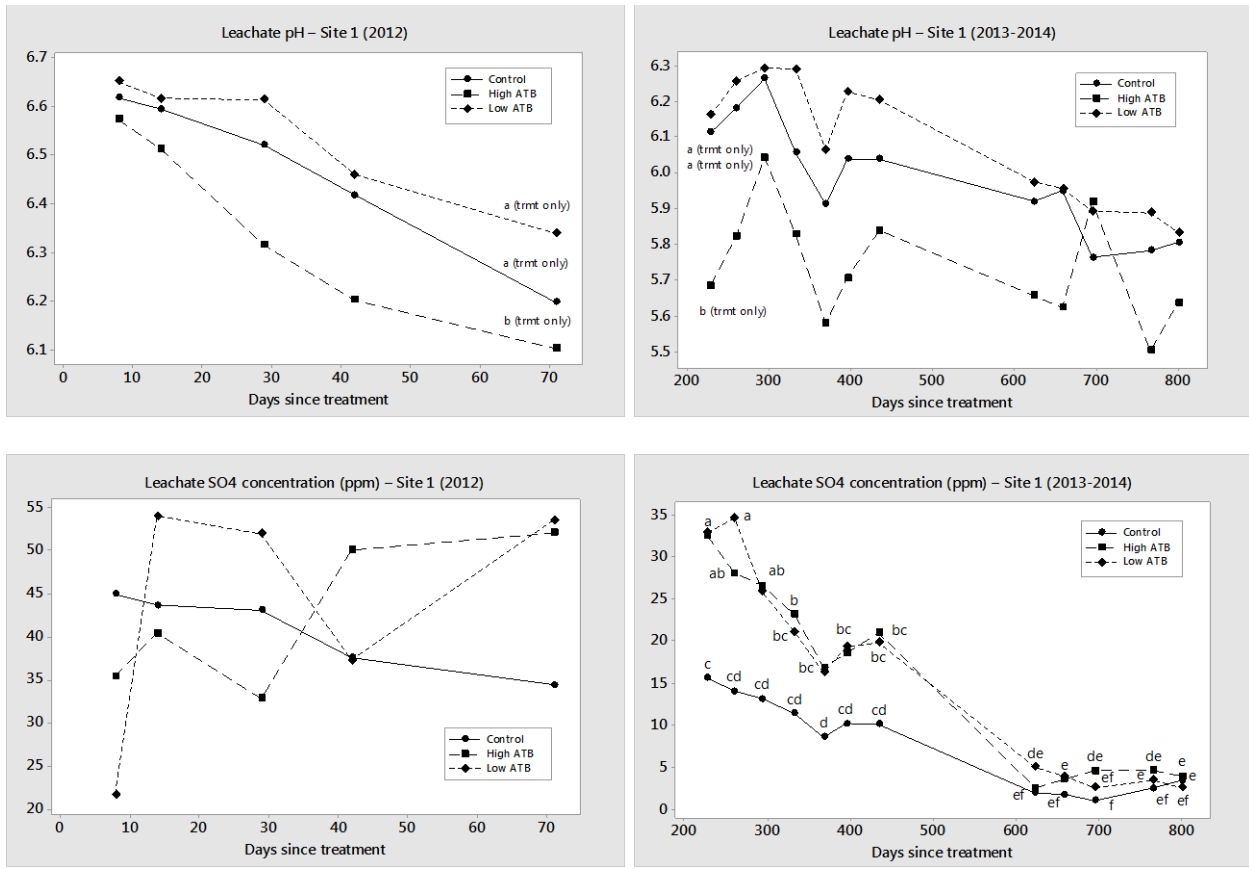


Figure 4.8. Porous cup sampler pH and SO₄²⁻ concentrations from Site 1 by treatment and time. 2012 data (left) are separate from 2013-2014 data (right) due to a cup installation impact on leachate chemistry. Values with different letters are statistically different at p = 0.05 (trmt only) or p = 0.01 (trmt x time).

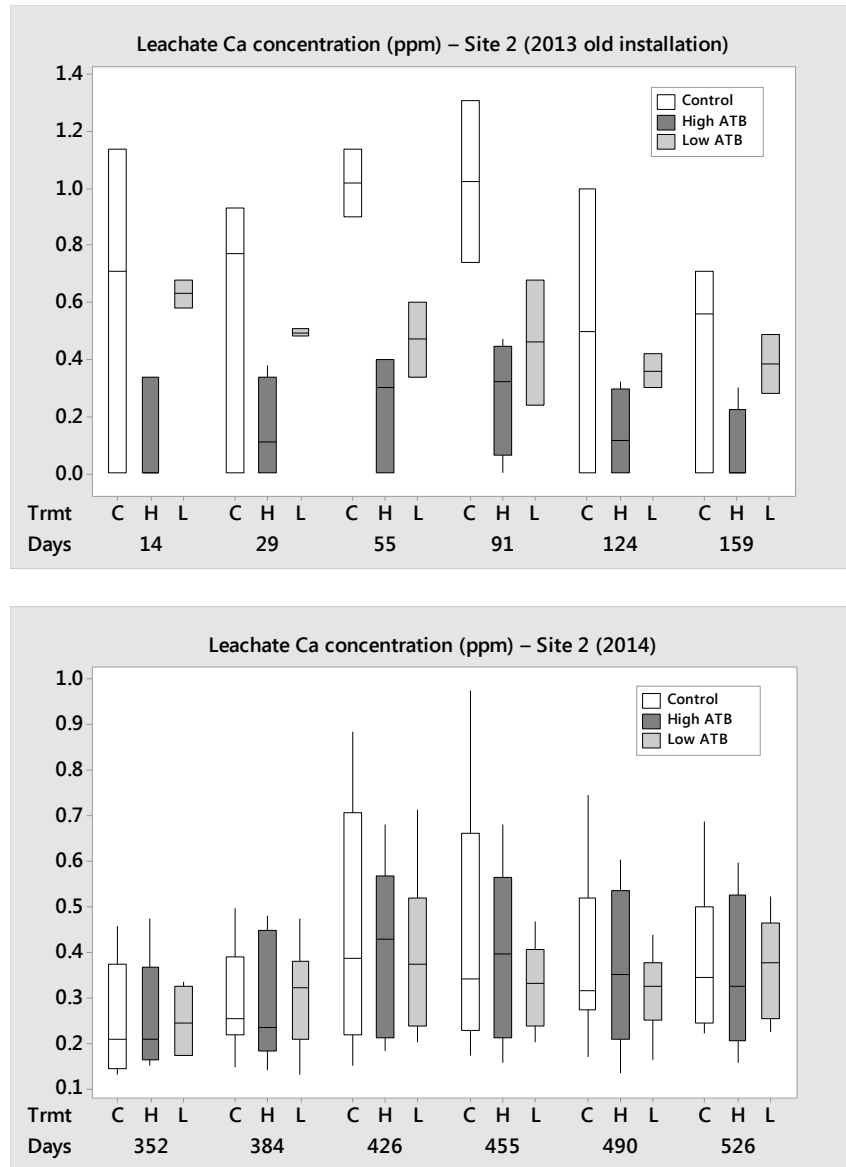


Figure 4.9. Porous cup sampler Ca^{2+} concentrations from Site 2 by treatment and time. 2013 data (top) are from older installations only. 2014 data (bottom) are from all samplers.

Table 4.1. Summary of ATB chemistry and estimated application rates for Site 1 and Site 2. All values are from on-site samples except Cd and Pb values which came from quarterly analyses conducted by N-Viro Systems Canada for 2012 and 2013.

Site 1															
Statistic	pH	EC	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn
(n=6)		(mS) (% wt) (mg kg ⁻¹)							
Mean	10.9	4.65	20.6	0.41	1.72	0.85	0.13	0.52	7,571	218	18,358	107	<0.1	79	494
Stdev	0.5	0.25	1.16	0.03	0.04	0.17	0.06	0.04	416	15	1,135	5	nd	nd	58
% CV	5	5	6	7	2	20	44	7	5	7	6	5	nd	nd	12
Estimated element application (kg ha⁻¹)															
	Trmt	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn	
	High	1,914	38	160	79	12	49	70	2.0	171	1.00	<0.001	0.74	4.6	
	Low	957	19	80	39	6	24	35	1.0	85	0.50	<0.001	0.37	2.3	

Site 2															
Statistic	pH	EC	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn
(n=4)		(mS) (% wt) (mg kg ⁻¹)							
Mean	9.7	5.47	20.1	0.34	1.65	0.87	0.08	0.58	7,550	226	20,765	101	<0.1	117	180
Stdev	1.3	1.18	2.38	0.04	0.32	0.04	0.01	0.05	767	38	2,240	9	nd	nd	14
% CV	13	22	12	10	19	5	13	9	10	17	11	8	nd	nd	8
Estimated element application (kg ha⁻¹)															
	Trmt	Ca	Mg	K	N	S	P	Fe	Mn	Al	Cu	Cd	Pb	Zn	
	High	1,870	32	153	81	7	54	70	2.1	193	0.94	<0.001	1.09	1.7	
	Low	935	16	77	41	4	27	35	1.1	97	0.47	<0.001	0.54	0.8	

Table 4.2. Average texture, coarse fragment (CF), and bulk density (Db) data for Site 1 and Site 2 soils.

Statistic (n=9)	Site 1 Upper Mineral					Site 1 Lower Mineral				
	%Sand	%Silt	%Clay	%CF	Db (Mg m⁻³)	%Sand	%Silt	%Clay	%CF	Db (Mg m⁻³)
Mean	39	43	17	16	1.00	45	40	15	31	1.46
Stdev	5	4	3	2	0.16	8	6	4	4	0.09
% CV	14	9	15	10	16	19	14	24	14	6

Statistic (n=6)	Site 2 Upper Mineral					Site 2 Lower Mineral				
	%Sand	%Silt	%Clay	%CF	Db (Mg m⁻³)	%Sand	%Silt	%Clay	%CF	Db (Mg m⁻³)
Mean	63	29	8	28	0.63	60	34	6	41	0.99
Stdev	7	5	2	5	0.10	7	6	1	8	0.16
% CV	10	18	20	17	16	11	16	23	18	16

Table 4.3. Repeated measures analysis results for mineral soil chemistry and microbial biomass carbon (MBC) at Site 1 (upper and lower soil). Significant treatment and treatment-x-time interactions have been shaded (alpha = 0.05).

Site 1 Upper Soil												
Effect	Ca	Mg	K	P	NH ₄	NO ₃	N	S	C	Al	pH	MBC
Block 1	0.196	0.630	0.044	0.130	0.146	0.381	0.415	0.093	0.248	0.573	0.345	0.614
Block 2	0.297	0.495	0.106	0.105	0.405	0.427	0.780	0.066	0.207	0.796	0.368	0.783
Pre-trmt Value	0.430	0.548	0.042	0.095	0.348	0.376	0.653	0.067	0.181	0.939	0.803	0.516
Treatment	0.048	0.451	0.026	0.108	0.692	0.414	0.903	0.135	0.392	0.879	0.387	0.605
Days	0.014	0.031	0.048	0.294	0.105	<.0001	0.000	0.001	0.615	0.183	<.0001	0.409
Trmt x Days	0.005	0.220	0.241	0.651	0.227	0.959	0.607	0.506	0.970	0.767	0.736	0.841

Site 1 Lower Soil												
Effect	Ca	Mg	K	P	NH ₄	NO ₃	N	S	C	Al	pH	MBC
Block 1	0.851	0.322	0.770	0.039	0.143	0.587	0.767	0.757	0.742	0.142	0.210	0.762
Block 2	0.679	0.511	0.927	0.040	0.261	0.503	0.771	0.284	0.780	0.609	0.108	0.451
Pre-trmt Value	0.807	0.344	0.679	0.627	0.328	0.663	0.700	0.763	0.847	0.207	0.085	0.547
Treatment	0.196	0.395	0.429	0.090	0.547	0.623	0.929	0.631	0.970	0.222	0.062	0.813
Days	0.185	0.057	0.036	0.044	0.766	<.0001	<.0001	0.002	0.137	0.272	<.0001	0.116
Trmt x Days	0.314	0.247	0.205	0.721	0.254	0.291	0.270	0.237	0.998	0.461	0.207	0.745

Table 4.4. Repeated measures analysis results for mineral soil chemistry and microbial biomass carbon (MBC) at Site 2 (upper and lower soil). Significant treatment and treatment-x-time interactions have been shaded (alpha = 0.05).

Site 2 Upper Soil												
Effect	Ca	Mg	K	P	NH₄	NO₃	N	S	C	Al	pH	SMB
Pre-trmt Value	0.806	0.521	0.056	0.673	0.474	0.017	0.205	0.690	0.291	0.497	0.234	0.257
Treatment	0.054	0.479	0.036	0.685	0.371	0.150	0.673	0.703	0.838	0.366	0.483	0.079
Days	0.004	0.116	0.404	0.246	0.003	0.000	0.040	0.001	0.077	0.005	0.396	0.031
Trmt x Days	0.029	0.197	0.460	0.638	0.615	0.586	0.588	0.526	0.703	0.230	0.659	0.857

Site 2 Lower Soil												
Effect	Ca	Mg	K	P	NH₄	NO₃	N	S	C	Al	pH	SMB
Pre-trmt Value	0.933	na	0.108	0.109	0.028	0.609	0.416	0.802	0.011	0.820	0.437	0.847
Treatment	0.009	0.077	0.585	0.778	0.246	0.732	0.890	0.816	0.295	0.748	0.477	0.729
Days	0.010	0.034	0.235	0.352	0.002	<.0001	0.002	0.011	0.262	0.009	0.024	0.241
Trmt x Days	0.155	0.771	0.375	0.390	0.174	0.563	0.622	0.320	0.662	0.773	0.057	0.980

Table 4.5. Fraction of exchangeable acidity comprised of exchangeable Al³⁺ in upper and lower mineral soil samples at Site 1 and Site 2 (pre-treatment samples only).

Statistic	Site 1		Site 2	
	Upper	Lower	Upper	Lower
Mean	0.78	0.92	0.97	0.99
Stdev	0.15	0.11	0.04	0.02
% CV	12	10	4	2
n	7	9	9	9

Table 4.6. Mean base cation mass (kg ha⁻¹) and aluminum ratios for upper and lower mineral soils before treatment (2012) and at end-of-trial (2014) for Site 1 and Site 2. Masses were calculated using data contained in Table 4.2 (assuming 15 cm thick horizons).

Site 1 Upper Mineral Soil						
Year	Trmt	Ca	Mg	K	Ca/Al	BC/Al
2012	H	24.9	9.9	29.7	0.02	0.05
	L	18.8	8.1	22.1	0.02	0.05
	C	22.3	6.6	22.2	0.02	0.04
2014	H	194.3	17.6	54.1	0.19	0.25
	L	57.5	11.3	41.4	0.06	0.10
	C	22.1	12.1	36.0	0.02	0.06
Site 1 Lower Mineral Soil						
Year	Trmt	Ca	Mg	K	Ca/Al	BC/Al
2012	H	11.8	3.0	29.6	0.02	0.04
	L	24.7	3.3	27.6	0.04	0.06
	C	30.7	3.0	26.9	0.04	0.07
2014	H	106.8	7.3	49.6	0.18	0.25
	L	38.9	6.2	43.8	0.06	0.10
	C	14.1	7.7	37.4	0.02	0.06
Site 2 Upper Mineral Soil						
Year	Trmt	Ca	Mg	K	Ca/Al	BC/Al
2012	H	29.6	8.8	18.8	0.04	0.08
	L	42.2	8.2	23.9	0.07	0.11
	C	22.1	6.9	40.0	0.03	0.08
2014	H	40.9	7.2	26.8	0.05	0.09
	L	33.5	6.3	22.8	0.04	0.07
	C	17.3	5.3	19.8	0.02	0.04
Site 2 Lower Mineral Soil						
Year	Trmt	Ca	Mg	K	Ca/Al	BC/Al
2012	H	14.9	3.0	15.8	0.03	0.07
	L	23.8	3.0	23.8	0.06	0.11
	C	17.8	3.0	22.8	0.04	0.07
2014	H	19.2	3.1	23.1	0.04	0.07
	L	16.8	2.6	21.9	0.04	0.07
	C	9.4	1.6	28.0	0.02	0.05

Table 4.7. Mean metal concentrations from end-of-trial soil samples at Site 1 and Site 2 (upper and lower soils combined) along with allowable soil concentrations by land-use as reported by the Canadian Council of Ministers of the Environment (CCME 2007).

Site 1 mean soil concentration (mg kg⁻¹)			
Element	Control	Low ATB	High ATB
Cd	0.00	0.01	0.00
Cu	17.9	23.0	18.7
Pb	4.5	6.5	10.9
Zn	62.1	60.8	61.8

Site 2 mean soil concentration (mg kg⁻¹)			
Element	Control	Low ATB	High ATB
Cd	0.34	0.00	0.15
Cu	13.3	10.2	9.3
Pb	3.7	3.9	9.8
Zn	87.6	73.5	64.7

CCME allowable soil concentration (mg kg⁻¹)			
Element	Residential/		
	Agricultural	Parkland	Commercial
Cd	1.4	10	22
Cu	63	63	91
Pb	70	140	260
Zn	200	200	360

CHAPTER 5

TREE GROWTH, FOLIAGE CHEMISTRY, AND GROUND VEGETATION CHANGES AFTER SURFACE APPLICATION OF ALKALINE-TREATED BIOSOLIDS UNDER TWO WHITE SPRUCE (*Picea glauca*) PLANTATIONS IN NOVA SCOTIA, CANADA

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ABSTRACT

Two field trials were established to evaluate the use of alkaline-treated biosolids (ATB) to offset current or predicted Ca deficits in Nova Scotia forest soils under juvenile white spruce (*Picea glauca*) plantations. ATB treatments generally increased foliage Ca, K, and (to a lesser extent) P concentrations in white spruce in relation to application rate, but in natural red spruce, only foliage K and P were increased. Lack of Ca uptake by red spruce may have been due to a combination of competition and Al stress on fine root development and function, but this could not be confirmed. Lack of Mg uptake by both species was likely due to low amendment inputs, Mg leaching loss, and cation antagonism. Despite improvements in foliage nutrient contents in

treated plots, there were only slight increases in tree diameter increment over two growing seasons, and no change in height increment. This was probably due to a lack of significant N inputs and/or to Mg deficiencies or imbalances resulting from ATB treatment. With respect to ground vegetation, ATB treatments led to changes in red raspberry (*Rubus idaeus*) foliage chemistry that mirrored those found in white spruce, but treatments had no significant effect on overall species abundance and diversity. This was attributed to minimal changes in available N from ATB treatments, and to the already current dominance of pioneer and disturbance species in these young plantation sites. Results from this study, and related assessments of forest floor, mineral soil, and leachate chemistry, suggest that ATB could be a good source of Ca in Ca-limited plantation sites, while also enhancing K and P availability and uptake. However, possible increases in plantation productivity may be hindered by decreases in available Mg and/or base cation imbalances caused by ATB treatment.

KEYWORDS

Alkaline-Treated Biosolids, Spruce Plantations, Foliage Chemistry, Tree Growth, Ground Vegetation

INTRODUCTION

Globally, the main use of fertilization in forestry has been to increase biomass production in forest plantations (Smethurst 2010). However, lime and wood ash have also been routinely used in some European forests (e.g., Tomlinson 1990; Nilsson et al. 2001; Levin and Eriksson 2010) to mitigate the impacts of acid deposition, biomass harvesting, and/or calcium (Ca) and magnesium (Mg) deficiencies. In northeastern North America, decades of acid deposition has

resulted in significant Ca, Mg, and potassium (K) depletion in many forest soils (e.g., Watmough and Ouimet 2005; Lawrence et al. 2012), but to date there has been little use of liming amendments to mitigate these impacts (Moore et al. 2015). In Nova Scotia, Canada, nutrient budget assessments have shown Ca to be a potentially limiting nutrient in many spruce plantation sites (Keys et al. 2016), in keeping with regional trends. This is related to the presence of relatively nutrient-poor, slowly weatherable soils in many parts of the province (Whitfield et al. 2006; Keys et al. 2016).

In addition to lime and wood ash, alkaline-treated biosolids (ATB) are also considered a liming amendment and nutrient supplement, but despite routine use in agriculture, use in forests has been minimal to date (Banaitis et al. 2009). Appropriate use of ATB in forests could, however, offset Ca deficiencies caused by acid deposition and harvesting, while also providing another end-use for this waste-stream product. To evaluate the potential for ATB to offset current or predicted Ca deficits in Nova Scotia forest soils, two field trials were established to measure the effects of surface applied ATB on white spruce (*Picea glauca*) plantation soils and vegetation.

ATB treatment effects on forest floor, near-surface ion fluxes, mineral soil chemistry, and rooting zone leachate chemistry have already been discussed in Chapters 3 and 4. Results showed: (i) significant and persistent increases in forest floor pH and Ca, (ii) no increase in forest floor Mg and K, (iii) significantly reduced forest floor exchangeable aluminum (Al^{3+}), (iv) variable changes in mineral soil base cation / Al ratios, (v) no change in mineral soil pH, (vi) minimal effect on total or available nitrogen (N), (vii) slightly enhanced surface soil phosphorous (P) availability, (viii) significant loss of Mg in rooting zone leachate, (ix) no increase in nitrate (NO_3^-) leaching, (x) no changes in mineral soil microbial biomass, and (xi) negligible leaching or

accumulation of trace metals [copper (Cu), cadmium (Cd), lead (Pb) and zinc (Zn)]. This chapter discusses effects of ATB treatments on ground vegetation cover, foliage chemistry, and plantation tree growth.

METHODS

A full description of experimental design is found in Chapter 3. A brief summary is presented here for context, along with details related to vegetation and tree growth assessments.

Two 10-15-year old upland white spruce plantations in central Nova Scotia, Canada were selected for independent field trials. Site 1 was moderately well to imperfectly drained and underlain by a shaly loam soil derived from slate till. Site 2 was well to rapidly drained and underlain by a gravelly/cobbly sandy loam soil derived from granitic till. Soils at both sites were field-classified as Humo-Ferric Podzols (SCWG 1998) or Typic Haplorthods (USDA 1999).

An ATB product manufactured by N-Viro Systems Canada and trademarked as Halifax Soil Amendment™ was used in this study. This product is categorized as a Class A biosolids under Nova Scotia Department of Environment regulations (NSE 2010) and is sold commercially under an approved fertilizer label. Target application rates were 1,000 kg ha⁻¹ Ca (low rate) and 2,000 kg ha⁻¹ Ca (high rate) with default concentrations used for other elements. Actual Ca application rates were determined to be 957 kg ha⁻¹ and 1,914 kg ha⁻¹ (Site 1: low and high rates), and 935 kg ha⁻¹ and 1,870 kg ha⁻¹ (Site 2: low and high rates) (Table 5.1). Site 1 – a Latin square design – was treated in September, 2012. Site 2 – a random plot design – was treated in June, 2013. Post-treatment measurements at both trial sites were taken to November, 2014.

A systematic sampling scheme was developed to assess soil and vegetation variables at each plot (Figure 5.1). Vegetation responses included tree growth measurements (height and

diameter) and pre- and post-treatment assessment of tree and dominant ground vegetation foliage chemistry [total Ca, Mg, K, P, N, sulphur (S), carbon (C), manganese (Mn), and Zn]. Tree measurements were conducted in October of each year within a 10.3 m radius circle from plot centre (Figure 5.1). Natural ingrowth at Site 1 also allowed for measurement of red spruce (*Picea rubens*) trees along with planted white spruce. Only visually healthy, undamaged trees were selected and tagged for ongoing assessment. Tree heights were measured to the nearest centimetre using a height pole. Diameters at 1.3 m from the ground were measured to the nearest millimetre using a diameter tape. Total sample sizes ranged from 45 to 150 trees depending on species, measurement year, and site. Tree foliage samples were collected in October from eight randomly selected trees in each plot. Two current-year shoots of equal size were collected from the upper half of selected trees and pooled for analysis. Due to its wide distribution and coverage at each site, red raspberry (*Rubus idaeus*) was chosen as a representative ground vegetation species for foliage assessment. Samples were collected from scattered plants in each plot during July and pooled for analysis. After pooling, foliage sample size was equal to three for each species/treatment combination.

In addition to growth and foliage assessments, three 1 m x 1 m vegetation subplots were systematically established prior to treatment and ocular estimates of percent cover recorded for each species found (Figure 5.1). This assessment was repeated two years later by the same personnel to determine if ATB treatments had any effect on species abundance and diversity as measured by changes in species counts, average percent cover, and Shannon diversity index (Shannon 1948). Vegetation assessment sample size was equal to nine for each treatment.

Sample Analysis and Statistical Procedures

Sample analysis: ATB amendment Ca, Mg, K, P, and metals were determined through nitric acid digestion using a Microwave Accelerated Reaction System (MARS) followed by AAS analysis. Amendment and foliage CNS were measured using a LECO induction furnace, with other foliage elements determined through dry ashing (400 °C) followed by HCl digestion and colorimetric analysis (P) or AAS analysis (all other elements).

Data analysis: All foliage concentration data were analyzed as repeated measures using SAS PROC MIXED (version 9.3, SAS Institute Inc.). Fixed factors and effects were treatment (control, low ATB rate, high ATB rate), time (days since treatment), and treatment-x-time interaction. Two blocking factors were used for the Latin square design at Site 1, and pre-treatment data were used as covariates at both sites. Normality of error terms was assessed for each variable using normal probability plot of residuals, and if violated, power transformations were used. Compound symmetry was the best covariance structure found and was used for all analyses. Significance of model terms and multiple means comparisons were assessed at $\alpha = 0.05$. Tree growth data data were assessed using Welch's ANOVA with Games-Howell comparison of means ($\alpha = 0.05$). Plant cover data were assessed using standard t-tests at $\alpha = 0.05$ and 0.10. Plots of means were generated using Minitab software version 17 (Minitab Inc.).

Due to differences in ATB chemical composition and application schedules, results from each trial site were treated as independent. Differences and similarities noted between trials are intended to highlight possible effects of ATB chemistry and/or site variables on treatment responses.

RESULTS

Foliage Chemistry and Tree Growth

Tree foliage Site 1: Compared to the control, ATB treatments led to significant increases in mean foliage Ca for white spruce at Site 1, with estimated concentration ratios ranging from 1.30-1.80 (high rate) and 1.31-1.90 (low rate) (Table 5.2 and Figure 5.2). In contrast, except for an initial pulse in high rate plots, there was little change in red spruce foliage Ca over the entire trial period (Table 5.2 and Figure 5.4). Mg concentrations increased in both species in the year of treatment, but then decreased in subsequent years (Table 5.2, Figures 5.2 and 5.4). Although not statistically significant, Mg concentrations in high rate plots were generally lower than controls by 2014 (white spruce ratio 0.94, red spruce ratio 0.84). Both species showed significant, rate dependent increases in K concentrations (Table 5.2, Figures 5.2 and 5.4) with treatment versus control K ratios ranging from 1.59-1.80 (high rate) and 1.41-1.50 (low rate) for white spruce, and 1.05-1.20 (high rate) and 0.98-1.13 (low rate) for red spruce. There were also consistent increases in P concentrations for both species, with white spruce showing statistically significant increases (Table 5.2, Figures 5.2 and 5.4).

White spruce showed an early significant increase in N (especially in high rate plots), but concentrations were back to control plot levels by 2014 (Table 5.2, Figure 5.3). Red spruce did not show any significant N response with either application rate (Table 5.2, Figure 5.5). Both species showed overall increases in foliage S, but results were neither significant nor consistent (Table 5.2, Figures 5.3 and 5.5). White spruce Zn concentrations were significantly greater in treated plots over all years, with treatment versus control ratios ranging from 1.35 to 1.64 (high rate) and 1.55-1.74 (low rate) (Table 5.2 and Figure 5.3). Zn concentrations were also generally higher in red spruce, but increases were not significant (Figure 5.5). In contrast, after an initial

increase, end-of-trial foliage Mn concentration ratios in treated versus control plots decreased from 1.26 to 0.73 and 1.15 to 0.88 (white spruce), and 1.03 to 0.71 and 0.97 to 0.82 (red spruce), with the largest decreases found in high rate plots (Table 5.2, Figures 5.3 and 5.5). Finally, mean foliage C concentrations did not change for either species over the entire trial period (Table 5.2).

Tree foliage Site 2: Site 2 did not contain red spruce ingrowth so only white spruce crop trees were assessed. Mean foliage Ca and K both showed significant increases, with K increases also being rate dependent – the same trend as Site 1 (Table 5.3, Figure 5.6). Treatment versus control Ca ratios ranged from 1.38-1.45 (high rate) and 1.23-1.67 (low rate), while K ratios ranged from 1.22-1.32 (high rate) and 1.13-1.14 (low rate). Foliage Mg and P showed non-significant increases in high rate plots in the year of treatment, but concentrations were at or below control plot levels by 2014 (Table 5.3, Figure 5.6). This trend was similar to that found at Site 1 for Mg, but not for P. Mean foliage N also showed an early non-significant increase after treatment, returning to control plot levels by 2014 (Table 5.3, Figure 5.7).

Both S and Zn had increased concentrations in treated plots, with Zn increases being statistically significant (Table 5.3, Figure 5.7). However, in both cases, treatment versus control concentration ratios were decreasing over time – the same general trend as all other elements except K (Table 5.3). Though not statistically significant, the same end-of-trial decreases in foliage Mn concentrations found at Site 1 were also found at Site 2 (Table 5.3, Figure 5.7), with concentration ratios dropping from 1.34 to 0.90 (high rate) and 1.23 to 0.90 (low rate) between 2013 and 2014. Finally, as with Site 1, mean foliage C concentrations were unaffected by both ATB applications (Table 5.3).

Raspberry foliage: Treatment related concentration changes were generally more pronounced for raspberry foliage than for tree foliage at both sites, with consistent and often

significant increases in Ca, K, and P for the entire study period (Tables 5.2 and 5.3, Figures 5.8 and 5.10). At Site 1, end-of-trial treatment versus control concentration ratios for Ca were 2.13 (high rate) and 2.02 (low rate), with K ratios of 1.58 and 1.49 and P ratios of 1.40 and 1.38 (Table 5.2). Ratios at Site 2 were not quite as high, but still greater than those associated with tree foliage (Table 5.3). There were also consistent and significant decreases in mean foliage Mn concentrations at both sites (Tables 5.2 and 5.3, Figures 5.9 and 5.11). Compared to the control, Mn concentrations decreased by as much as 72% at Site 1 and 48% at Site 2. Other element concentrations in raspberry foliage were either unaffected by ATB treatments (S and C), or inconsistently affected (Mg, N, and Zn). Mg response was mixed at Site 1, but showed significant decreases at Site 2 (Tables 5.2 and 5.3, Figures 5.8 and 5.10). In contrast, N concentrations showed significant decreases in treated plots at Site 1, but a significant increase in high rate plots at Site 2 (Tables 5.2 and 5.3, Figures 5.9 and 5.11). Zn concentrations generally increased at both sites, but response varied between application rates (Figures 5.9 and 5.11).

Tree growth: There were small, but statistically significant, increases in tree diameter increment for all treated plots versus controls at both sites (Table 5.4). Increases were variable in the first year of measurement, but cumulative 2-year measures were more consistent for both white and red spruce. Unlike diameter measures, there were no treatment related increases in height increment for either species at either site (Table 5.4).

Ground Vegetation Cover

Site 1: A total of 48 species were recorded in the 27 vegetation sub-plots established at Site 1. In terms of both species counts and diversity, there were no significant differences between pre- and post-treatment conditions approximately two years after treatment (Table 5.5).

With respect to abundance, there were 18 species with at least one possible t-test comparison for pre- and post-treatment percent cover. Four species showed significant decreases in percent cover at the 95% confidence level and six at the 90% confidence level (Table 5.6). However, mean percent cover change was low for many of these species (generally < 7%), and no species showed a significant response to both treatment rates. Two species, red raspberry and Canada goldenrod (*Solidago canadensis*), also showed a significant decrease in control plot cover, with a particularly large drop in mean raspberry cover (47% to 24%). This drop was, however, mainly associated with decreases in two of the nine control plots assessed (data not shown).

Site 2: A total of 40 species were recorded in the 27 vegetation sub-plots established at Site 2. In terms of both species counts and diversity, there were no significant differences between pre- and post-treatment conditions approximately one year after treatment (Table 5.7). With respect to abundance, there were 21 species with at least one possible t-test comparison for pre- and post-treatment percent cover. One species showed a significant decrease in percent cover at the 95% confidence and two at the 90% confidence level (Table 5.8). However, neither of these species showed a response to both treatment rates, and one also showed a decrease in control plot cover along with treated plot cover.

DISCUSSION

Foliage Chemistry and Tree Growth

There are no local data available on what are considered optimum nutrient concentrations in plantation white spruce foliage. However, Quesnel et al. (2006) estimated optimum Ca, Mg, K, P, N, and Mn foliage concentrations for boreal white spruce in Ontario and Quebec, Canada. Pre- and post-treatment foliage N at Sites 1 and 2 (range 12.3-15.4 mg g⁻¹) were in line with

optimum and maximum values reported by Quesnel et al. (2006) (range 12.3-14.0 mg g⁻¹), but other macronutrient concentrations (Ca, Mg, K, P) were generally lower than suggested minimum values (Table 5.9). The opposite was true for Mn concentrations which were generally higher than suggested maximum values. All this suggests that one or more nutrients (Ca, Mg, K, P) were limiting or out of balance with respect to N at both trial sites before ATB treatment.

White spruce foliage Ca, K, P, and Mn concentrations were generally improved by ATB applications (especially at Site 1), but Mg concentrations were not (Table 5.9). The lack of sustained Mg uptake in treated plots at both sites was likely due to a combination of low amendment input (Table 5.1), Mg leaching loss (Chapter 4), and cation antagonism (Ende and Evers 1997). Røsborg et al. (2006) also reported a decrease in foliage Mg in a limed Scots pine (*Pinus sylvestris*) stand in Norway which they attributed to Ca displacement and competition. In the case of Mn, decreased foliage concentrations were likely due to reduced availability after treatment-related increases in forest floor and surface soil pH (Chapter 3). This type of Mn response is commonly found in forest liming studies (e.g., Kreutzer 1995; Sikström 2002; Long et al. 2015). Mn results were even more pronounced for raspberry foliage (Tables 5.2 and 5.3).

With respect to Zn, Merilä and Derome (2008) found a significant positive relationship between current year foliage Zn and forest floor Zn for Norway spruce in Finland. This relationship could also exist for white spruce and is consistent with the greater Zn inputs and sustained foliage Zn concentrations found at Site 1 versus Site 2 (Table 5.1, Figures 5.3 and 5.7). Although foliage Zn increased with ATB treatments at both sites, the maximum concentrations found were still within the natural range reported by Watt et al. (2012) for boreal white spruce in northwestern Canada (21.5-76.3 mg·kg⁻¹).

Increases in Site 1 white spruce foliage Ca, K, and (to a lesser extent) P reflected increased availability of these nutrients in plantation soils after ATB treatment (Chapters 3 and 4), but red spruce only showed increases in foliage K and P. Smallidge et al. (1993) and Smallidge and Leopold (1997) discussed the variable responses of red spruce to liming in the northeastern US and attributed contrasting results (including in some cases no change in foliage Ca) to a range of possible causes such as differences in lime chemistry, soil chemistry, tree age, tree genetics, and treatment methodologies. In this study, red spruce at Site 1 came from natural ingrowth, and even though only visually healthy trees were sampled, they were not as large or as vigorous as planted white spruce. Base saturation at Site 1 was less than 10% before ATB treatments (Chapter 4), so it is also possible that Al toxicity had impacted fine-root production before treatment (Rengel 1992; Godbold et al. 2003). In addition, Al^{3+} can interfere with Ca^{2+} and Mg^{2+} uptake (but not K^+) by adsorbing onto cells in the root cortex and blocking normal uptake of these nutrients (Tomlinson 1990; Rengel 1992). It is therefore possible that a combination of competition and Al stress may have prevented an increase in Ca uptake by red spruce over the study period. If this was the case, treatment related increases in mineral soil base cation / Al ratios (Chapter 4) should eventually improve root function and Ca uptake by red spruce at this site. This will be examined in future plot assessments. Uptake of Mg would also have been impacted by the same factors noted for Ca, with the added burden of cation antagonism.

There are very few data available on what would be considered optimum or adequate nutrient concentrations in red spruce foliage, and no such data exist for Nova Scotia. However, a study conducted in the early 1970s in Maine, USA looked at benchmark red spruce foliage data in support of planned fertilization studies (Schomaker 1973). The author reported on current

foliage nutrient concentrations from mature red spruce (55-87 years) collected from three unfertilized sites in eastern and northern Maine over three years (1969-1971). Assuming the cumulative impacts of acid deposition were still relatively low during this time period, these data provide some indication of typical nutrient concentrations in red spruce foliage on average sites before significant base cation depletion. Reported N concentrations (mean 1.04%) were similar to those found for control plot red spruce in this study (1.17%), but reported base cation and P concentrations were about double those found in Site 1 control plot trees (Table 5.10). This supports the contention that Ca and Mg uptake by red spruce was restricted at this site, whereas foliage K and P concentrations in high rate plots appear to be moving in the right direction with respect to historic data.

Based on foliage results, the modest growth response to ATB treatments at both sites is not unexpected (Table 5.4). N levels in both white and red spruce were close to optimum or historic estimates (Tables 5.9 and 5.10), and there was little available N added with ATB treatments (Chapters 3 and 4). In addition, although foliage Ca, K, and P concentrations were variably enhanced by ATB applications, Mg concentrations were not, and Mg concentrations and/or imbalances may now be the main limiting factor for increased growth at these sites – at least in the near-term (see also Chapter 4). In their meta-analysis of forest ecosystem lime and wood ash trials, Reid and Watmough (2014) noted that time-since-treatment was the most important variable related to tree growth response, but that other nutrient limitations could also prevent growth responses in more acidic sites (despite increases in pH and Ca availability).

Ground Vegetation Cover

Along with red raspberry, which is a commonly found pioneer species on harvest sites in Nova Scotia, hair-cap moss (*Polytrichum commune* var. *perigoniale*) cover was unusually high in all plots, in the range of 41-67% before treatment at Site 1 and 25-58% at Site 2 (Tables 5.6 and 5.8). Hair cap moss is generally associated with open, disturbed sites (Ireland 1982), so enhanced coverage was likely a direct result of site preparation treatments conducted prior to planting at both sites. Indeed, surface impressions from the drum chopper used for site preparation could still be seen in some sections of Site 1 at the time of study.

Although no data are available from other forest related ATB studies, it has been shown that lime and wood ash amendments can have variable impacts on ground vegetation communities – sometimes affecting diversity through promotion of more N-demanding or pioneer species, or sometimes affecting abundance and/or vigour of already established species (e.g., Falkengren-Grerup et al. 1995; Dulière et al. 1999; Pitman 2006; Pabian et al. 2012). Vegetation communities can also be severely impacted when liming leads to increases in worm populations that drastically alter forest floor habitat conditions (e.g., Moore et al. 2015).

Despite significant treatment related increases in forest floor Ca and pH (Chapter 3), ground vegetation communities in this study were not appreciably altered by ATB treatments in the time periods assessed (Tables 5.5 to 5.8). This was likely due, in part, to insignificant treatment effects on available N as shown by both foliage (this Chapter) and soil data (Chapters 3 and 4), and to the already dominant presence of pioneer and/or disturbance related species in these juvenile plantations. However, ground cover health and vigour may have been enhanced in some cases, as evidenced by changes in red raspberry foliage chemistry (Figures 5.8 to 5.11).

This type of response was also noted by Pabian et al. (2012) in their study of liming impacts on deer forage.

CONCLUSIONS

Alkaline-treated biosolids (ATB) were surface applied at 7.5 t ha⁻¹ and 15 t ha⁻¹ (wet weight) on two 10-15-year old white spruce plantations in Nova Scotia, Canada. Analysis of current year foliage from planted white spruce and natural red spruce suggested pre-treatment N concentrations were adequate, but that Ca, Mg, K, and P concentrations were low at both sites. ATB treatments generally increased foliage Ca, K, and (to a lesser extent) P concentrations in white spruce in relation to treatment rate, but in red spruce only foliage K and P were enhanced. Lack of Ca uptake by red spruce may have been due to a combination of competition and Al stress on fine root development and function, but this could not be confirmed. Lack of Mg uptake by both species was likely due to low amendment inputs, Mg leaching loss, and cation antagonism. Foliage Zn increased in both species (especially white spruce), but concentrations were still within the normal range for this element. In contrast, Mn concentrations showed delayed decreases which were attributed to reduced availability after increases in forest floor and surface soil pH.

With respect to tree growth, ATB treatments led to modest, but consistent, increases in tree diameter increment after two growing seasons (especially in white spruce), but had no effect on height increment. Lack of significant N inputs and/or Mg deficiencies likely prevented more of an early treatment response.

Concentration trends in red raspberry foliage generally mirrored those found in white spruce, but to a greater degree. However, ATB treatments had no significant effect on ground

vegetation species abundance or diversity. This was attributed to minimal changes in available N from ATB treatments, and to the already current dominance of pioneer and disturbance species in these young plantation sites.

Vegetation uptake is vital to the success of any forest soil amendment program aimed at increasing or replenishing nutrient stores (Binkley 1986), especially on sites with limited cation exchange capacity (such as Site 2 in this study). Even in a plantation setting, it would be desirable for non-crop vegetation to take up nutrients rather than have them lost to leaching, as most of these nutrients will eventually be made available to crop trees when competing vegetation declines. Nutrient sustainability and plantation productivity can both be enhanced if amendment regimes provide necessary nutrients while keeping competing vegetation in check.

Results from this study, and related assessments of forest floor, mineral soil, and leachate chemistry, suggest that ATB could be a good source of Ca in Ca-limited plantation sites, while also enhancing K and P uptake. Furthermore, application of ATB in juvenile, free-to-grow plantations does not appear to affect ground vegetation diversity, and may even enhance the quality of some browse species. However, assuming adequate N supplies, possible increases in plantation productivity may be hindered by decreases in available Mg and/or base cation imbalances caused by ATB treatment. An additional Mg fertilizer, or possibly a mixture of dolomitic limestone and ATB, may be required on sites deficient in both Ca and Mg.

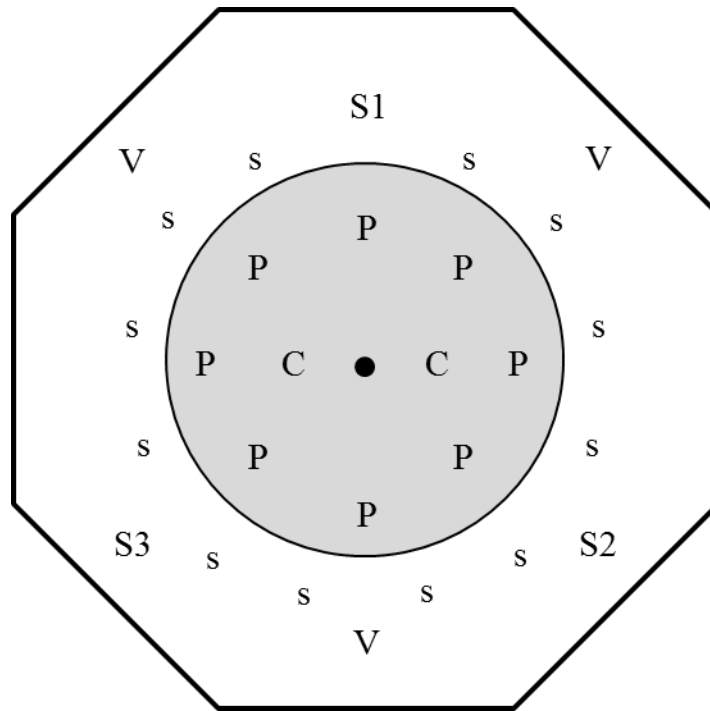


Figure 5.1. Schematic of a single field plot with systematic sample points based on a random initial bearing for Soil Pit 1 (S1). Octagon: ATB treatment area within 40 m x 40 m plot. Grey circle: tree growth assessment area (10.3 m radius). C = porous cup solution samplers (4 m from centre). P = paired PRSTM-probes (8 m from centre). S1-S3 = pre-treatment soil pit sampling sites (13 m from centre). s = post-treatment soil sampling sites (12 m from centre). V = ground vegetation monitoring sub-plots (13-14 m from centre).

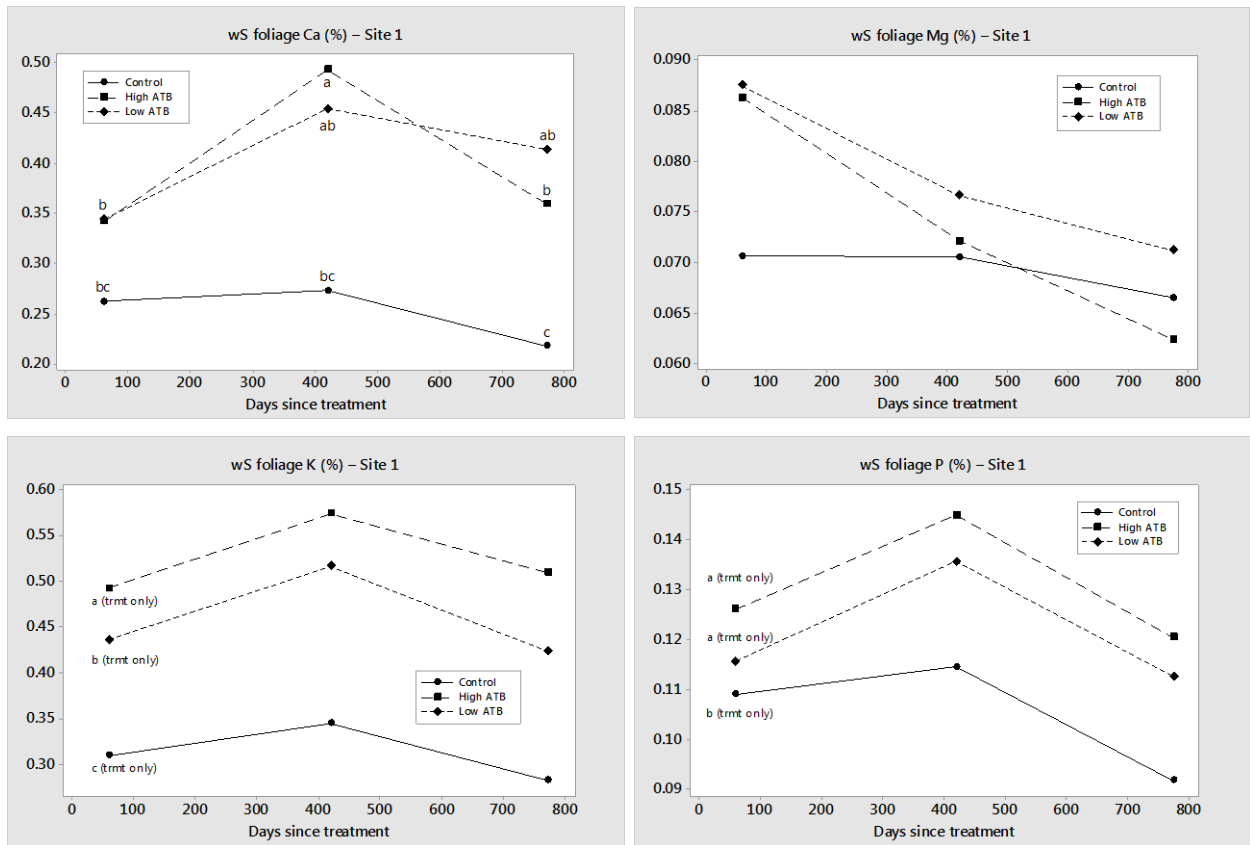


Figure 5.2. Mean foliage Ca, Mg, K, and P by treatment and time for Site 1 white spruce (wS). Values with different letters are statistically different at $p = 0.05$.

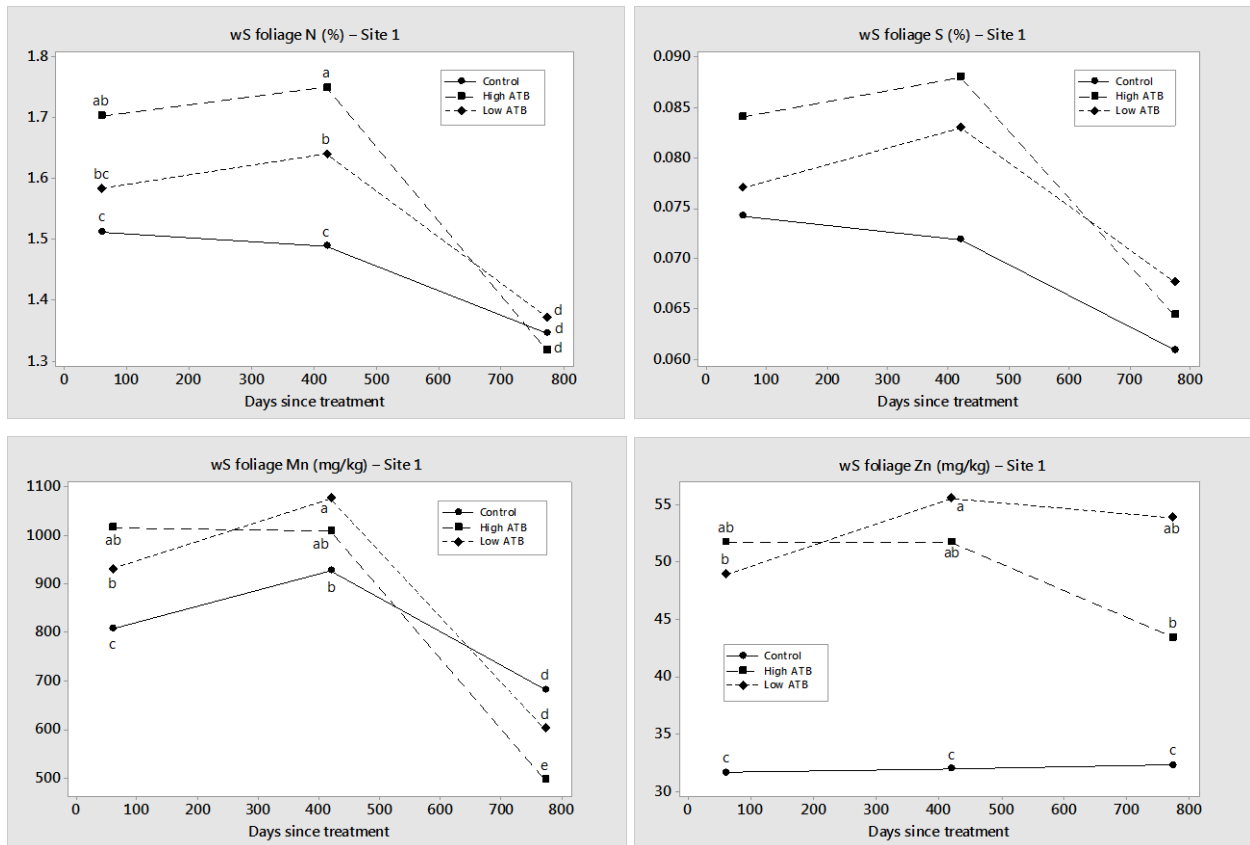


Figure 5.3. Mean foliage N, S, Mn, and Zn by treatment and time for Site 1 white spruce (wS). Values with different letters are statistically different at $p = 0.05$.

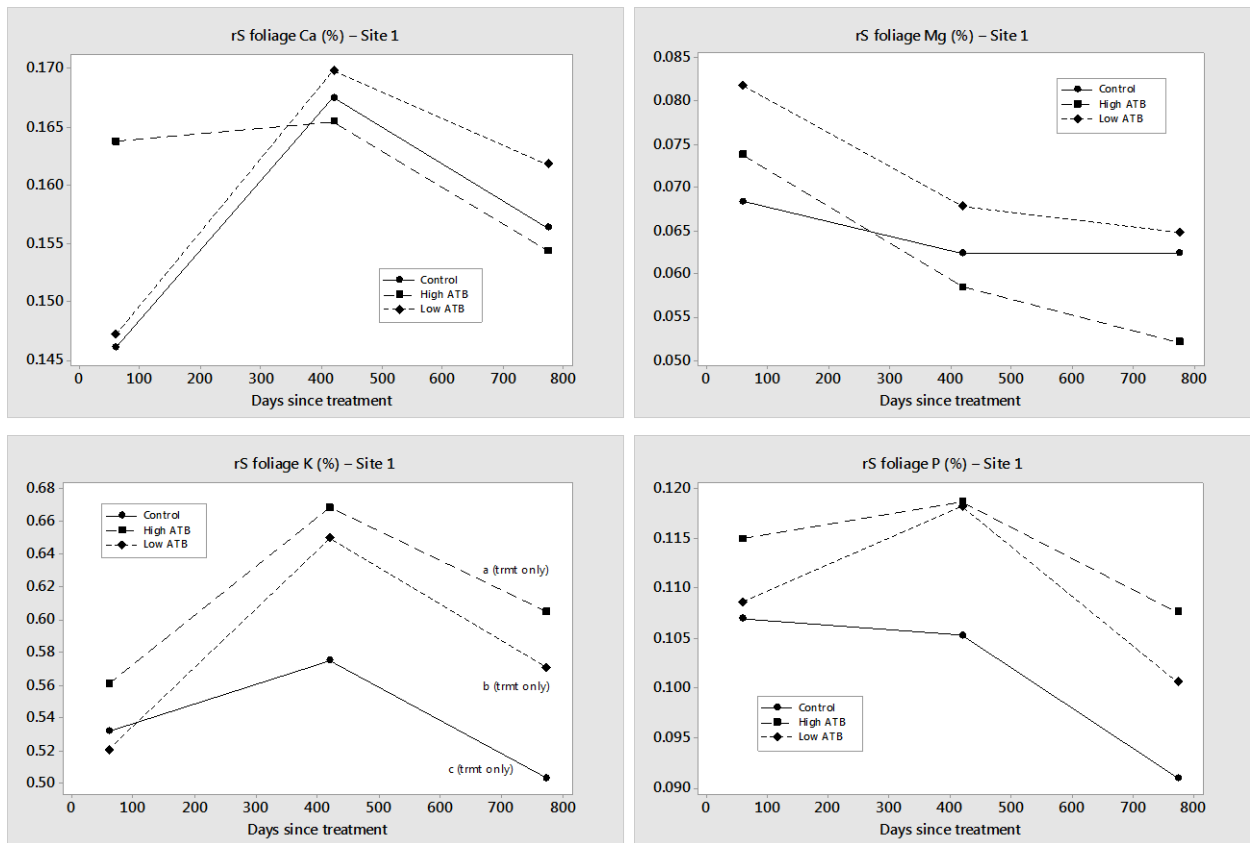


Figure 5.4. Mean foliage Ca, Mg, K, and P by treatment and time for Site 1 red spruce (rS). Values with different letters are statistically different at $p = 0.05$.

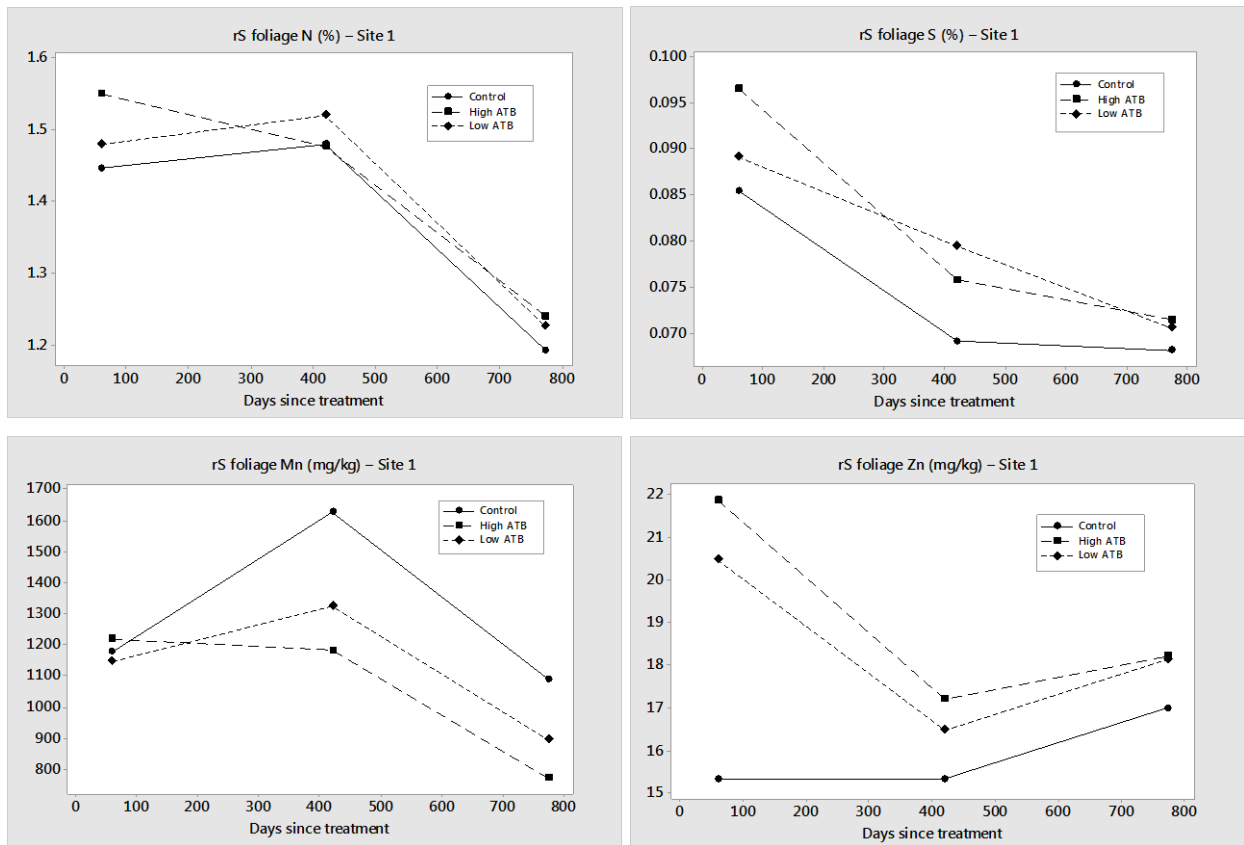


Figure 5.5. Mean foliage N, S, Mn, and Zn by treatment and time for Site 1 red spruce (rS). Values with different letters are statistically different at $p = 0.05$.

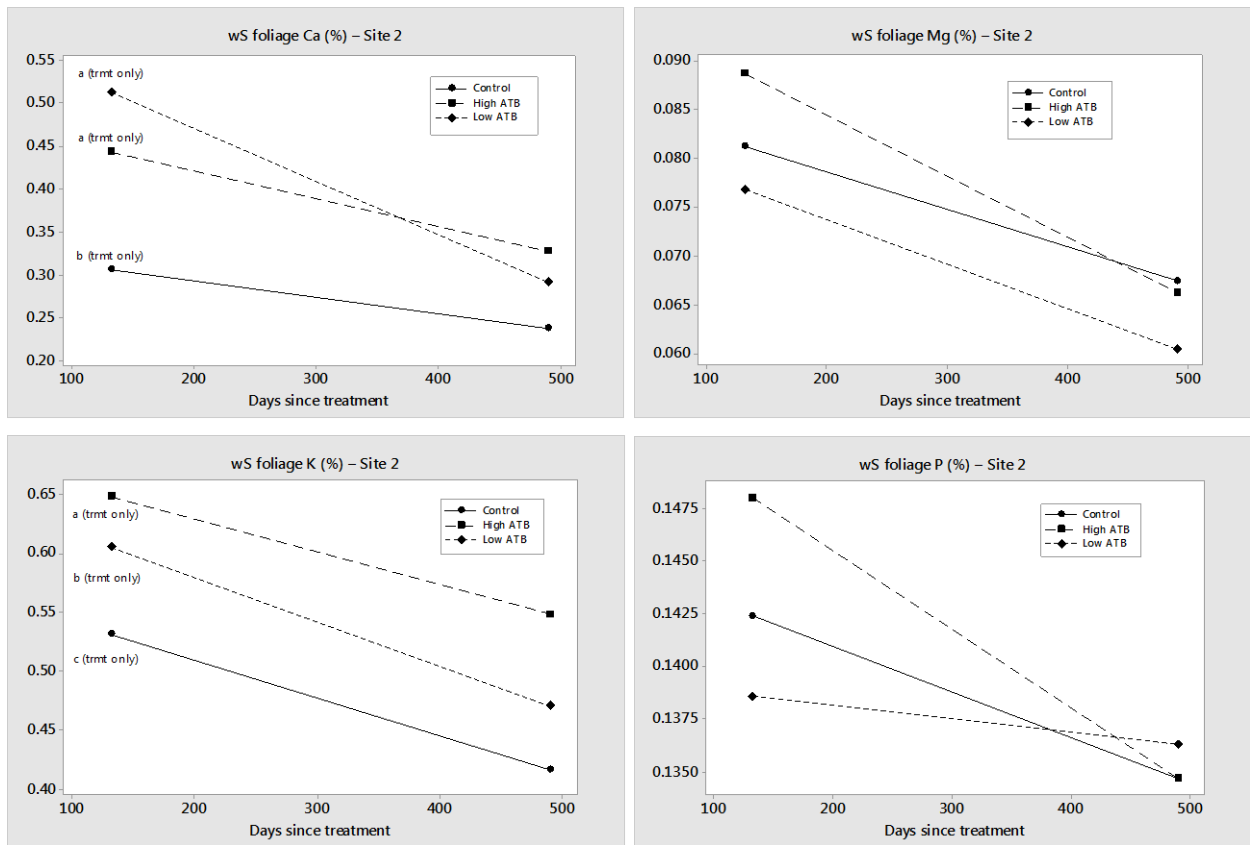


Figure 5.6. Mean foliage Ca, Mg, K, and P by treatment and time for Site 2 white spruce (wS). Values with different letters are statistically different at $p = 0.05$.

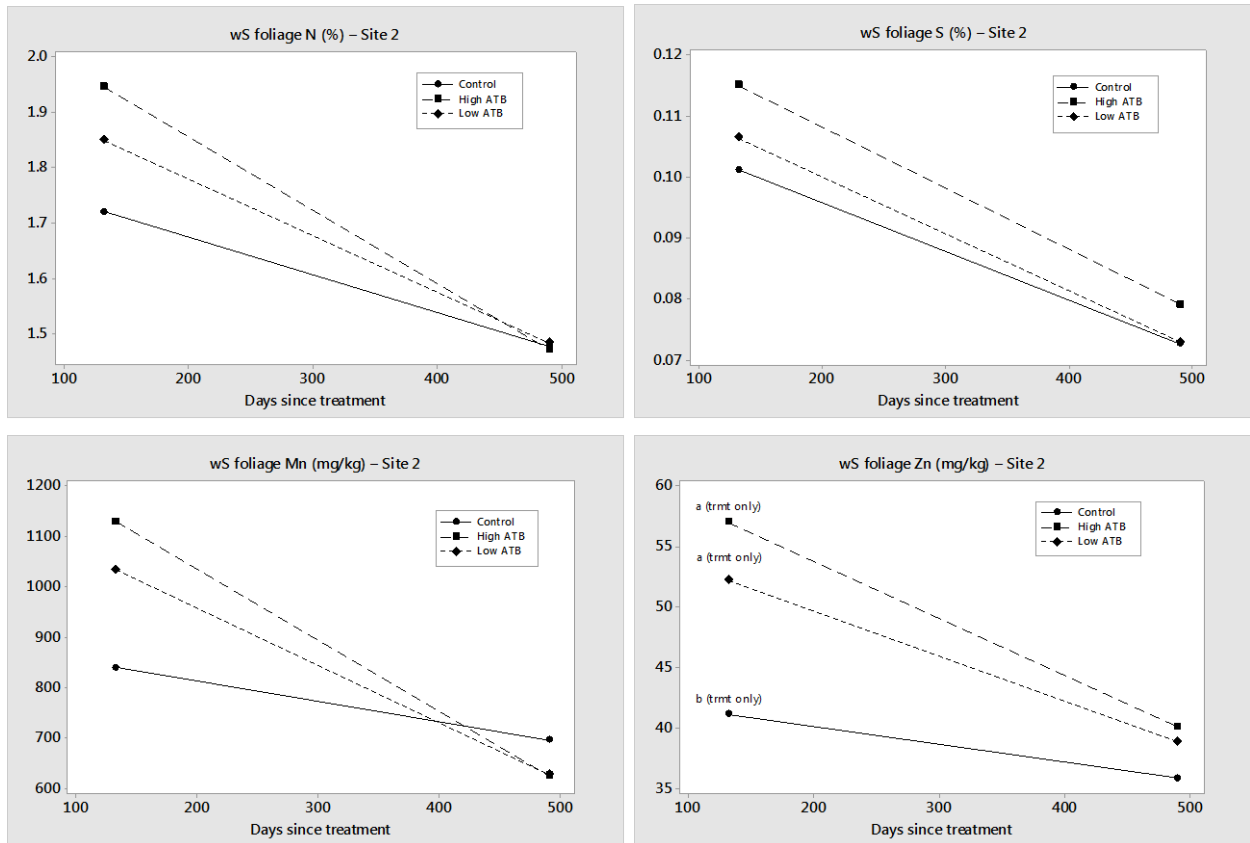


Figure 5.7. Mean foliage N, S, Mn, and Zn by treatment and time for Site 2 white spruce (wS). Values with different letters are statistically different at $p = 0.05$.

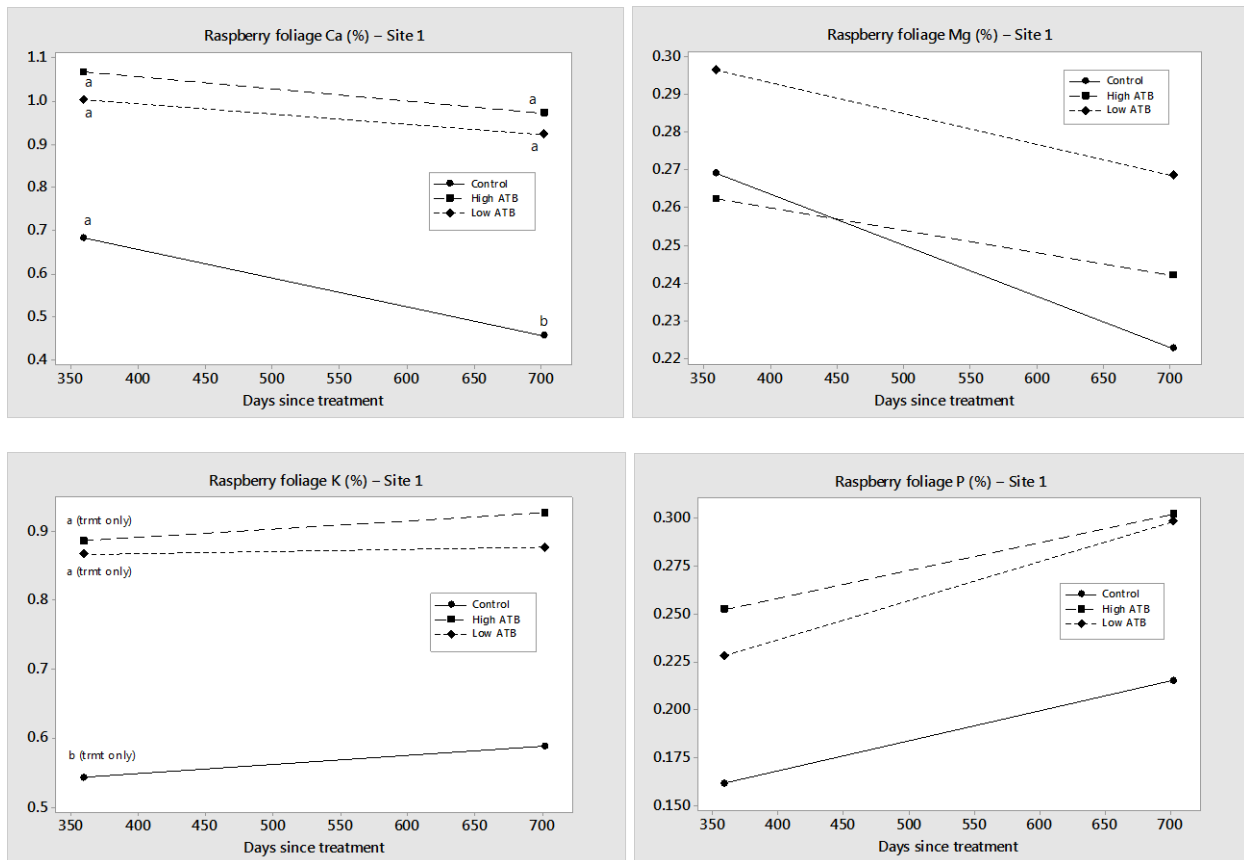


Figure 5.8. Mean foliage Ca, Mg, K, and P by treatment and time for Site 1 raspberry. Values with different letters are statistically different at $p = 0.05$.

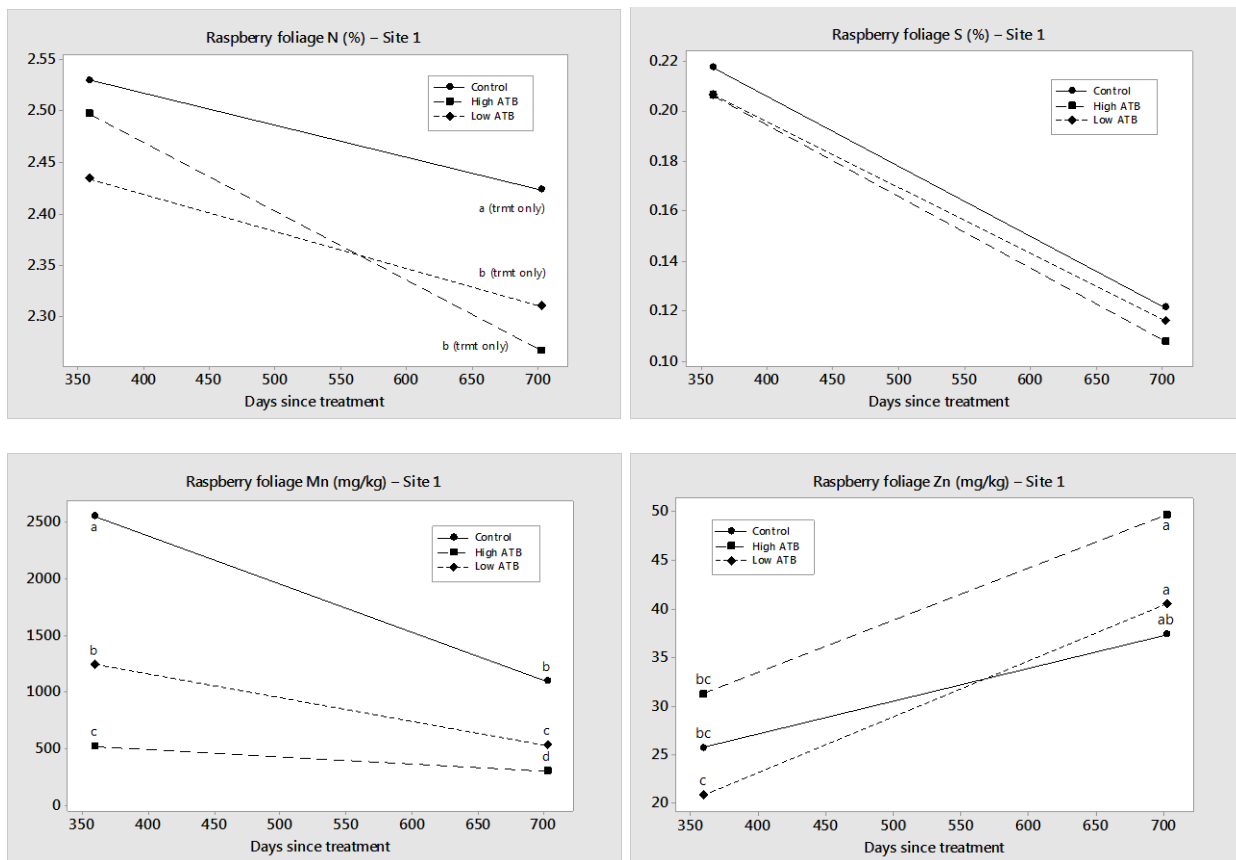


Figure 5.9. Mean foliage N, S, Mn, and Zn by treatment and time for Site 1 raspberry. Values with different letters are statistically different at $p = 0.05$.

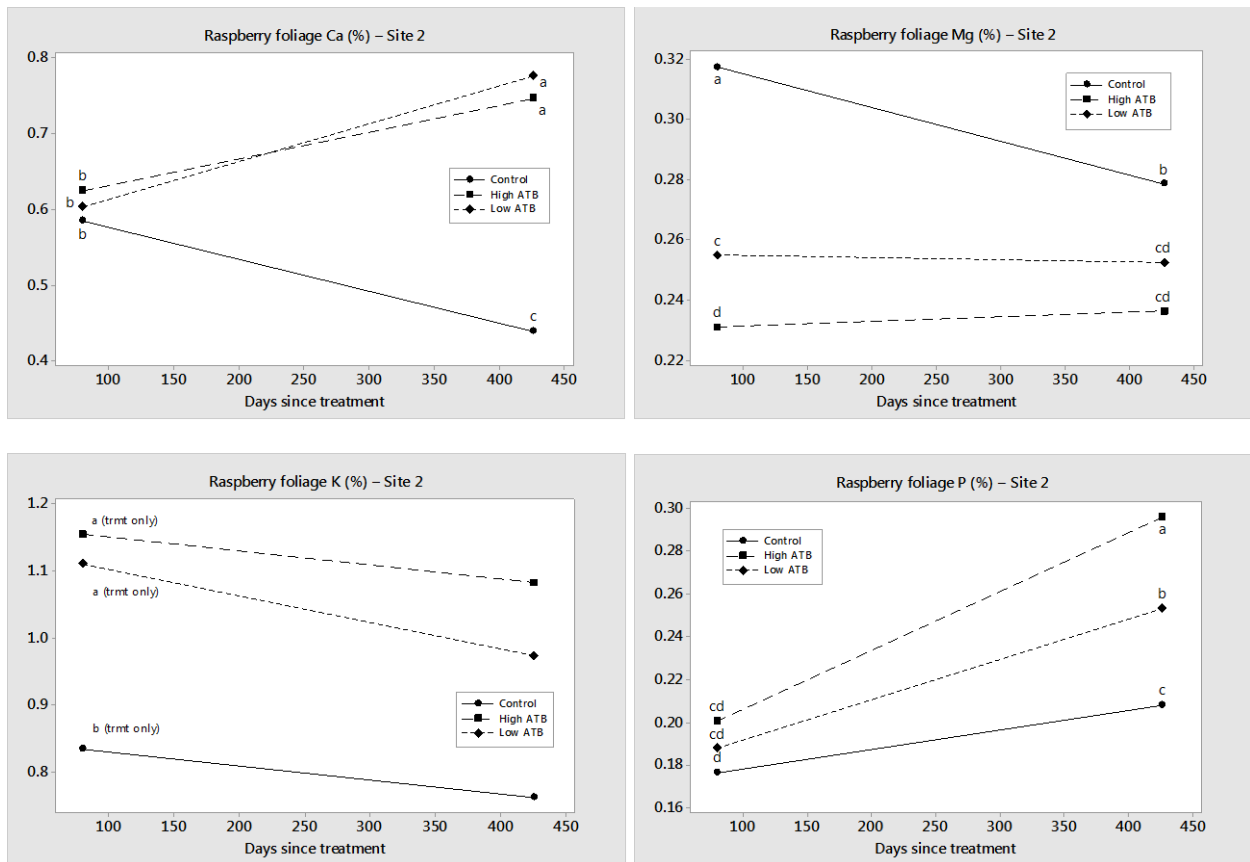


Figure 5.10. Mean foliage Ca, Mg, K, and P by treatment and time for Site 2 raspberry. Values with different letters are statistically different at $p = 0.05$.

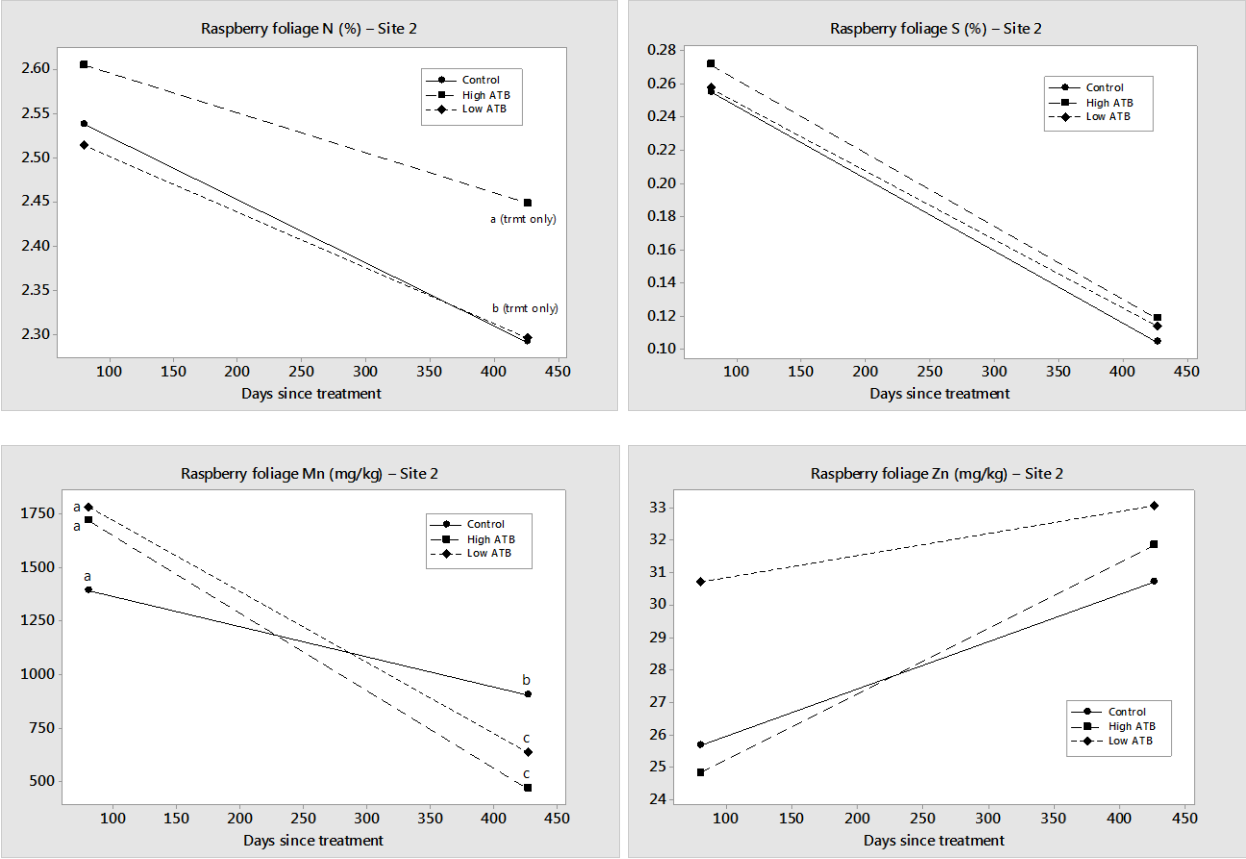


Figure 5.11. Mean foliage N, S, Mn, and Zn by treatment and time for Site 2 raspberry. Values with different letters are statistically different at $p = 0.05$.

Table 5.1. Summary of ATB chemistry and estimated application rates for Site 1 and Site 2. All values were derived from on-site sampling.

Site 1								
Statistic	Ca	Mg	K	N	S	P	Mn	Zn
(n=6) (% wt) (mg kg ⁻¹)	
Mean	20.6	0.41	1.72	0.85	0.13	0.52	218	494
Stdev	1.16	0.03	0.04	0.17	0.06	0.04	15	58
% CV	6	7	2	20	44	7	7	12
Estimated element application (kg ha⁻¹)								
Trmt	Ca	Mg	K	N	S	P	Mn	Zn
High	1,914	38	160	79	12	49	2.0	4.6
Low	957	19	80	39	6	24	1.0	2.3

Site 2								
Statistic	Ca	Mg	K	N	S	P	Mn	Zn
(n=4) (% wt) (mg kg ⁻¹)	
Mean	20.1	0.34	1.65	0.87	0.08	0.58	226	180
Stdev	2.38	0.04	0.32	0.04	0.01	0.05	38	14
% CV	12	10	19	5	13	9	17	8
Estimated element application (kg ha⁻¹)								
Trmt	Ca	Mg	K	N	S	P	Mn	Zn
High	1,870	32	153	81	7	54	2.1	1.7
Low	935	16	77	41	4	27	1.1	0.8

Table 5.2. Estimated foliage nutrient concentration ratios in treated plots versus controls at Site 1 – from repeated measures analysis.
 (wS = white spruce, rS = red spruce, Rasp = red raspberry, H = high ATB rate, L = low ATB rate).

Species	Days	Trmt	Ca	Mg	K	P	N	S	C	Mn	Zn
wS	60	H	1.30	1.22	1.59	1.16	1.13	1.13	0.99	1.26	1.64
		L	1.31	1.24	1.41	1.06	1.05	1.04	1.00	1.15	1.55
	420	H	1.80	1.02	1.66	1.27	1.18	1.22	0.99	1.09	1.62
		L	1.66	1.09	1.50	1.18	1.10	1.15	0.99	1.16	1.74
	773	H	1.65	0.94	1.80	1.31	0.98	1.06	nd	0.73	1.35
		L	1.90	1.07	1.50	1.23	1.02	1.11	nd	0.88	1.67
rS	60	H	1.12	1.08	1.05	1.07	1.07	1.13	1.00	1.03	1.43
		L	1.01	1.20	0.98	1.02	1.02	1.04	1.03	0.97	1.34
	420	H	0.99	0.94	1.16	1.13	1.00	1.10	1.01	0.73	1.12
		L	1.01	1.09	1.13	1.12	1.03	1.15	1.00	0.81	1.07
	773	H	0.99	0.84	1.20	1.18	1.04	1.05	nd	0.71	1.07
		L	1.03	1.04	1.13	1.11	1.03	1.04	nd	0.82	1.07
Rasp	359	H	1.56	0.97	1.63	1.56	0.99	0.95	1.00	0.21	1.21
		L	1.47	1.10	1.60	1.41	0.96	0.95	1.01	0.49	0.81
	702	H	2.13	1.09	1.58	1.40	0.94	0.89	1.00	0.28	1.33
		L	2.02	1.21	1.49	1.38	0.95	0.96	0.99	0.49	1.08

Table 5.3. Estimated foliage nutrient concentration ratios in treated plots versus controls at Site 2 – from repeated measures analysis.
 (wS = white spruce, Rasp = red raspberry, H = high ATB rate, L = low ATB rate).

Species	Days	Trmt	Ca	Mg	K	P	N	S	C	Mn	Zn
wS	132	H	1.45	1.09	1.22	1.04	1.13	1.14	0.99	1.34	1.39
		L	1.67	0.95	1.14	0.97	1.07	1.05	0.99	1.23	1.27
	490	H	1.38	0.98	1.32	1.00	1.00	1.09	1.00	0.90	1.12
		L	1.23	0.90	1.13	1.01	1.00	1.00	1.00	0.90	1.08
Rasp	80	H	1.07	0.73	1.38	1.14	1.03	1.07	0.99	1.23	0.97
		L	1.03	0.80	1.33	1.06	0.99	1.01	0.97	1.28	1.20
	426	H	1.70	0.85	1.42	1.42	1.07	1.14	0.98	0.52	1.04
		L	1.77	0.91	1.28	1.22	1.00	1.09	0.99	0.70	1.08

Table 5.4. Cumulative dbh and height increments for trees at Site 1 and Site 2 by treatment and year (wS = white spruce, rS = red spruce, H = high ATB rate, L = low ATB rate, C = control).

Site 1		dbh increment (cm)					Height increment (m)				
		2013		2014			2013		2014		
Species	Trmt	n	Mean		Mean		n	Mean		Mean	
wS	H	63	1.27	a	2.70	a	63	0.36	a	0.75	a
	L	53	1.15	ab	2.50	a	53	0.35	a	0.73	a
	C	46	1.06	b	2.30	b	45	0.37	a	0.72	a
rS	H	82	1.07	a	2.30	a	81	0.41	a	0.77	a
	L	85	1.00	b	2.20	a	81	0.43	a	0.76	a
	C	82	0.94	c	2.00	b	84	0.42	a	0.75	a

Site 2		dbh increment (cm)					Height increment (m)				
		2013		2014			2013		2014		
Species	Trmt	n	Mean		Mean		n	Mean		Mean	
wS	H	150	1.47	a	2.93	a	150	0.52	ab	1.00	a
	L	150	1.45	a	2.80	a	146	0.54	a	0.99	a
	C	121	1.35	b	2.62	b	121	0.49	b	0.95	a

Table 5.5. Species counts and calculated Shannon diversity index values for vegetation ground plots before (2012) and after (2014) ATB treatment at Site 1.

Species Count						Shannon Diversity Index					
Control		Low ATB		High ATB		Control		Low ATB		High ATB	
2012	2014	2012	2014	2012	2014	2012	2014	2012	2014	2012	2014
10	8	7	12	9	7	1.41	1.19	1.71	2.00	1.16	1.01
7	5	8	7	7	8	1.07	0.82	1.48	1.35	1.29	1.48
4	2	9	12	8	10	0.45	0.69	1.77	1.58	1.42	1.26
10	10	12	10	11	13	1.12	1.31	1.79	1.88	1.80	1.47
7	7	10	9	9	8	1.05	1.65	1.07	0.94	0.79	0.66
8	8	8	9	8	9	1.25	1.34	1.26	1.31	0.88	1.09
12	10	6	8	11	8	1.88	1.16	1.25	1.26	1.24	1.19
6	9	5	7	10	8	0.57	0.67	0.67	0.51	1.54	1.41
11	15	6	8	9	10	1.24	1.02	0.88	0.54	1.40	1.25
Mean		Mean		Mean		Mean		Mean		Mean	
8.3	8.2	7.9	9.1	9.1	9.0	1.12	1.09	1.32	1.26	1.28	1.20
Paired t-test		Paired t-test		Paired t-test		Paired t-test		Paired t-test		Paired t-test	
0.89		0.14		0.87		0.87		0.39		0.23	

Table 5.6. Mean percent cover for all Site 1 ground vegetation species with at least one possible t-test comparison for percent cover before (2012) and after (2014) ATB treatment. Results with at least 90% confidence have been shaded.

Species	Control				Low ATB				High ATB				Paired t-test		
	2012		2014		2012		2014		2012		2014		C	L	H
	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev			
<i>Agrostis spp.</i>	0.1	-	0	-	1	-	1	-	0.8	0.5	0.6	0.5	-	-	0.39
<i>Carex brunnescens</i>	13	18	8	11	5	7	0.6	0.6	0.1	0.0	0.1	0.1	0.50	0.50	0.50
<i>Carex communis</i>	5	-	2	-	7	4	3	2	2	2	1	1	-	0.00	0.39
<i>Carex debilis</i>	15	0	9	1	13	4	8	10	22	25	21	27	0.11	0.50	0.50
<i>Cladina rangiferina</i>	-	-	0.1	-	9	8	7	5	0.1	-	0.1	0.1	-	0.50	-
<i>Cladonia cristatella</i>	1	1	0.4	0.5	3	2	1	1	0.6	0.6	0.0	0.1	0.50	0.05	0.44
<i>Cornus canadensis</i>	-	-	-	-	34	30	35	18	69	20	61	17	-	0.87	0.67
<i>Danthonia spicata</i>	9	9	2	3	28	13	20	10	23	4	13	17	0.17	0.04	0.50
<i>Dicranum spp.</i>	2	2	3	1	18	18	7	11	0.6	0.6	2	2	0.37	0.50	0.22
<i>Euthamia graminifolia</i>	4	2	0.8	0.5	3	2	2	2	3	2	1	1	0.19	0.20	0.20
<i>Hieracium spp.</i>	1	0.8	0.5	1	4	4	3	5	4	4	0.5	1	0.15	0.25	0.14
<i>Maianthemum canadense</i>	2	1	2	1	0.6	0.6	0.3	0.4	3	1	1	1	0.96	0.37	0.09
<i>Polytrichum commune</i>	41	32	38	31	67	25	58	33	45	28	44	26	0.61	0.88	0.66
<i>Rubus allegheniensis</i>	-	-	-	-	-	-	-	-	3	1	0	0	-	-	0.20
<i>Rubus idaeus</i>	47	29	24	25	11	7	11	11	16	11	11	10	0.06	0.97	0.28
<i>Solidago canadensis</i>	7	6	0.8	1	8	6	6	6	11	8	7	6	0.03	0.07	0.22
<i>Trientalis borealis</i>	1	1	0.9	1	3	4	3	4	2	1	2	1	0.34	1.00	0.65
<i>Veronica officinalis</i>	1	-	0	-	1	-	0.1	0.1	1	0.5	1	3	-	-	0.83

Table 5.7. Species counts and calculated Shannon diversity index values for vegetation ground plots before (2012) and after (2014) ATB treatment at Site 2.

Species Count						Shannon Diversity Index					
Control		Low ATB		High ATB		Control		Low ATB		High ATB	
2012	2014	2012	2014	2012	2014	2012	2014	2012	2014	2012	2014
9	11	9	8	8	8	1.32	1.19	1.86	1.65	1.44	1.02
5	7	6	7	7	7	0.89	1.20	1.34	1.46	0.67	0.57
9	7	9	10	7	8	1.49	1.39	1.54	1.36	1.68	1.60
8	9	7	8	9	6	1.62	1.83	0.83	0.90	1.61	1.19
8	9	9	9	9	8	1.90	2.05	1.99	1.91	1.05	1.04
7	11	9	11	6	7	1.62	1.71	1.11	1.83	0.82	1.30
8	6	7	6	8	7	1.59	1.19	0.33	0.40	1.61	1.39
9	8	7	9	5	5	1.17	1.04	0.40	0.69	1.19	0.96
9	7	12	9	7	6	0.97	1.13	1.20	0.86	1.19	0.76
Mean		Mean		Mean		Mean		Mean		Mean	
8.0	8.3	8.3	8.6	7.3	6.9	1.40	1.41	1.18	1.23	1.25	1.09
Paired t-test		Paired t-test		Paired t-test		Paired t-test		Paired t-test		Paired t-test	
0.66		0.70		0.31		0.83		0.64		0.13	

Table 5.8. Mean percent cover for all Site 2 ground vegetation species with at least one possible t-test comparison for percent cover before (2012) and after (2014) ATB treatment. Results with at least 90% confidence have been shaded.

Species	Control				Low ATB				High ATB				Paired t-test		
	2012		2014		2012		2014		2012		2014		C	L	H
	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev			
<i>Aster acuminatus</i>	12	12	4	2	13	11	14	14	22	14	16	21	0.50	0.74	0.62
<i>Betula allegheniensis</i>	-	-	-	-	12	10	25	21	-	-	-	-	-	0.17	-
<i>Carex brunnescens</i>	14	13	7	10	4	1	4	2	4	4	2	2	0.07	0.42	0.08
<i>Carex communis</i>	8	9	12	19	7	7	7	9	12	21	12	21	0.64	1.00	0.37
<i>Cladonia cristatella</i>	3	5	1	2	0.1	-	0.1	0.1	2	2	0.2	0.3	0.44	-	0.24
<i>Danthonia spicata</i>	3	-	17	19	6	6	3	1	20	-	30	-	-	0.58	-
<i>Dicranum spp.</i>	4	4	4	4	1	1	2	2	1	1	1	1	0.37	0.65	0.39
<i>Epilobium angustifolium</i>	14	12	9	6	7	8	6	6	12	5	6	6	0.33	0.41	0.03
<i>Euthamia graminifolia</i>	4	-	2	-	1	-	0	-	7	4	3	0.0	-	-	0.41
<i>Hieracium spp.</i>	5	0	4	3	2	1	1	0.0	2	2	5	0.0	0.87	0.50	0.25
<i>Maianthemum canadense</i>	2	1	1	1	1	1	1	1	1	0	1	1	0.50	0.91	0.97
<i>Osmunda claytoniana</i>	-	-	-	-	1	0	2	3	-	-	-	-	-	0.70	-
<i>Plurozium schreberi</i>	14	16	10	7	1	1	4	3	6	8	7	5	0.59	0.26	0.80
<i>Polytrichum commune</i>	25	32	20	20	58	26	54	32	46	28	45	29	0.46	0.36	0.77
<i>Ribes triste</i>	-	-	5	-	8	4	9	2	-	-	-	-	-	0.50	-
<i>Rubus allegheniensis</i>	3	-	-	-	31	51	33	54	-	-	-	-	-	0.50	-
<i>Rubus hispidus</i>	19	29	24	41	10	12	8	7	35	7	21	13	0.32	0.79	0.18
<i>Rubus idaeus</i>	17	24	27	28	21	21	9	9	18	14	26	29	0.26	0.12	0.26
<i>Sambucus racemosa</i>	5	-	2	2	5	-	6	6	5	1	3	4	-	-	0.80
<i>Solidago canadensis</i>	25	23	5	5	-	-	1	0.4	5	-	0.3	0.4	0.25	-	-
<i>Viola spp.</i>	1	1	1	1	-	-	0.1	-	10	14	10	14	0.47	-	0.50

Table 5.9. Mean nutrient concentrations (mg g⁻¹) in current year white spruce foliage before treatment (2012) and at end-of-trial (2014) for Site 1 and Site 2 along with optimal concentrations for boreal white spruce reported by Quesnel et al. (2006). H = high ATB rate, L = low ATB rate, C = control.

Site 1							
Date	Trmt	Ca	Mg	K	P	N	Mn
2012	H	2.4	0.6	2.4	1.0	12.6	0.56
(Pre)	L	1.8	0.5	2.6	1.0	12.3	0.52
	C	2.3	0.6	2.7	1.1	13.4	0.62
2014	H	3.6	0.6	5.1	1.2	13.2	0.50
(End)	L	4.1	0.7	4.2	1.1	13.7	0.60
	C	2.3	0.7	2.8	0.9	13.5	0.68
Site 2							
Date	Trmt	Ca	Mg	K	P	N	Mn
2012	H	2.2	0.6	3.3	1.2	14.4	0.61
(Pre)	L	2.3	0.6	3.4	1.2	15.4	0.57
	C	2.3	0.6	3.4	1.1	13.6	0.59
2014	H	3.3	0.7	5.5	1.4	14.7	0.63
(End)	L	2.9	0.6	4.7	1.4	14.8	0.63
	C	2.4	0.7	4.2	1.4	14.8	0.70
Optimum		6.5	1.0	7.3	1.9	12.3	0.39
Minimum		4.3	nd	5.5	1.6	10.6	0.30
Maximum		8.6	1.3	9.2	2.3	14.0	0.50

Table 5.10. Mean nutrient concentrations in current year red spruce foliage before treatment (2012) and at end-of-trial (2014) for Site 1 along with reference concentrations found for mature red spruce in Maine, USA (Schomaker 1973). H = high ATB rate, L = low ATB rate, C = control.

Date	Trmt	Ca (%)	Mg (%)	K (%)	P (%)	N (%)
2012	H	0.129	0.057	0.437	0.096	1.23
(Pre)	L	0.134	0.060	0.445	0.103	1.22
	C	0.124	0.058	0.393	0.090	1.15
2014	H	0.154	0.052	0.605	0.108	1.24
(End)	L	0.162	0.065	0.571	0.101	1.23
	C	0.156	0.062	0.503	0.091	1.17
Data from Maine, USA						
	Mean	0.346	0.115	0.847	0.220	1.04
	Stdev	0.114	0.025	0.205	0.039	0.09

CHAPTER 6

ELEMENT FLUXES IN TWO FOREST SOILS AMENDED WITH LIME, FLY ASH, AND ALKALINE-TREATED BIOSOLIDS UNDER CONTROLLED CONDITIONS

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ABSTRACT

Two forest soil B-horizons were treated with alkaline-treated biosolids (ATB), powdered lime, and fly ash under controlled conditions to compare pH impacts and ion diffusion fluxes over a 10-week period. Soil pH increased significantly in all treated soils versus the control, with lime providing the greatest increase followed by ATB and fly ash. ATB was the most effective in providing available Ca^{2+} , but least effective in providing available Mg^{2+} , where lime was most effective. K^+ and SO_4^{2-} -S availability was highest in fly ash due to increased inputs and the high electrical conductivity of this amendment. Total N (NO_3^- -N + NH_4^+ -N) availability increased in ATB treated soils, stayed the same in lime treated soils, and decreased in ash treated soils. This was related to higher N content in ATB versus lime and the high C:N ratio of fly ash which led to increased N immobilization. PO_4^{3-} -P availability was low in all treated soils, but slightly

enhanced in ATB treated soils. Fe^{3+} and Al^{3+} fluxes were not greatly affected by any treatment, but trace metal (Mn^{2+} , Cd^{2+} , Cu^{2+} , Pb^{2+} , Zn^{2+}) fluxes generally decreased over time with increasing pH. Enhanced organic retention of metals was likely also a factor in ash treated soils. Results suggest ATB is equally or more effective at providing Ca as lime and fly ash, but less effective at providing Mg due to low inputs and cation competition. This is consistent with results found in ATB field trials and suggests this amendment could be a good source of Ca in Ca-limited soils as long as potential impacts on other base cations (especially Mg) are also considered and addressed.

KEYWORDS

Alkaline-Treated Biosolids; Lime, Fly Ash, Forest Soil, Ion Fluxes

INTRODUCTION

Northeastern North American forests have been impacted by decades of acid deposition resulting in variable loss of nutrient base cations [calcium (Ca), magnesium (Mg), potassium (K)] in many affected soils (Watmough and Ouimet 2005; Lawrence et al. 2012). The impact on Ca has been of particular concern due to: (i) its documented widespread loss (e.g., Likens et al. 1998; Watmough and Dillon 2003; Bedison and Johnson 2010), (ii) its importance in overall forest ecosystem health (e.g., Schaberg et al. 2001; Houle et al. 2006), (iii) its importance in commercial tree productivity and health (e.g., DeHayes et al. 1999; Huggett et al. 2007), and (iv) its significant potential for added loss through commercial harvesting (e.g., Federer et al. 1989; Adams et al. 2000).

Even though the potential impacts of acid deposition on northeastern forest soils have been well documented, there has been little use of soil amendments to offset the loss of base cations in affected soils (Moore et al. 2015). In contrast, lime has been used in some European countries since the 1980s to mitigate the impacts of acidic deposition (e.g., Tomlinson 1990; Nilsson et al. 2001; Meiwes et al. 2002), with wood ash also being used in Nordic countries to mitigate the impacts of acid deposition and biomass harvesting on long-term site productivity (e.g., Levin and Eriksson 2010). Use of wood ash in Canada is also being investigated as a potential soil amendment to offset nutrient losses. In a recent trial in Ontario, Pugliese et al. (2014) found ash amendments to be of overall benefit with little negative impact on soil chemical or biological processes. However, they also indicated that ash properties (and therefore appropriate application rates) were variable and dependent on source supply.

Despite the potential for lime or wood ash amendments to offset soil acidity and base cation loss, there can be negative impacts if these amendments are improperly used. For example, when application rates are too high: (i) microbial, meso-fauna, and ground vegetation communities can be significantly altered; (ii) organic matter decomposition rates can be greatly accelerated; and (iii) nitrate (NO_3^-) production can be increased beyond ecosystem demands leading to increased leaching (Kreutzer 1995; Pitman 2006). As is usually the case in forestry, these risks are soil and/or site-type specific. In addition, treatment responses can be highly variable, as recently discussed by Reid and Watmough (2014) in their meta-analysis of liming and wood ash treatment effects. These authors found that, in general, soil pH and foliar Ca concentrations were more responsive to liming treatments, whereas tree growth and percent base saturation were more responsive to ash treatments. However, there were also a significant

number of studies assessed where neither treatment resulted in any significant response, or where response varied by site conditions.

Papermill sludge and municipal biosolids (sewage sludge) have also been used as forest soil amendments in some jurisdictions (Matysik et al. 2001). Papermill sludge can be a significant source of base cations (e.g., Evanylo et al. 2004), but its potential use is limited by source location and the type of pulping process used which affects sludge chemistry (Scott and Smith 1995). In addition, this material often has a very high C:N ratio which can negatively impact post-treatment decomposition and N availability (Matysik et al. 2001). In their most common form, municipal biosolids are mainly a source of N and phosphorous (P) (Cogger et al. 2006), unless they have been alkaline-treated in which case they are also considered a liming amendment (USEPA 2000). Although alkaline-treated biosolids (ATB) are routinely used in agriculture, their use in northeastern forests has been minimal to date (Banaitis et al. 2009). Appropriate use of ATB could, however, offset nutrient deficiencies caused by acid deposition and forest harvesting, while also providing another end-use for this waste-stream product.

A field trial was established in Nova Scotia, Canada to study the dynamics of surface applied ATB on white spruce plantations (Chapters 3-5). As part of this study, a complementary greenhouse trial was also set up to assess pH impacts and ion flux rates in ATB treated mineral soil, and to compare results with those found using more traditional lime and wood ash amendments. This chapter describes and discusses results from this trial.

METHODS

Treatments: Two different forest soil B-horizons were used for this study. Soil 1 was a shaly, loam soil derived from slate dominated glacial till and Soil 2 a gravelly/cobbly, sandy

loam soil derived from granitic glacial till. Soils were sourced from untreated areas within spruce plantation sites used for related ATB field trials (Chapter 3). A summary of soil attributes based on field trial sampling is shown in Table 6.1. Approximately 200 L of each soil was collected and coarsely sieved (6.4 mm) to remove larger-sized gravel and cobbles. Each sieved soil was mixed with a shovel in a large wooden box lined with tarp, then sequentially divided into seven smaller containers (one for each planned treatment) and mixed again to ensure homogeneity. Treatments consisted of two rates of ATB (B1, B2), lime (L1, L2), and fly ash (A1, A2) amendments plus a control (C). Mixed soils were placed into 1 quart (0.95 L) Coex square pots to about 2.5 cm from the top. Amendments were then added to the surface and mixed in using a stainless-steel spoon (Figure 6.1). Control pots were also mixed since this process caused soils to settle within the pots. After mixing, pots were topped-up with soil and placed randomly in 14 x 10 grids inside a greenhouse facility (Figure 6.2).

One cation and one anion Plant Root Simulator (PRSTM) probe (Western Ag Innovations Inc.) was inserted into each pot to capture ion diffusion fluxes (Figure 6.3). A PRSTM-probe is an ion exchange membrane encased in plastic that provides a dynamic measure of ion flux to a quantifiable surface area and represents plant nutrient supply rates for the duration of burial (Western Ag 2010). Pots were watered once a week to approximately 20-25% moisture content by volume (MC_v) based on readings from dummy soil pots placed alongside treated pots. Watering rates were not adjusted to account for possible effects of amendments on moisture holding capacity. Air temperature was kept at approximately 20°C via an automatic greenhouse heating and venting system. A clear plastic sheet was placed over the pots between watering to help conserve moisture. In essence, the watering and temperature regime mimicked a weekly rain event during the summer growing season that would keep soil moist, but not wet.

Sampling: Five pots associated with each treatment were randomly selected for sampling after 1 week, 3 weeks, 6 weeks, and 10 weeks. PRSTM-probes were removed from sampled pots and refrigerated in individual plastic bags for later cleaning with de-ionized water. Cleaned probes were sent to Western Ag Inc. for in-house extraction and analysis of calcium (Ca²⁺), magnesium (Mg²⁺), potassium (K⁺), ammonium (NH₄⁺-N), iron (Fe³⁺), manganese (Mn²⁺), aluminum (Al³⁺), cadmium (Cd²⁺), copper (Cu²⁺), lead (Pb²⁺), zinc (Zn²⁺), nitrate (NO₃⁻-N), sulphate (SO₄²⁻-S), and phosphate (PO₄³⁻-P). Probes were eluted with a counterion solution (0.5 N HCl) for one hour followed by colorimetric analysis (NO₃⁻-N, NH₄⁺-N) and ICP analysis (all other elements) (Western Ag 2010).

To aid analysis and interpretation of ion flux data, (i) percent MC_v was measured in each sampled pot after probe removal using a HydrosenseTM moisture meter, (ii) bulk density (Db) samples were collected from all 3-week sample pots after probe removal using a 2-inch soil corer (90.6 cm³ sample volume), and (iii) electrical conductivity (EC) and pH (5:1 water) were measured in all 10-week pot soils after probe removal using an ExStik EC500 meter (Extech Instruments).

Amendments: Amendments used for this study were locally sourced from N-Viro Systems Canada (ATB), JD Irving Ltd. (fly ash), and Mosher Limestone Company Ltd. (powdered lime). All three products are routinely used as liming amendments in Nova Scotia agricultural soils. ATB application rates were intended to roughly match those used in related plantation field trials (7.5 and 15 t ha⁻¹ ATB wet weight, Chapter 3) which were equivalent to about 1,000 kg ha⁻¹ Ca and 2,000 kg ha⁻¹ Ca respectively. Lime and fly ash application rates were calculated to roughly match the Ca content of ATB applications. All calculations were based on typical moisture and Ca contents for each product (Table 6.2).

Amendment samples (n = 5) were analyzed after trial set-up to quantify actual element loads, EC, pH, and C:N ratio. Total Ca, Mg, K, P, and metals were determined through nitric acid digestion using a Microwave Accelerated Reaction System (MARS) followed by AAS analysis. Total C, N, and S were measured using a LECO induction furnace. EC and pH (5:1 water) were measured using an ExStik EC500 meter (Extech Instruments). Moisture content was determined by oven drying samples at 105° C for 48 hours.

ATB and fly ash samples had much higher moisture contents than expected, approximately 2x for ATB (64% versus 33%) and 3x for ash (61% versus 19%). This was later attributed to condensation that had formed in the amendment sample containers acquired for this study. Bagged lime samples had low moisture content as expected (< 1%). Ca content in ash (7.4%) was in the expected range (Table 6.2), but Ca was higher than expected in ATB samples (28%) and lower in lime samples (19%). After adjusting for moisture content and average Ca concentrations, estimated Ca application rates were 791 kg ha⁻¹ (B1), 1,583 kg ha⁻¹ (B2), 992 kg ha⁻¹ (L1), and 1,984 kg ha⁻¹ (L2), 451 kg ha⁻¹ (A1), and 903 kg ha⁻¹ (A2) (Table 6.3). Amendment pH, EC, and C:N ratios are shown in Table 6.4. Although all amendments contain carbonates, ATB and fly ash also contain organic C (Gillis and Price 2010; Merino et al. 2017), and it is this organic fraction that is of interest here.

Statistical Analysis: All response variables were analyzed as repeated measures using SAS PROC MIXED (version 9.3, SAS Institute Inc.). Fixed factors and effects were treatment (B1, B2, L1, L2, A1, A2, C), time (week 1, 3, 6, and 10 since treatment), and treatment-x-time interaction, with soil moisture content at time of assessment used as a covariate. Normality of error terms was assessed for each variable using normal probability plot of residuals, and if violated, power transformations were used. Both compound symmetry and unstructured

covariance structures were tested to see which gave the best results. Significance of model terms was assessed at $\alpha = 0.05$. Significance of multiple means comparisons was assessed at $\alpha = 0.01$ to reduce the possibility of Type I error inflation resulting from the large number of treatment combinations and significant interaction effect found during data analyses. Plots of means were generated using Minitab software version 17 (Minitab Inc.). Soil moisture (MCv), Db, EC, and pH measures were assessed using Welch's ANOVA with Games-Howell comparisons of means ($\alpha = 0.05$).

RESULTS and DISCUSSION

Db, pH, EC, MCv: There were no differences in mean Db between treatments indicating relatively uniform preparation of test pots for each soil (Table 6.5). However, significant pH and EC differences were found which reflect differences in amendment attributes. Looking separately at low rate (A1, B1, L1) and high rate (A2, B2, L2) applications, lime treated soils had the highest pH in both soils studied followed by ATB and ash treated soils. Values ranged from 6.17 to 5.52 in Soil 1 and 5.94 to 5.16 in Soil 2, with significantly lower values of 4.23 and 4.20 in control soils (Table 6.5). Sharifi et al. (2013) found a similar relative pH response in agricultural soil using the same lime and fly ash amendments applied in this study, while Price et al. (2015) recorded the same type of relative response with ATB. The opposite trend was found for EC measures with ash treated soils having the highest EC values followed by ATB and lime treated soil (Table 6.5). Except for the L1 treatment in Soil 2, all treated soils also had significantly greater EC values than control soils. Higher EC values found in ash treated soils reflect the much greater EC values for this amendment (Table 6.4) as well as higher amendment

inputs (Table 6.2). Also, weekly watering rates did not allow for any leaching loss of accumulated salts/ions from any treated soil.

Except for $\text{PO}_4^{3-}\text{-P}$, soil moisture content at time of sampling was an important covariate for most treatment responses (Table 6.6). This is consistent with research that has shown ion diffusion in soils to be governed by soil water content (Olesen et al. 2001; Hu and Wang 2003), which in turn is related to soil texture, organic matter content, and porosity. Using methods described by Olesen et al. (2001), a diffusion threshold percent soil moisture content by volume (T-MCv) was calculated for Soil 1 and Soil 2 using average coarse fragment corrected Db (Table 6.5) and percent clay and silt data (Table 6.1). These thresholds reflect MCv values below which diffusivity is thought to be severely restricted by a breakdown of diffusion pathways. As shown in Table 6.7, mean soil MCv at time of sampling was greater in ash treated soils (12.6-14.6%) versus other soils (9.2-11.2%) and closer to the estimated diffusivity T-MCv for each soil (17% for Soil 1 and 14% for Soil 2). This suggests there were longer periods of time between weekly watering events when ash treated soils had MCv conditions more conducive to diffusion than either control, ATB, or lime treated soils. Higher organic matter content in fly ash versus other amendments was the likely cause of increased moisture retention in these soils.

Base cations: Base cation (Ca^{2+} , Mg^{2+} , K^+) fluxes showed highly significant ($p < 0.01$) treatment-x-time interactions for both soils (Table 6.6). Ca fluxes were significantly greater than the control for all treated soils by week 3, with fluxes in ATB and ash treated soils significantly greater (or nearly so) than fluxes in lime treated soils by week 10 (Figures 6.4 and 6.5). In contrast, Mg^{2+} fluxes in limed soils were 4-7 times greater than those found in ash treated soils by week 10, and up to 39 times greater than ATB treated soils (Figures 6.4 and 6.5, Tables 6.8 and 6.9). K^+ fluxes in ash treated soils were much greater than all other treatments, but showed a

decreasing trend over time – the opposite of Ca^{2+} and Mg^{2+} (Figures 6.4 and 6.5). Also, total K^+ fluxes in control soils were significantly greater than ATB and lime treated soils by week 10 (Figures 6.4 and 6.5, Tables 6.8 and 6.9).

Greater Ca^{2+} fluxes in ATB versus ash treated soils generally reflect differences in Ca inputs (Table 6.3), but higher average MCv in ash treated soils (Table 6.7) suggest differences would have been even greater under more uniform moisture conditions. Differences in Ca inputs do not, however, account for the lower Ca^{2+} fluxes recorded for lime treated soils (Tables 6.8 and 6.9) which had greater Ca inputs than both ATB and ash treated soils (Table 6.3). This suggests more Ca in ATB and fly ash was in an immediately available form compared to lime where ongoing dissolution of CaCO_3 was required for Ca^{2+} availability. This is also supported by the higher EC values for ATB and fly ash compared to lime (Table 6.4).

Similarly, the much higher Mg^{2+} fluxes found in lime treated soils versus other soils (Tables 6.8 and 6.9) cannot be fully accounted for by differences in Mg inputs alone. As noted, Mg^{2+} capture in lime treated soils was up to 7 times greater than ash treated soils and 39 times greater than ATB treated soils, but Mg inputs for lime and ash were only about 6 times greater than ATB (Table 6.3). Increased Mg^{2+} capture in lime treated soils could be related, in part, to lime having fewer “competing” cations than other amendments (especially ash), but the lack of any significant difference between Mg^{2+} capture in ATB treated and control soils (Figures 6.4 and 6.5) suggests there must have been a relative balance between the release of Mg^{2+} from ATB and the diffusion gradient and exchange balance in PRSTM-probes. This lack of Mg response in ATB treated soil is also consistent with plantation field trial results (Chapters 3-4).

With respect to K^+ fluxes, fly ash had much higher K inputs than other amendments (Table 6.3) and this was evident in the high K^+ fluxes found in ash treated soils (Figures 6.4 and

6.5). The immediate response of K^+ also reflects the high EC for this amendment (Table 6.4). However, decreasing K^+ measures over time indicates other cations (especially Ca^{2+}) were displacing K^+ in PRSTM-probes as equilibrium conditions were established. This was likely also the case for other treatments where low K inputs and cation displacement resulted in control soils having significantly greater total K^+ capture than ATB and lime treated soils by week 10 (Figures 6.4 and 6.5).

N, P, and S: As with base cations, total N (NO_3^- -N + NH_4^+ -N), PO_4^{3-} -P, and SO_4^{2-} -S fluxes showed significant treatment-x-time interactions for both soils, but PO_4^{3-} -P responses were not as highly significant as other ions (Table 6.6). Total N fluxes in ATB treated soils were significantly greater (or nearly so) than control soils by week 10, while fluxes in ash treated soils were significantly less (Figures 6.6 and 6.7). Except for one measurement, N fluxes in lime treated soils were the same as control soils over the entire study period (Figures 6.6 and 6.7). Also of note was the decrease in NH_4^+ -N fluxes for all treated soils compared to controls after week 3, with NO_3^- -N making up almost all N fluxes measured in weeks 6 and 10 (Figures 6.6 and 6.7). PO_4^{3-} -P fluxes were very low and more variable than total N fluxes (Tables 6.8 and 6.9), with only ATB treatments showing significant increases versus the control for Soil 1 (high rate) and Soil 2 (both rates) by week 10 (Figure 6.8). SO_4^{2-} -S fluxes in ash treated soils were much greater than all other treatments (about 5-15 times) as well as control soils (more than 20 times) (Figure 6.8, Tables 6.8 and 6.9), while fluxes in lime and ATB treated soils were also significantly greater than the control for Soil 1, but not for Soil 2 (Figure 6.8).

Increased N capture in ATB treated soils (Figures 6.6 and 6.7) reflects the greater N inputs in this amendment (10 times more than fly ash and over 200 times more than lime, Table 6.3), but studies have shown that only some of this N is immediately available (Price et al. 2015)

which limited total capture levels in this study (Tables 6.8 and 6.9). Similarly, no difference in N capture between lime treated and control soils reflects the lack of N in this amendment. The significant decrease in total N capture for ash treated soils was, however, likely more related to C inputs than N inputs. The C:N ratio of fly ash was 177, more than 10 times greater than the ratio for ATB (Table 6.4). Although fly ash usually has less organic C than bottom ash (Merino et al. 2017), the high C:N ratio of this product suggests significant organic C was added to ash treated soils which probably resulted in enhanced microbial uptake of N over the study period. Lastly, the drop in NH_4^+ -N and shift to NO_3^- -N fluxes from week 6 onward was likely related to the increase in soil pH noted for all treated soils (Table 6.5) which would tend to promote increased nitrification (e.g., Zebarth et al. 2015).

In addition to being low, PO_4^{3-} -P fluxes were not well correlated with MCV for all treated soils (Table 6.6). This suggests P was essentially unavailable in these soils for the duration of study, either bound as insoluble Al/Fe/Ca phosphates or as organic P. Only with ATB treatments were PO_4^{3-} -P fluxes starting to increase by week 10 (Figures 6.8). This was likely due, in part, to the more favourable Al/Fe:P ratio in ATB compared to fly ash (Table 6.3). Sharifi et al. (2013) found P uptake in ryegrass (*Lolium multiflorum*) to be reduced in agricultural soils treated with the same fly ash used in this study. They attributed this to the relatively high content of Ca, Fe, and Al (versus P) in this amendment.

As with K, fly ash had much higher S inputs than other amendments (Table 6.3) which led to the much greater SO_4^{2-} -S fluxes recorded in ash treated soils versus ATB and lime treated soils. Furthermore, this anion was immediately available as shown by high flux measures in week 1 samples (Figure 6.8) which also reflected the high EC of this amendment (Table 6.4).

This is consistent with other studies that show relatively high S content in fly ash (e.g., Pugliese et al. 2014).

Metal ions: Cd²⁺ fluxes in both soils were highly variable with many “below detection” results that hindered statistical analysis. However, looking only at the more consistent week 10 data set, Cd²⁺ fluxes were very low in all treated soils and no different than control soils (Figure 6.9). This reflected low Cd content in the amendments used (Table 6.3). All other metals (Fe³⁺, Mn²⁺, Al³⁺, Cu²⁺, Pb²⁺, Zn²⁺) showed significant treatment-x-time interactions for both soils (Table 6.6). Several trends in metal ion fluxes were evident:

- There was little difference in Fe³⁺ and Al³⁺ fluxes between treatments and controls, as noted by the lack of significant treatment response for Al³⁺ in both soils and Fe³⁺ in Soil 2 (Table 6.6).
- Despite initial increases in ash treated soils, Mn²⁺ fluxes tended to decrease over time in all treated soils compared to control soils (Figures 6.10 and 6.11). Early increases in ash treated soils reflected the greater Mn content in this amendment (Table 6.3).
- By week 10, total metal fluxes in all treated soils were generally the same or less than those measured in control soils (Tables 6.8 and 6.9). The main exception to this was Cu²⁺ where fluxes in ATB treated soils were significantly greater than control soils (Figures 6.12 and 6.13). Total Cu²⁺ fluxes were, however, low for all soils (less than 2 μg 10cm⁻² for Soil 1 and less than 1 μg 10cm⁻² for Soil 2) (Tables 6.8 and 6.9).
- By week 10, Zn²⁺ fluxes in control soils were significantly greater than fluxes in all treated soils (Figures 6.12 and 6.13).

- By week 10, the lowest Pb^{2+} and Zn^{2+} fluxes were found in ash treated soils (Figures 6.12 and 6.13).

Trace metal (Mn^{2+} , Cd^{2+} , Cu^{2+} , Pb^{2+} , Zn^{2+}) solubility and availability in soils is controlled by adsorption/desorption, precipitation/dissolution and complexation reactions, which are in turn affected by soil pH, CEC, organic matter content, and the presence of competing ions (Silveira et al. 2003). For trace metal fluxes in this study, the most relevant of these factors was the change in soil pH over time. Trace metals are generally less soluble at higher pH values (Basta et al. 2005; Gagnon et al. 2013) and this was especially evident with Zn^{2+} in all treated soils and Pb^{2+} and Cu^{2+} in ash treated soils. Pugliese et al. (2014) reported similar results in their study of various wood ash amendments, noting in particular a decrease in available Pb in treated soils. Yoo and James (2002) also reported pH as a major controlling factor in the availability of Zn in soils treated with biosolids. In this study, greater organic retention probably also contributed to the low metal fluxes found in ash treated versus ATB and lime treated soils, either through specific adsorption or increased pH-dependent CEC (Silveira et al. 2003).

CONCLUSIONS

This study compared cation and anion diffusion fluxes in two forest mineral soils treated with alkaline-treated biosolids (ATB), powdered lime, and fly ash under controlled conditions over a 10-week period.

Soil pH increased significantly in all treated soils versus the control, with lime providing the greatest increase followed by ATB and fly ash. ATB was the most effective in providing available Ca^{2+} , but least effective in providing available Mg^{2+} , where lime was most effective. K^{+}

availability was high early on in ash treated soils, but decreased over time with increasing cation competition. Cation competition also reduced K^+ capture in ATB and lime treated soils versus control soils. Compared to the control, total N (NO_3^- -N + NH_4^+ -N) availability increased in ATB treated soils, stayed the same in lime treated soils, and decreased in ash treated soils. This was related to higher N content in ATB versus lime and the high C:N ratio of fly ash which led to increased N immobilization. Increasing pH also promoted nitrification in all treated soils. PO_4^{3-} -P availability was low in all treated soils, but slightly enhanced in ATB treated soils. SO_4^{2-} -S availability was high in ash treated soils which reflected greater fly ash S content and electrical conductivity versus other amendments. Fe^{3+} and Al^{3+} fluxes were not greatly affected by any treatment, but trace metal (Mn^{2+} , Cd^{2+} , Cu^{2+} , Pb^{2+} , Zn^{2+}) fluxes generally decreased over time with increasing pH. Enhanced organic retention of metals was likely also a factor in ash treated soils.

This study was not designed to mimic the potential use of ATB, lime, or fly ash amendments in forests where these materials would need to be surface applied. Instead, the main objective was to see how ATB compared to fly ash and lime amendments under more traditional use, i.e., incorporated into mineral soil. However, general comments can be made regarding likely response trends in a forest setting.

Results suggest ATB is equally or more effective at providing Ca as lime and fly ash, but less effective at providing Mg due to low inputs and cation competition. Looking at K, fly ash is a much greater source than either ATB or lime, but leaching loss of this mobile cation would likely be extensive in a forest setting due to cation exchange and the high SO_4 input also associated with fly ash. With respect to N, ATB did not greatly increase availability early on, but lime does not contain any N, while fly ash application could induce short-term N deficiencies

due to its high C:N ratio. ATB and fly ash also contain P, but availability may be lower in fly ash amended soils due to its higher Fe and Al content and reduced impact on pH compared to ATB. Lastly, fly ash does appear to have more ability than ATB to retain trace metals, but metal concentrations are low in both amendments – with the possible exception of Mn in fly ash. Impurities in lime could also add more Mn to soil than ATB.

Overall, ATB treatment results from this study are consistent with those found in plantation field trials (Chapters 3-5), and suggest this amendment could be a good source of Ca in Ca-limited soils as long as potential impacts on Mg and K availability are also considered and addressed.



Figure 6.1. Sample soil pots before incorporation of amendments. Clockwise from top left: control, alkaline-treated biosolids (ATB), fly ash, and powdered lime.

1	2	3	4	5	6	7	8	9	10	11	12	13	14
15	16	17	18	19	20	21	22	23	24	25	26	27	28
29	30	31	32	33	34	35	36	37	38	39	40	41	42
43	44	45	46	47	48	49	50	51	52	53	54	55	56
57	58	59	60	61	62	63	64	65	66	67	68	69	70
71	72	73	74	75	76	77	78	79	80	81	82	83	84
85	86	87	88	89	90	91	92	93	94	95	96	97	98
99	100	101	102	103	104	105	106	107	108	109	110	111	112
113	114	115	116	117	118	119	120	121	122	123	124	125	126
127	128	129	130	131	132	133	134	135	136	137	138	139	140

1	2	3	4	5	6	7	8	9	10	11	12	13	14
15	16	17	18	19	20	21	22	23	24	25	26	27	28
29	30	31	32	33	34	35	36	37	38	39	40	41	42
43	44	45	46	47	48	49	50	51	52	53	54	55	56
57	58	59	60	61	62	63	64	65	66	67	68	69	70
71	72	73	74	75	76	77	78	79	80	81	82	83	84
85	86	87	88	89	90	91	92	93	94	95	96	97	98
99	100	101	102	103	104	105	106	107	108	109	110	111	112
113	114	115	116	117	118	119	120	121	122	123	124	125	126
127	128	129	130	131	132	133	134	135	136	137	138	139	140

C	L1	L2	A1	A2	B1	B2

Figure 6.2. Random number generated distribution of soil pots for Soil 1 (top) and Soil 2 (bottom) each in a 14 x 10 grid. C = control, L1 and L2 = powdered lime amendment, A1 and A2 = fly ash amendment, B1 and B2 = alkaline-treated biosolids (ATB) amendment.



Figure 6.3. Soil 2 treated pots with PRSTM-probes. Each pot was numbered and labelled by treatment as shown in Figure 6.2.

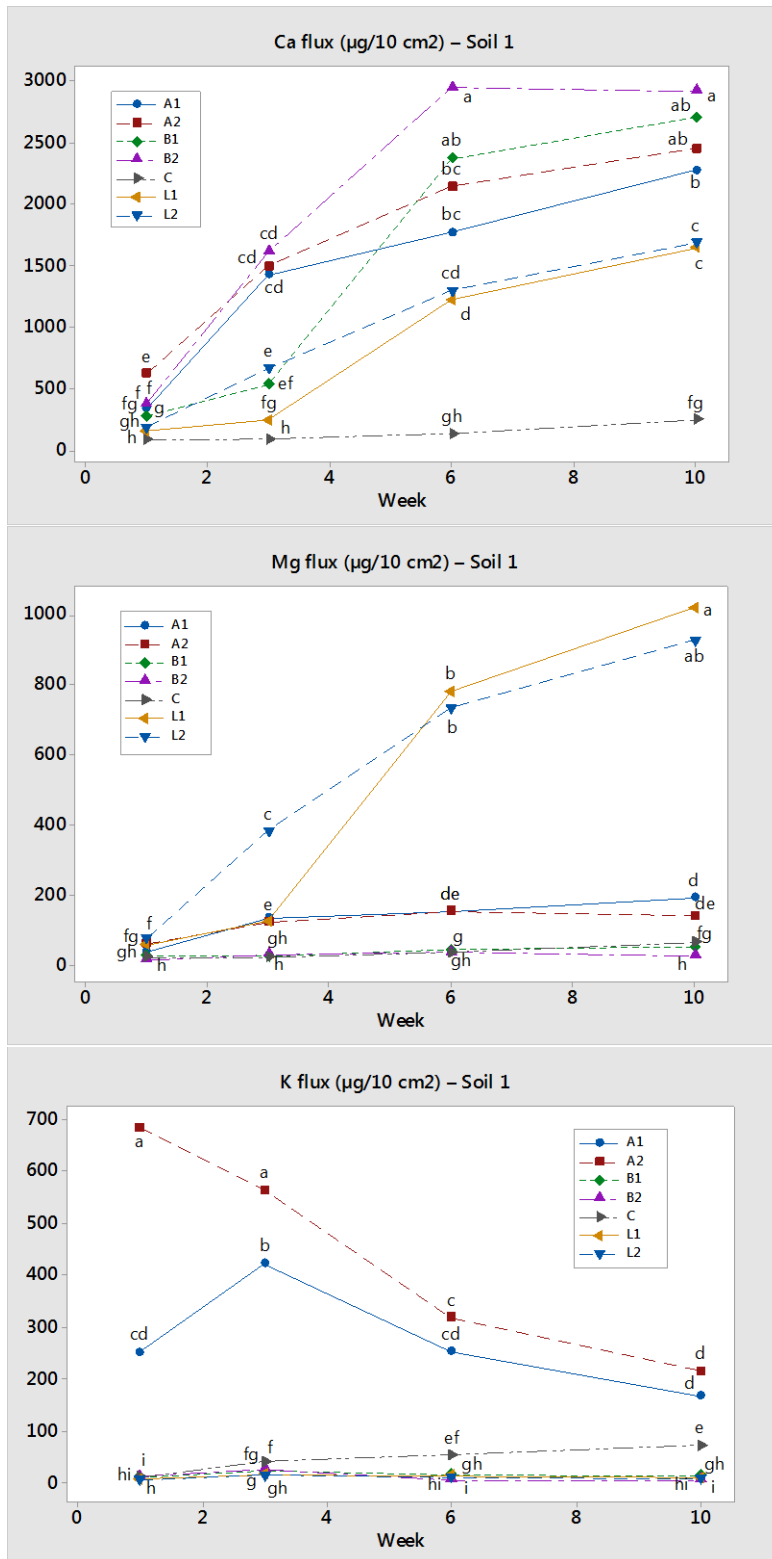


Figure 6.4. Mean Ca^{2+} , Mg^{2+} , and K^+ fluxes by treatment and week for Soil 1. Values with different letters are statistically different at $\alpha = 0.01$.

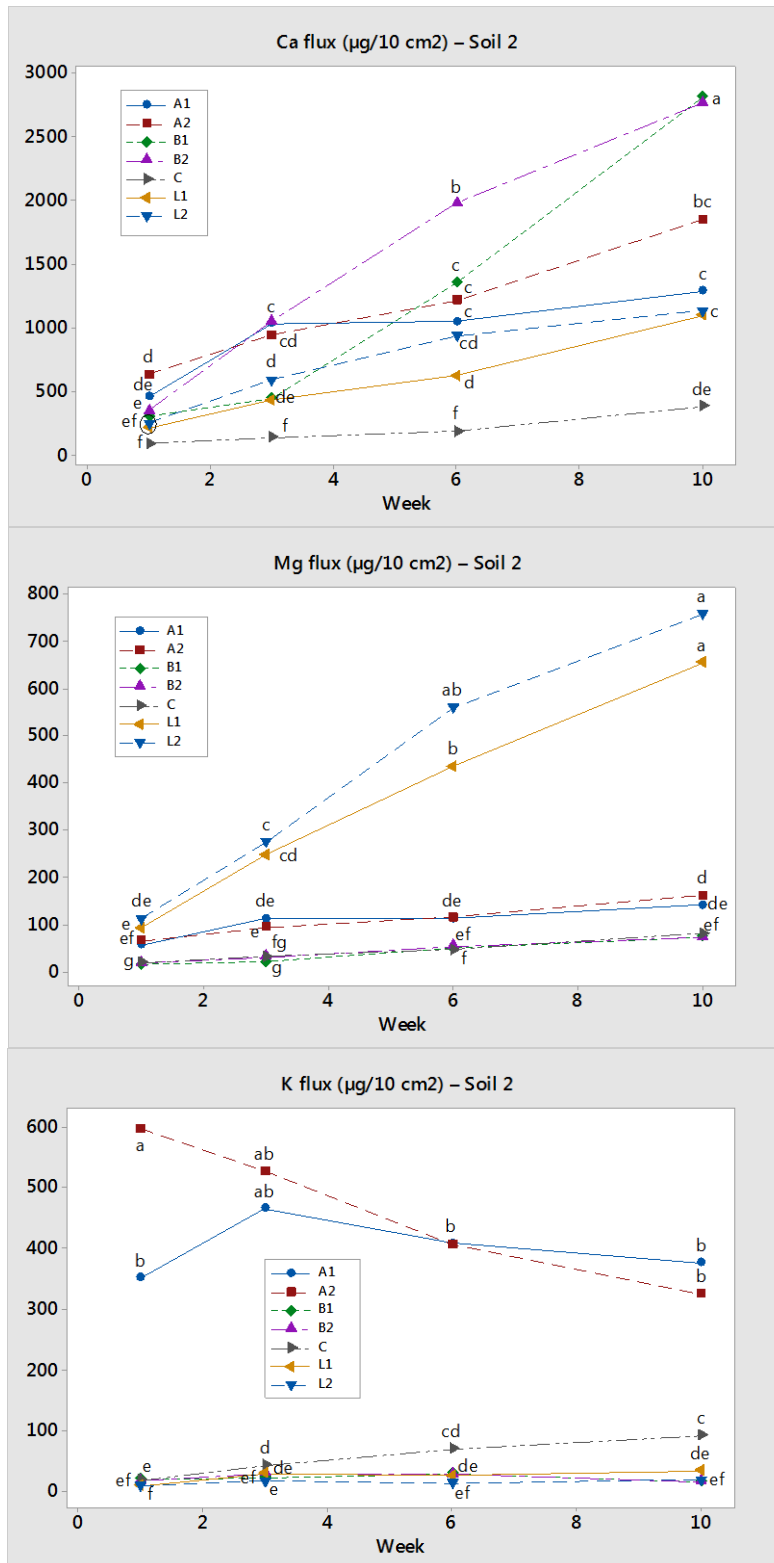


Figure 6.5. Mean Ca^{2+} , Mg^{2+} , and K^+ fluxes by treatment and week for Soil 2. Values with different letters are statistically different at $\alpha = 0.01$.

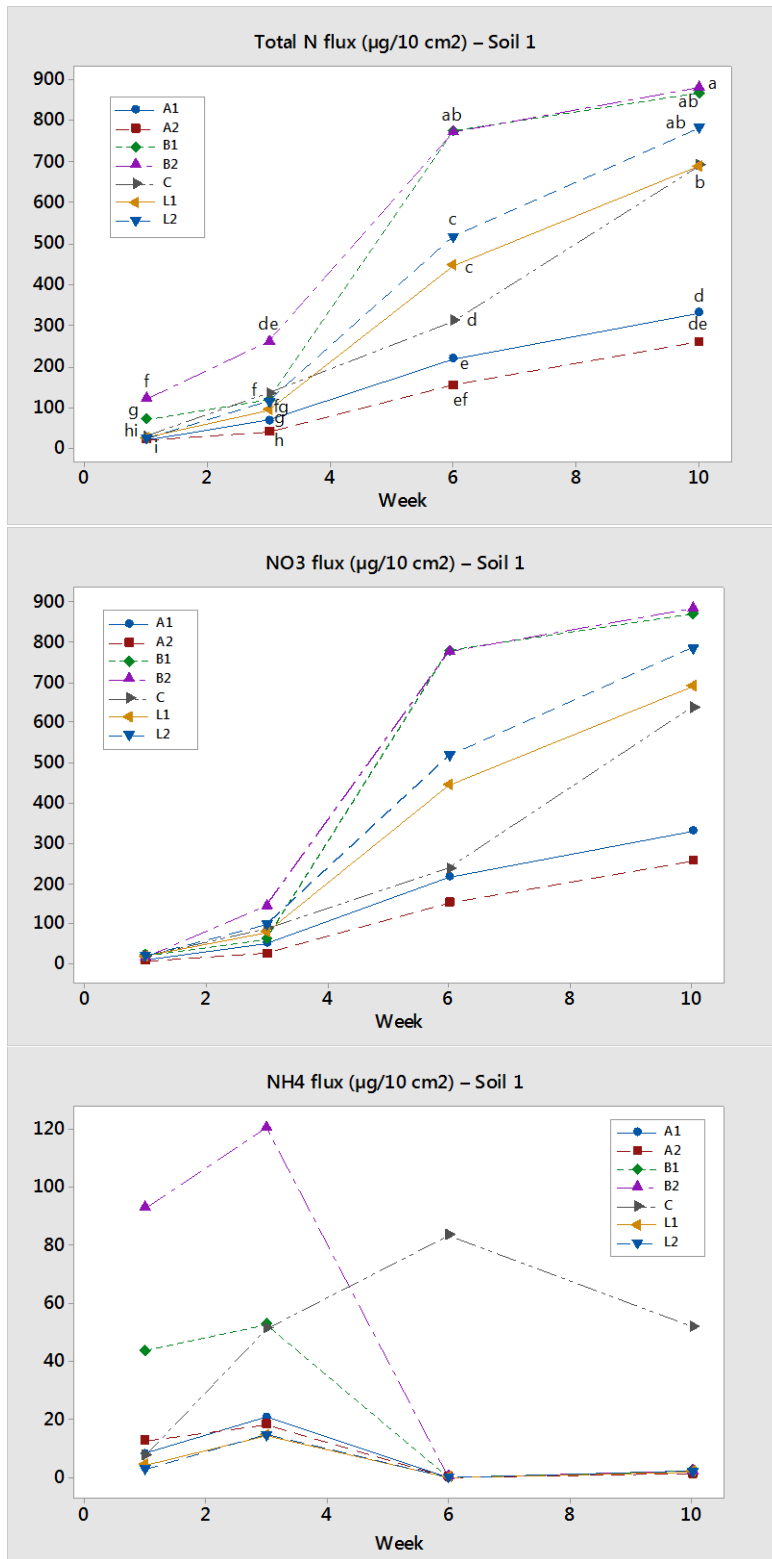


Figure 6.6. Mean total N (NO_3^- -N + NH_4^+ -N), NO_3^- -N, and NH_4^+ -N fluxes by treatment and week for Soil 1. Total N values with different letters are statistically different at $\alpha = 0.01$.

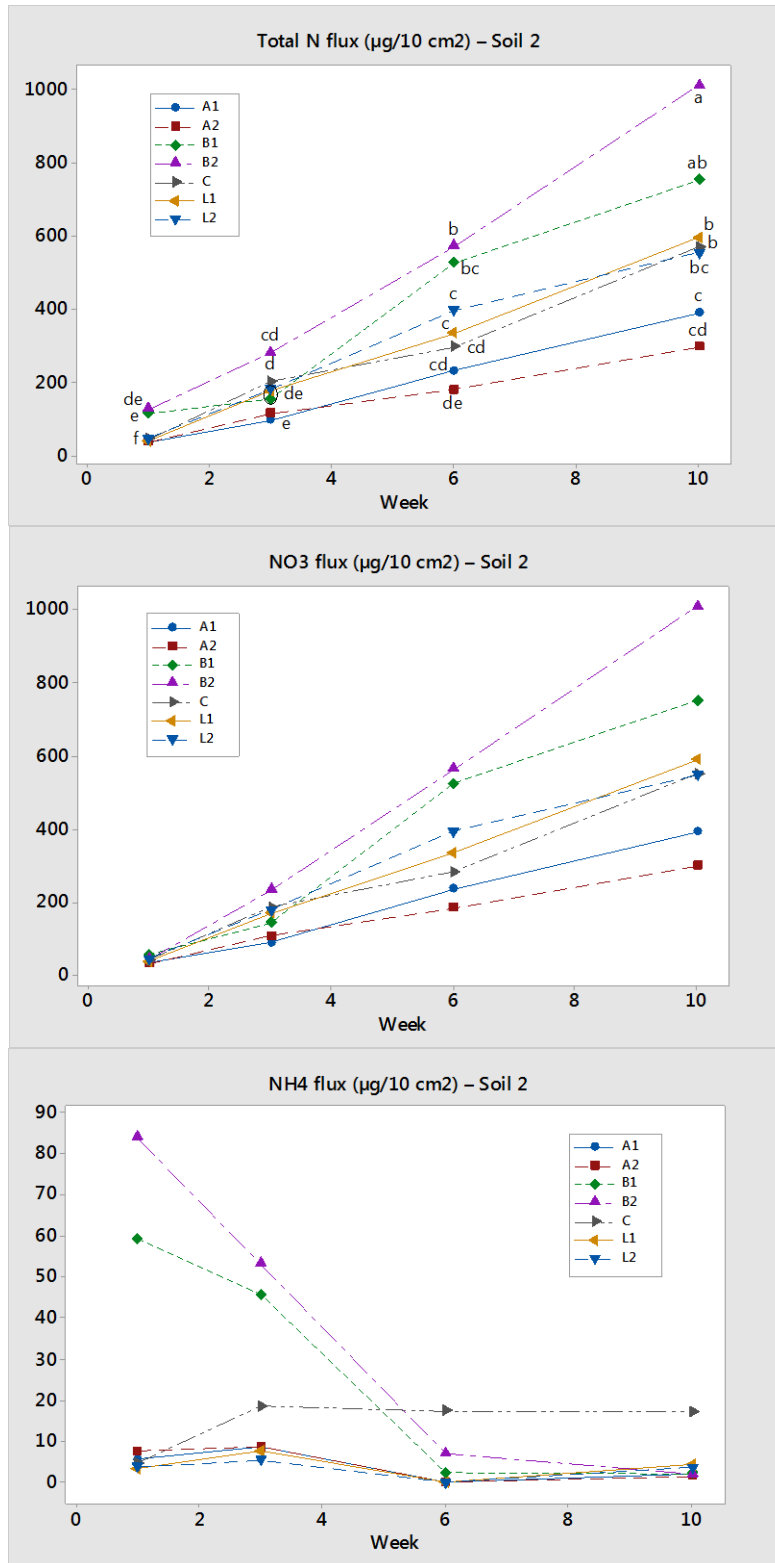


Figure 6.7. Mean total N (NO_3^- -N + NH_4^+ -N), NO_3^- -N, and NH_4^+ -N fluxes by treatment and week for Soil 2. Total N values with different letters are statistically different at $\alpha = 0.01$.

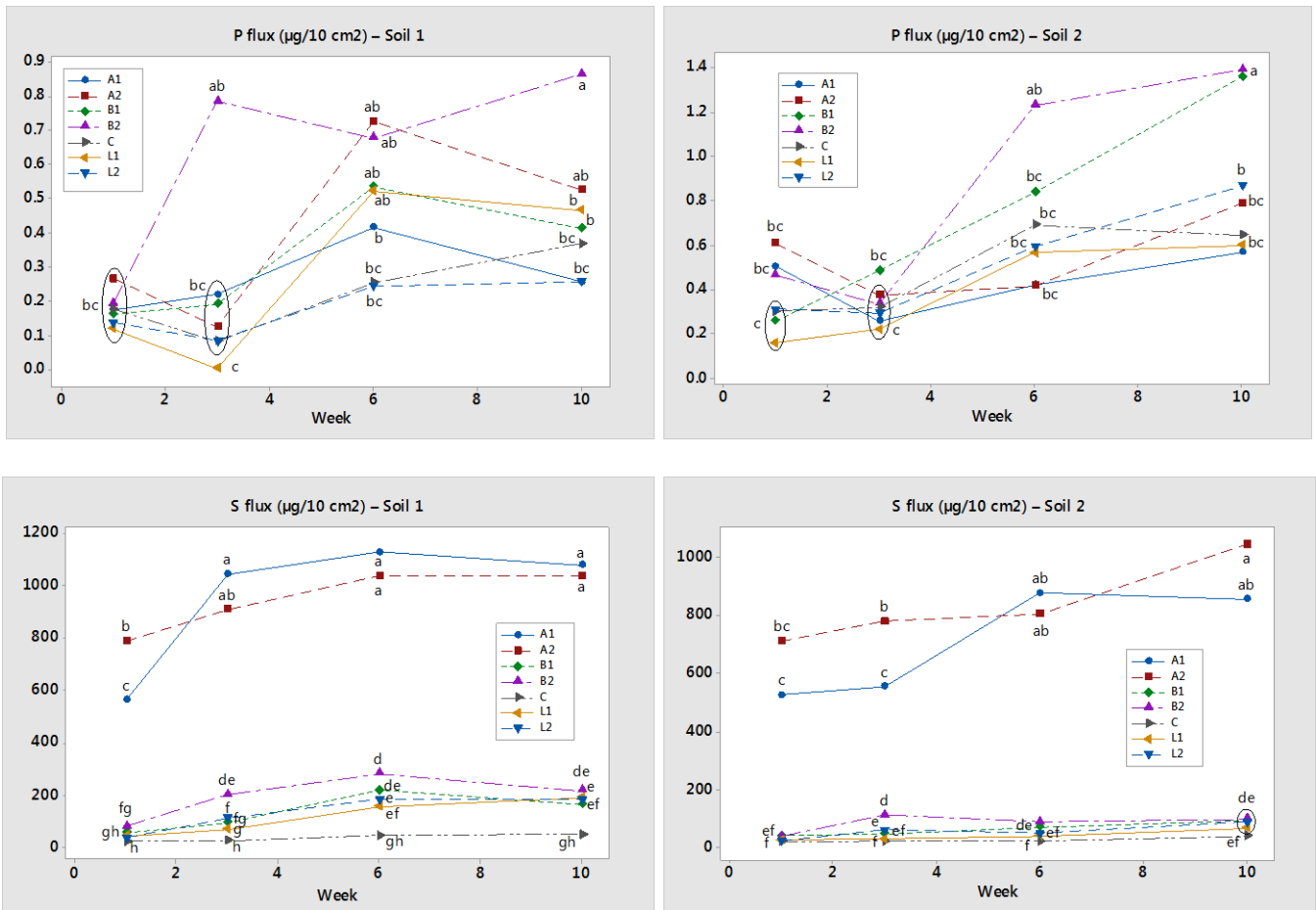


Figure 6.8. Mean PO₄³⁻-P and SO₄²⁻-S fluxes by treatment and week for Soil 1 (left side) and Soil 2 (right side). Values with different letters are statistically different at alpha = 0.01.

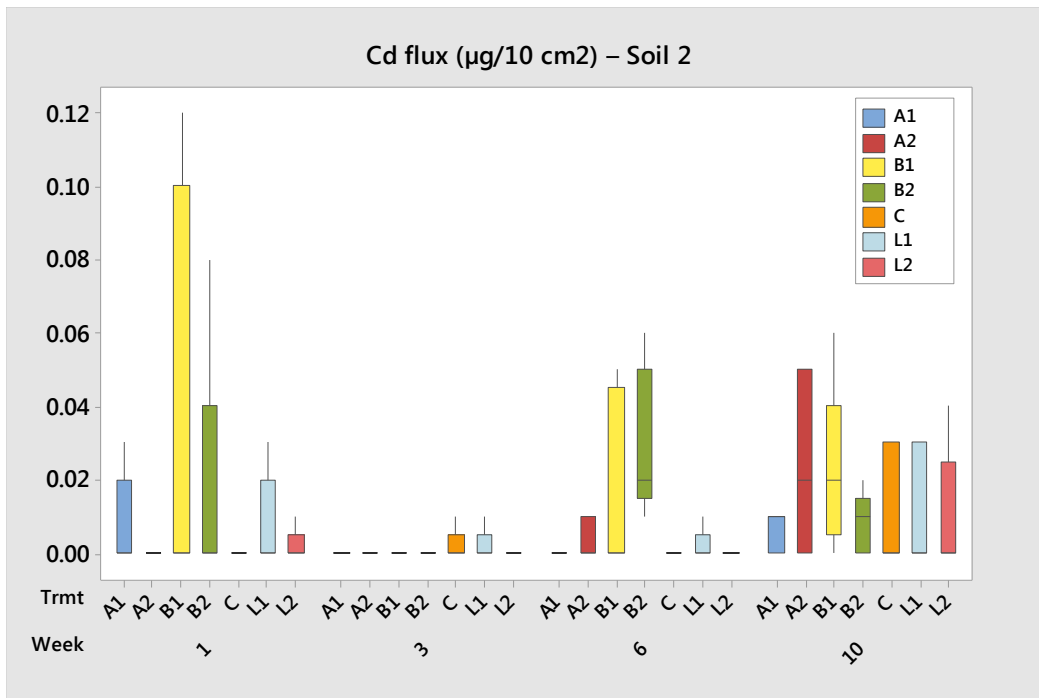
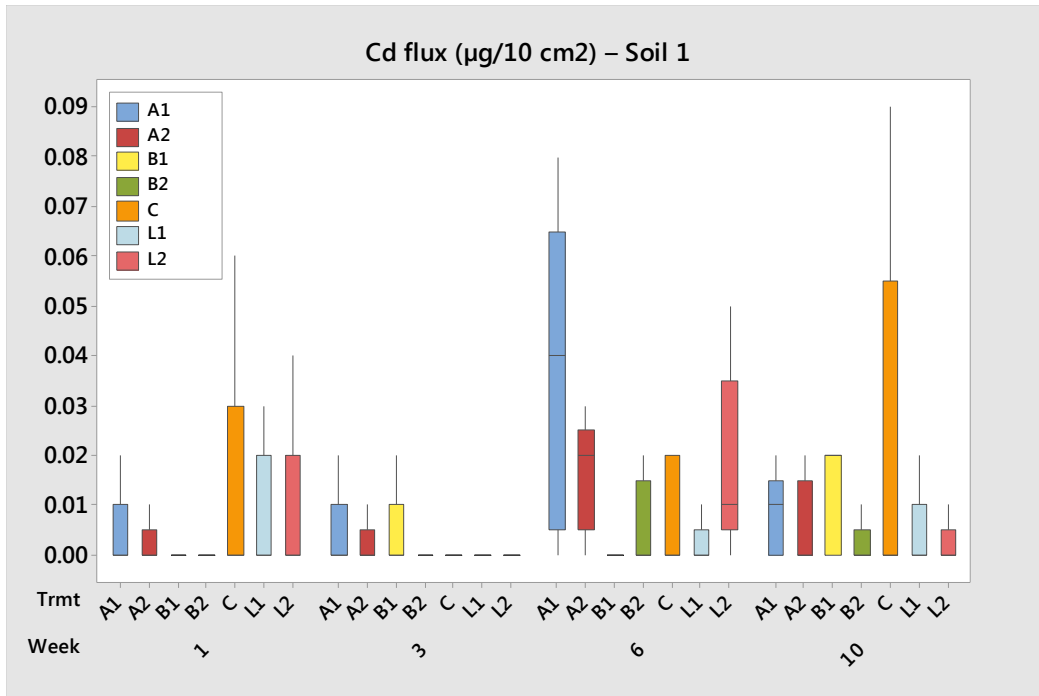


Figure 6.9. Cadmium (Cd^{2+}) fluxes by treatment and week for Soil 1 and Soil 2.

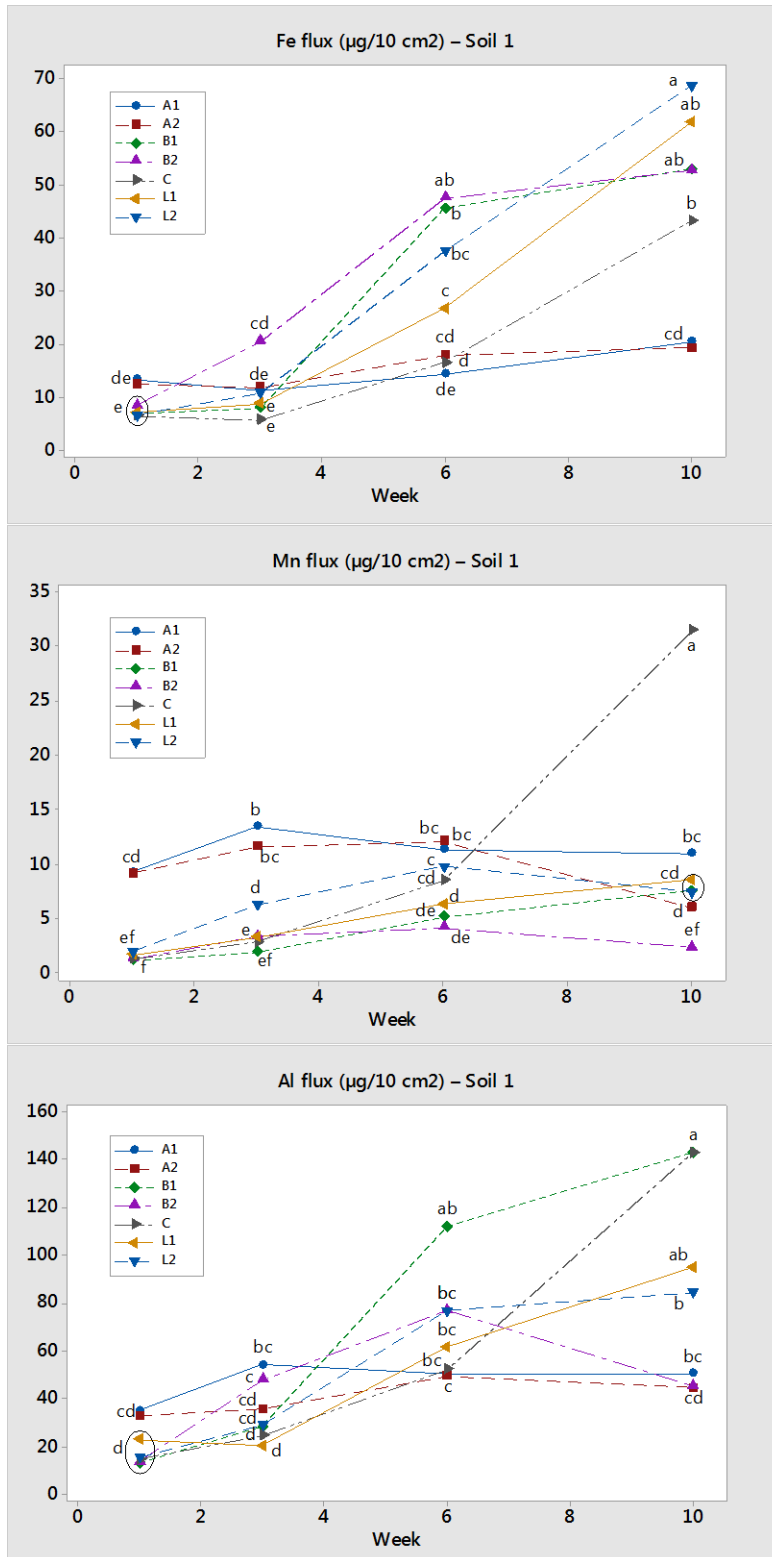


Figure 6.10. Mean Fe^{3+} , Mn^{2+} , and Al^{3+} fluxes by treatment and week for Soil 1. Values with different letters are statistically different at $\alpha = 0.01$.

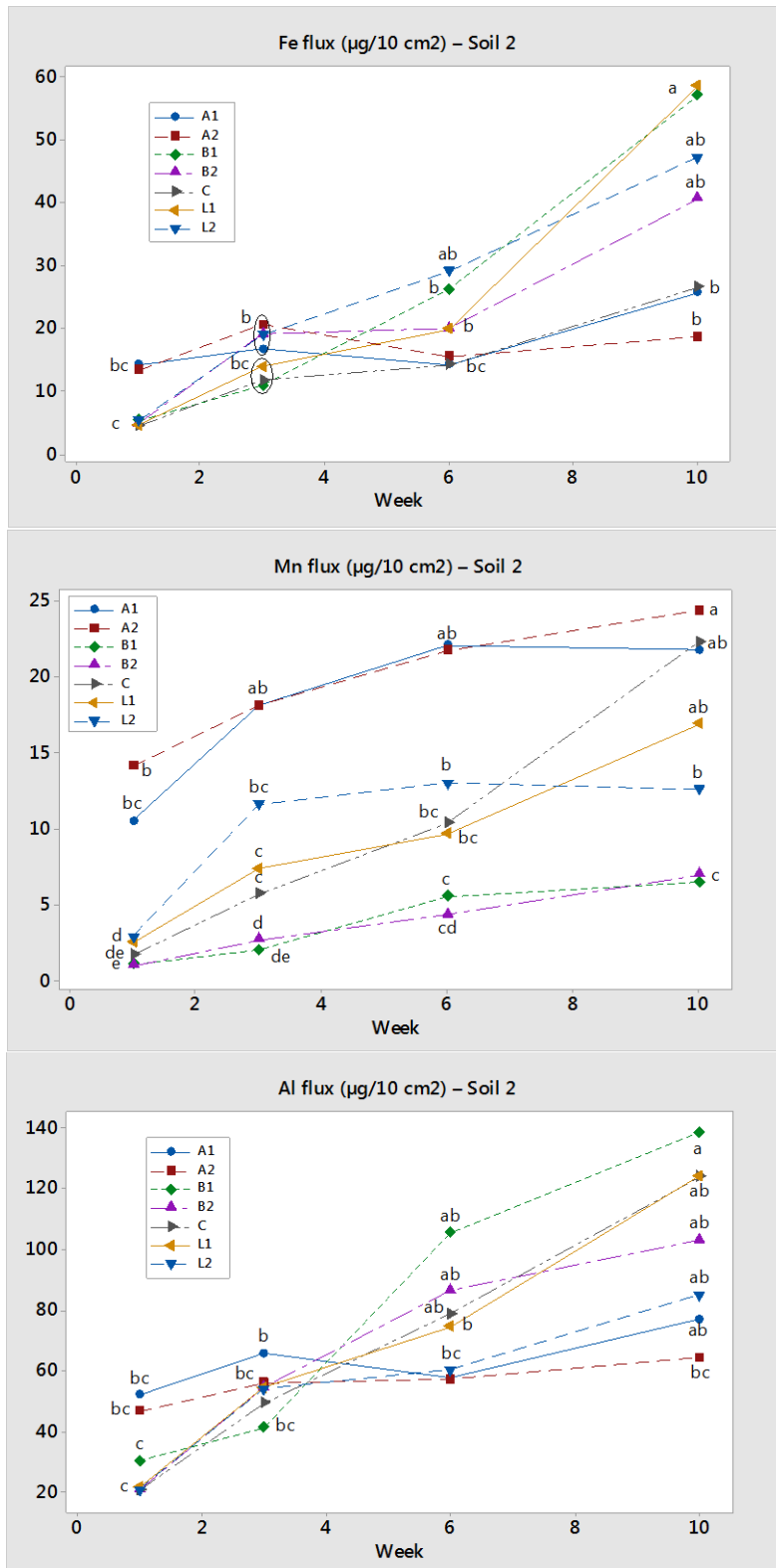


Figure 6.11. Mean Fe^{3+} , Mn^{2+} , and Al^{3+} fluxes by treatment and week for Soil 2. Values with different letters are statistically different at $\alpha = 0.01$.

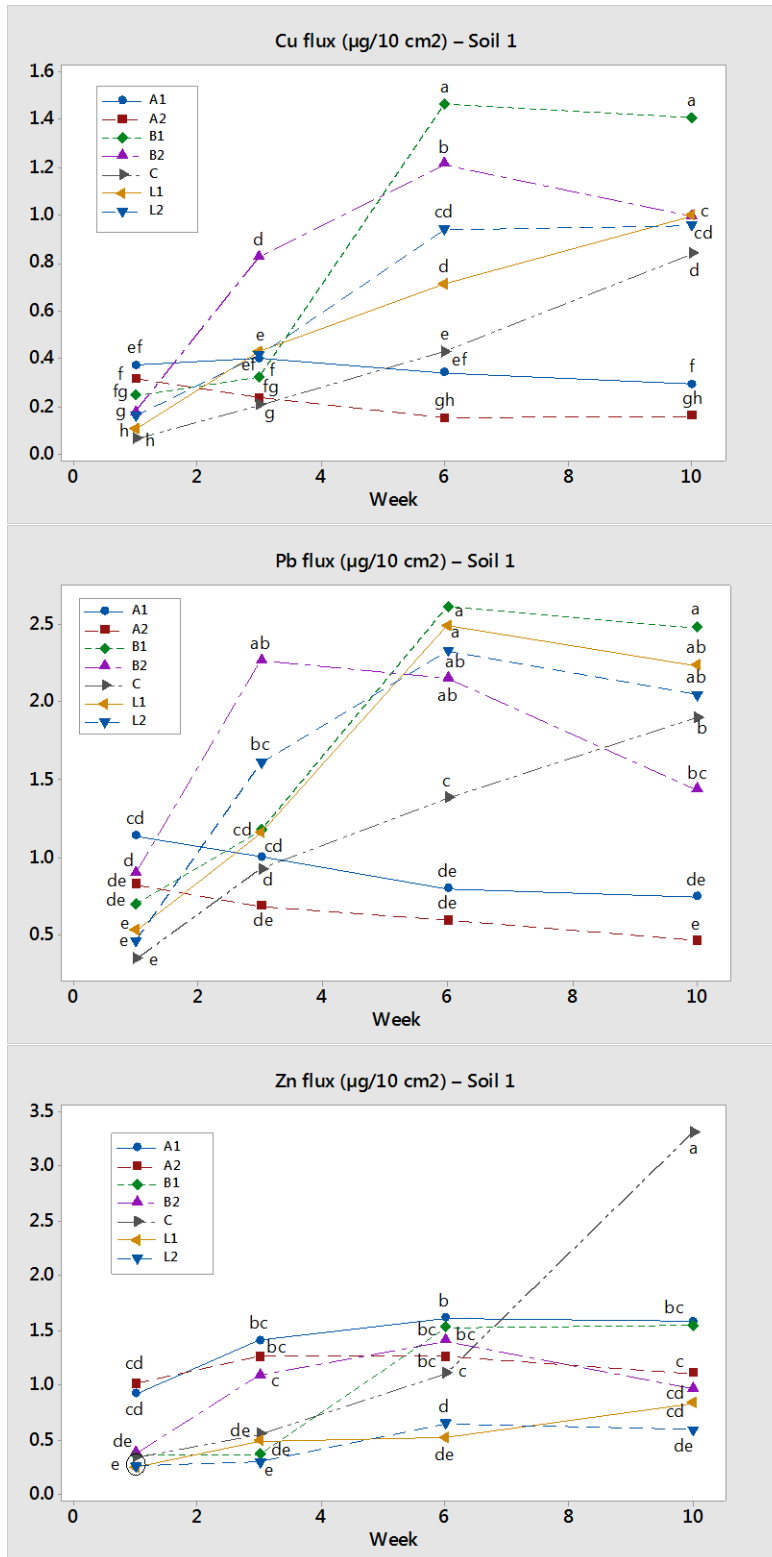


Figure 6.12. Mean Cu^{2+} , Pb^{2+} , and Zn^{2+} fluxes by treatment and week for Soil 1. Values with different letters are statistically different at $\alpha = 0.01$.

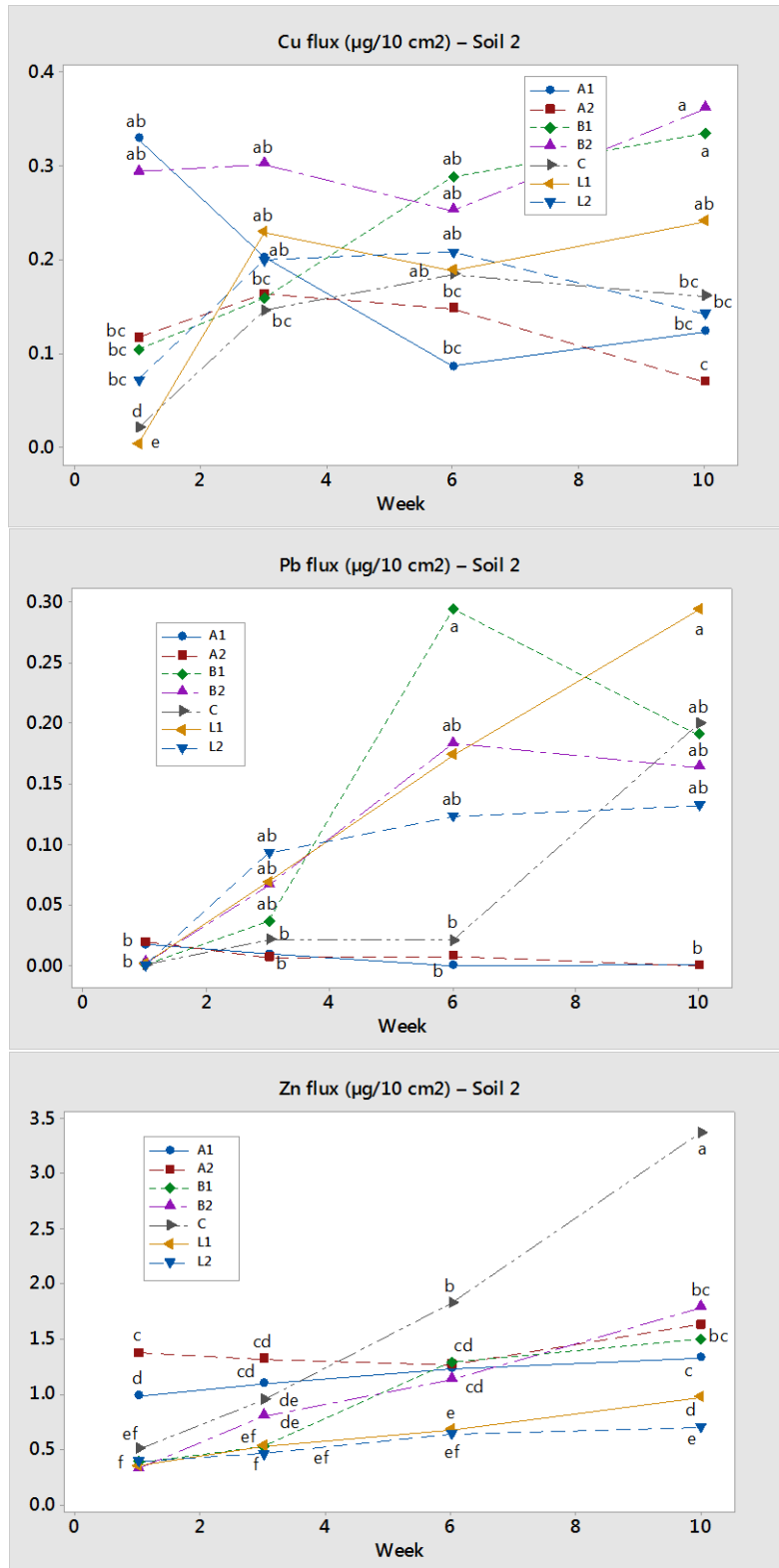


Figure 6.13. Mean Cu^{2+} , Pb^{2+} , and Zn^{2+} fluxes by treatment and week for Soil 2. Values with different letters are statistically different at $\alpha = 0.01$.

Table 6.1. Mineral soil attributes based on earlier sampling of related field trial soils. Db = bulk density; Ca, Mg, K, and Al are exchangeable cation concentrations; Cd, Cu, Pb, and Zn are total element concentrations.

Soil	Statistic	% Sand	% Silt	% Clay	Db (Mg m ⁻³)	pH	% C	% N	C:N
1	Mean	39	43	17	1.00	4.03	3.59	0.27	13.5
	Stdev	5	4	3	0.16	0.05	0.47	0.05	0.6
2	Mean	63	29	8	0.63	4.28	6.24	0.38	16.1
	Stdev	7	5	2	0.1	0.16	2.21	0.10	1.6

Soil	Statistic	Ca (cmol kg ⁻¹)	Mg	K	Al	Cd (mg kg ⁻¹)	Cu	Pb	Zn
1	Mean	0.145	0.085	0.052	3.90	0.00	12.61	0.00	56.17
	Stdev	0.073	0.044	0.005	0.52	0.00	6.75	0.00	8.26
2	Mean	0.098	0.058	0.058	2.86	0.55	7.65	3.01	76.78
	Stdev	0.032	0.019	0.024	0.96	0.77	1.07	3.97	10.07

Table 6.2. Typical amendment moisture and calcium (Ca) contents used to calculate amendment application rates.

Amendment	Typical Moisture Content (% wt)	Typical Ca Content (% wt)	Calculated Low Rate Application (g)	Calculated High Rate Application (g)
Fly Ash	19	6.5-9.5	16.0 (A1)	32.0 (A2)
ATB	33	10-20	8.0 (B1)	16.0 (B2)
Lime	1	25-30	5.4 g (L1)	10.8 (L2)

Table 6.3. Estimated element application rates and ratios by treatment. Ratios were based on the lowest application rate for each element. A1 and A2 = low and high rate fly ash treatments, B1 and B2 = low and high rate ATB treatments, L1 and L2 = low and high rate lime treatments, bd = below detection, na = not applicable.

Application Rates (kg ha⁻¹)													
Trmt	Ca	Mg	K	P	N	S	Fe	Mn	Al	Cu	Pb	Zn	Cd
A1	451	48	136	25	3	106	73	48	88	0.24	0.23	3.39	0.032
A2	903	95	271	50	6	212	146	96	176	0.48	0.46	6.78	0.065
B1	791	8	2	16	32	0.6	13	1.4	40	0.31	0.24	0.58	0.011
B2	1,583	15	4	33	64	1.1	26	2.7	81	0.62	0.48	1.15	0.022
L1	992	51	7	0.8	0.1	bd	48	32	37	0.05	0.08	0.06	0.002
L2	1,984	103	14	1.6	0.3	bd	96	63	73	0.09	0.15	0.12	0.004

Application Ratios													
Trmt	Ca	Mg	K	P	N	S	Fe	Mn	Al	Cu	Pb	Zn	Cd
A1	1.0	6.2	67	32	21	188	5.6	35	2.4	5.4	3.0	55	17
A2	2.0	12.4	134	64	42	376	11.2	70	4.8	10.8	6.1	110	34
B1	1.8	1.0	1.0	21	221	1.0	1.0	1.0	1.1	6.9	3.2	9.4	5.9
B2	3.6	2.0	2.0	42	442	2.0	2.0	2.0	2.2	13.8	6.4	18.8	11.8
L1	2.2	6.7	3.4	1.0	1.0	na	3.7	23	1.0	1.0	1.0	1.0	1.0
L2	4.4	13.4	6.9	2.0	2.0	na	7.3	46	2.0	2.0	2.0	2.0	2.0

Table 6.4. Mean pH, electrical conductivity (EC), and C:N ratio for fly ash, ATB, and lime amendments. C:N ratios for fly ash and ATB include an unknown proportion of inorganic C.

Amendment	Mean pH	Mean EC (mS)	Mean C:N
Fly Ash	11.9	13.9	177
ATB	9.2	1.86	15
Lime	9.3	0.22	na

Table 6.5. Bulk density (Db) and coarse fragment corrected Db measured in 3-week sample pots, and pH and electrical conductivity (EC) measured in 10-week sample pots. Values with different letters are statistically different ($\alpha = 0.05$). A1 and A2 = low and high rate fly ash treatments, B1 and B2 = low and high rate ATB treatments, L1 and L2 = low and high rate lime treatments.

Soil 1								
Trmt	Db (kg Mg ⁻¹)		corr. Db (kg Mg ⁻¹)		pH		EC (μ S cm ⁻¹)	
C	1.05	a	0.80	a	4.23	e	125	d
A1	1.03	a	0.77	a	5.52	d	665	b
A2	1.02	a	0.76	a	5.81	bc	1511	a
B1	1.05	a	0.79	a	5.69	c	271	c
B2	1.08	a	0.77	a	6.15	a	377	b
L1	1.05	a	0.77	a	6.01	ab	257	c
L2	1.03	a	0.76	a	6.17	a	300	bc
Soil 2								
Trmt	Db (kg Mg ⁻¹)		corr. Db (kg Mg ⁻¹)		pH		EC (μ S cm ⁻¹)	
C	0.85	a	0.73	a	4.20	d	171	f
A1	0.87	a	0.74	a	5.16	c	942	b
A2	0.82	a	0.70	a	5.61	b	1759	a
B1	0.85	a	0.72	a	5.41	c	350	d
B2	0.86	a	0.73	a	5.78	ab	536	c
L1	0.84	a	0.72	a	5.63	b	249	ef
L2	0.86	a	0.74	a	5.94	ab	309	de

Table 6.6. Repeated measures analysis results for Soil 1 and Soil 2. MCv = percent soil moisture content by volume measured at time of sampling. TN = NO₃⁻-N + NH₄⁺-N. Significant MCv, treatment, and treatment-x-time interactions have been shaded (alpha = 0.05).

p values - Soil 1												
Variable	Ca ²⁺	Mg ²⁺	K ⁺	PO ₄ ³⁻	TN	SO ₄ ²⁻	Fe ³⁺	Mn ²⁺	Al ³⁺	Cu ²⁺	Pb ²⁺	Zn ²⁺
MCv	0.0190	0.2362	0.0376	0.4729	<.0001	<.0001	0.0575	0.1395	0.1857	0.0564	0.0507	0.0329
Trmt	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	0.1530	<.0001	<.0001	<.0001
Week	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Trmt x Week	<.0001	<.0001	<.0001	0.0468	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001

p values - Soil 2												
Variable	Ca ²⁺	Mg ²⁺	K ⁺	PO ₄ ³⁻	TN	SO ₄ ²⁻	Fe ³⁺	Mn ²⁺	Al ³⁺	Cu ²⁺	Pb ²⁺	Zn ²⁺
MCv	<.0001	0.0001	0.1294	0.8132	0.0008	0.0019	0.0300	0.0056	0.0009	0.3209	0.0784	<.0001
Trmt	<.0001	<.0001	<.0001	0.0029	<.0001	<.0001	0.1749	<.0001	0.2707	0.004	0.0001	<.0001
Week	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	0.0315	<.0001	<.0001
Trmt x Week	<.0001	<.0001	<.0001	0.0022	0.0014	0.0023	<.0001	<.0001	0.0003	0.0031	0.0003	<.0001

Table 6.7. Mean percent soil moisture content by volume (MCv) measured at time of sampling by treatment (all weeks combined). Values with different letters are statistically different at alpha = 0.05. T-MCv = estimated threshold MCv below which diffusivity approaches zero due to discontinuous diffusion pathways (derived from Olesen et al. 2001). A1 and A2 = low and high rate fly ash treatments, B1 and B2 = low and high rate ATB treatments, L1 and L2 = low and high rate lime treatments, C = control.

Soil 1			Soil 2		
Trmt	Mean MCv (%)		Trmt	Mean MCv (%)	
A2	14.6	a	A2	14.0	a
A1	12.6	ab	A1	12.6	ab
C	10.4	bc	B2	11.2	ab
B2	10.2	bc	B1	10.6	ab
L2	10.1	bc	C	10.2	b
L1	9.9	bc	L2	9.8	b
B1	9.2	c	L1	9.7	b
T-MCv	17		T-MCv	14	

Table 6.8. Mean element ion fluxes ($\mu\text{g } 10\text{cm}^{-2}$) by week and treatment for Soil 1. TN = NO_3^- -N + NH_4^+ -N. C = control, A1 and A2 = low and high rate ash treatments, B1 and B2 = low and high rate ATB treatments, L1 and L2 = low and high rate lime treatments.

Soil 1													
Week	Trmt	Ca^{2+}	Mg^{2+}	K^+	PO_4^{3-} - P	TN	SO_4^{2-} - S	Fe^{3+}	Mn^{2+}	Al^{3+}	Cu^{2+}	Pb^{2+}	Zn^{2+}
1	C	79	23	10	0.18	27	23	7	1.2	14	0.06	0.34	0.33
1	A1	334	38	252	0.17	19	563	13	9.3	35	0.37	1.14	0.92
1	A2	619	60	685	0.27	19	789	13	9.1	32	0.31	0.82	1.01
1	B1	272	26	11	0.16	70	56	7	1.1	13	0.24	0.70	0.35
1	B2	374	14	12	0.19	119	80	8	1.3	13	0.17	0.90	0.37
1	L1	152	58	7	0.12	24	43	7	1.6	23	0.10	0.53	0.24
1	L2	184	77	5	0.14	24	36	7	1.9	15	0.16	0.46	0.25
3	C	85	23	41	0.08	134	26	6	2.8	24	0.20	0.92	0.54
3	A1	1,425	135	423	0.22	68	1,043	11	13.5	54	0.40	1.00	1.41
3	A2	1,494	122	565	0.12	39	910	12	11.5	35	0.23	0.68	1.26
3	B1	531	24	22	0.19	117	94	8	1.9	28	0.32	1.17	0.36
3	B2	1,613	29	27	0.79	260	202	21	3.3	48	0.82	2.26	1.08
3	L1	241	125	15	0.00	93	69	9	3.3	20	0.43	1.16	0.48
3	L2	665	383	14	0.08	113	111	11	6.2	29	0.41	1.61	0.29
6	C	132	37	53	0.25	311	44	17	8.5	52	0.43	1.38	1.10
6	A1	1,772	155	253	0.42	219	1,128	14	11.3	50	0.34	0.80	1.61
6	A2	2,144	155	319	0.73	154	1,037	18	12.1	49	0.15	0.59	1.26
6	B1	2,371	45	16	0.54	776	218	46	5.1	112	1.46	2.61	1.52
6	B2	2,950	36	6	0.68	773	281	48	4.1	77	1.21	2.15	1.40
6	L1	1,219	782	13	0.52	446	156	27	6.3	62	0.71	2.49	0.51
6	L2	1,297	737	10	0.24	517	184	38	9.8	77	0.94	2.33	0.64
10	C	247	65	72	0.37	691	48	43	31.5	143	0.84	1.90	3.32
10	A1	2,275	193	167	0.25	331	1,077	21	11.0	51	0.29	0.74	1.58
10	A2	2,454	140	215	0.52	260	1,038	19	6.0	44	0.16	0.46	1.10
10	B1	2,707	51	13	0.41	868	165	53	7.5	143	1.41	2.48	1.54
10	B2	2,924	26	5	0.87	882	215	53	2.3	45	0.99	1.43	0.96
10	L1	1,644	1,023	10	0.47	689	189	62	8.5	95	1.00	2.23	0.83
10	L2	1,688	929	8	0.26	784	184	69	7.4	85	0.96	2.05	0.59

Table 6.9. Mean element ion fluxes ($\mu\text{g } 10\text{cm}^{-2}$) by week and treatment for Soil 2. TN = NO_3^- -N + NH_4^+ -N. C = control, A1 and A2 = low and high rate ash treatments, B1 and B2 = low and high rate ATB treatments, L1 and L2 = low and high rate lime treatments.

Soil 2													
Week	Trmt	Ca ²⁺	Mg ²⁺	K ⁺	PO ₄ ³⁻ P	TN	SO ₄ ²⁻ S	Fe ³⁺	Mn ²⁺	Al ³⁺	Cu ²⁺	Pb ²⁺	Zn ²⁺
1	C	88	20	18	0.30	46	20	4	2	21	0.02	0.00	0.50
1	A1	452	57	351	0.51	39	526	14	11	52	0.33	0.02	0.99
1	A2	628	66	596	0.61	39	711	13	14	47	0.12	0.02	1.37
1	B1	297	16	21	0.26	117	42	5	1	30	0.10	0.00	0.38
1	B2	343	18	17	0.47	127	37	5	1	21	0.29	0.00	0.33
1	L1	209	93	10	0.16	41	25	5	3	22	0.00	0.00	0.35
1	L2	245	113	10	0.31	49	24	5	3	21	0.07	0.00	0.39
3	C	138	33	43	0.32	203	22	12	6	49	0.15	0.02	0.95
3	A1	1,029	113	465	0.26	97	556	17	18	66	0.20	0.01	1.10
3	A2	940	95	526	0.38	116	780	21	18	56	0.16	0.01	1.32
3	B1	445	21	24	0.49	154	48	11	2	41	0.16	0.04	0.52
3	B2	1,043	31	29	0.34	281	111	19	3	55	0.30	0.07	0.80
3	L1	428	248	29	0.22	178	30	14	7	55	0.23	0.07	0.53
3	L2	588	275	17	0.30	184	62	19	12	54	0.20	0.09	0.46
6	C	181	46	70	0.69	297	22	14	10	79	0.18	0.02	1.82
6	A1	1,045	113	409	0.42	233	878	14	22	58	0.09	0.00	1.23
6	A2	1,210	115	406	0.42	180	805	15	22	57	0.15	0.01	1.27
6	B1	1,349	49	30	0.84	527	71	26	6	105	0.29	0.29	1.29
6	B2	1,969	54	29	1.23	570	89	20	4	86	0.25	0.18	1.13
6	L1	621	434	26	0.57	334	40	20	10	74	0.19	0.17	0.67
6	L2	933	559	14	0.59	397	48	29	13	60	0.21	0.12	0.64
10	C	379	81	92	0.64	569	39	26	22	124	0.16	0.20	3.37
10	A1	1,287	142	376	0.57	390	856	26	22	77	0.12	0.00	1.33
10	A2	1,845	161	325	0.79	298	1,045	19	24	64	0.07	0.00	1.63
10	B1	2,805	75	16	1.36	752	93	57	6	138	0.33	0.19	1.50
10	B2	2,762	73	17	1.39	1,008	98	41	7	103	0.36	0.16	1.79
10	L1	1,091	653	34	0.60	595	67	58	17	124	0.24	0.29	0.97
10	L2	1,131	756	20	0.87	554	89	47	13	85	0.14	0.13	0.70

CHAPTER 7

CONCLUSIONS

To paraphrase Binkley (1984), forest nutrition management draws on the fields of silviculture, soil science, ecology, and economics to provide information on how to manage the nutritional status of forests and forest soils to maintain or enhance their long-term productivity. Although nutrition management is not new, it has yet to be fully integrated into forest management planning in northeastern North America. Chapters 1 and 2 in this thesis discuss why, as we move towards a more ecosystem based approach to forest management, this needs to change.

Decades of acid deposition has resulted in base cation (Ca, Mg, K) loss and increased exchangeable Al concentrations in many forest soils across the region. Although not always immediately evident, this has likely reduced potential productivity on many sites and/or caused increased environmental stress in these ecosystems – conditions that may be exacerbated by forest harvesting and predicted climate change impacts (Chapter 1). As noted in Chapter 2, gradual rather than abrupt changes in growth limiting factors and the extended time frames associated with forest management can make these impacts difficult to recognize and quantify. However, in Nova Scotia, previous research (e.g., Ro and Vet 2002; Yanni et al. 2000; Whitfield et al. 2006), coupled with development of an ecologically based nutrient budget model (Keys et al. 2016, Chapter 2), have now allowed for stand-level estimation of sustainable mean annual increment (SusMAI) based on site-specific soil conditions, atmospheric deposition data, covertype data, and prediction of associated growth-limiting nutrients. Initial use of this model (NBM-NS) has suggested that projected yields from spruce plantations growing on slowly

weatherable soils may not be sustainable in the long-term, with Ca recognized as a major limiting nutrient (Chapter 2).

As outlined in Chapter 1, the use of liming amendments in spruce plantations may be one way to compensate for predicted Ca loss from acid deposition and harvesting, but there is little history of such amendment use in northeastern North America and none in Nova Scotia. In this study, the possibility of using ATB on spruce plantations was investigated as an alternative to the lime and wood ash amendments that have been used elsewhere. It was hypothesized that ATB could be a good source of Ca in Ca-limited sites, while also providing an environmentally sound and socially beneficial end-use for this waste-stream product.

Based on field trials of surface applied ATB at 7.5 t ha^{-1} and 15 t ha^{-1} (wet weight) under two 10-15-year old white spruce plantations, and a greenhouse comparison of ion fluxes captured in mineral soils treated with ATB, lime, and fly ash, the following conclusions can be made:

- ATB is as good (or better) a source of Ca as lime or fly ash and can be used to increase soil Ca and base cation / Al ratios in Ca-limited sites. However, as outlined below, soil and site conditions need to be considered for effective and appropriate use of ATB in a forest or plantation setting (Chapters 3-6).
- While ATB produced using CKD as the alkaline stabilizer is a good source of Ca (and to a lesser degree K), it is a poor source of Mg. Use of CKD derived ATB on sites with low soil Mg will likely cause major Ca:Mg imbalances due to imbalanced inputs and increased displacement and leaching loss of Mg. This can also be the case for K, although losses and imbalances are potentially less severe due to increased K inputs. Soils at both field sites in this study had low Mg concentrations that were exacerbated by ATB treatments (Chapters 3 and 4), and although NBM-NS does not predict Mg as a long-term limiting nutrient in Nova Scotia (Chapter 2), this does not mean Mg can be ignored when attempting to mitigate the impacts of previous cation depletion. In the case of inadequate

soil Mg, use of an additional Mg fertilizer, or possibly a mixture of dolomitic limestone and ATB, may be required. Alternatively, with sufficient demand, it should be possible to produce ATB using a blend of alkaline stabilizers and amendments specifically tailored for forestry use.

- Other soil and site features must also be considered when developing ATB treatment regimes. It is suggested that sites with loamy soils (clay > 10%) and an exchangeable H⁺ acidity of ≥ 20% can probably benefit from a single, high rate ATB application (e.g., 15 t ha⁻¹), while sites with coarse soils (clay ≤ 10%), an exchangeable H⁺ acidity of < 20%, and well to rapid drainage should be initially treated with a lower ATB rate to avoid excessive leaching loss of added base cations (Chapter 4).
- In this study, surface applied ATB resulted in significantly increased forest floor pH, but unlike some forest liming studies, this did not result in any significant increase in N mineralization, nitrification, or NO₃⁻ leaching (Chapters 3 and 4). However, this type of response is still a possibility on other sites (especially N-rich sites), and should be monitored if ATB is applied elsewhere.
- Despite significant increases in forest floor pH, ATB treatments also did not result in any appreciable impacts to ground vegetation abundance or diversity (Chapter 5). This was attributed to minimal changes in available N from ATB treatments, and to the already current dominance of pioneer and disturbance species in the juvenile plantation sites treated. This is an important finding since, due to access constraints, application of ATB in plantations would likely only take place shortly after establishment.
- Use of ATB did not greatly increase plantation tree growth over the two years of assessment (Chapter 5). This was likely due to a lack of significant N input and possibly to Mg limitations. More growth response may be detected in future on these sites, or on other sites with different conditions, but perhaps use of ATB should not be viewed in the same way as traditional fertilization. Use of liming amendments is intended to improve or restore forest soil nutrient conditions in the wake of significant human-caused

acidification and base cation loss. Maintenance of potential site productivity should be the main goal, with any immediate increase in growth or yield considered a bonus.

- Although tree growth was not greatly enhanced by ATB treatments, there were increased concentrations of Ca, K, and P found in crop tree white spruce foliage. This type of response (if it persists) needs to be incorporated into nutrient budget models like NBM-NS that use species-specific nutrient concentration values for foliage, wood, bark, and branches as model inputs. In this case, possible changes in other biomass component nutrient concentrations should also be investigated.
- As with fly ash, the risk of trace metal contamination from infrequent applications of ATB to forests appears to be quite low, due in part to the influence of increased pH on metal availability (Chapters 3, 4, and 6). In this study, post-treatment metal concentrations in mineral soils were well below acceptable limits for various land uses including agriculture, and capture of metals by ion exchange resins in surface soils was negligible.

This study has clearly shown the need for fully integrating nutrient management into forest management planning in Nova Scotia and elsewhere in the region. With respect to the current state of knowledge of Nova Scotia forest soils, the following recommendations are made:

1. Establish a province-wide, geo-statistically based soil sampling program to collect current soil chemistry data for improved nutrient budget modelling and on-going soil health monitoring. Such a program should target dominant soil series units that can then be related to established mapping and modelling tools.
2. Develop a soil health and productivity indicator system (e.g., as described by Burger and Kelting 1999) to monitor and assess (i) ongoing recovery from acidic deposition, (ii) impacts of climate change, and (iii) impacts of forest management treatments over time. Such a system should be tied to the sampling program described above.

3. Given the importance of soil weathering in nutrient budget assessments (and the uncertainties associated with these estimates), continue to evaluate and refine methodologies for estimating soil weathering inputs for dominant soil series units across the province. This type of work is particularly important in the era of climate change since potential weathering rates are directly related to climate variables.
4. Continue to evaluate and research the possible use of soil amendments (including ATB) to mitigate nutrient losses and correct nutrient imbalances in affected soils. This includes monitoring of treated sites to assess the impacts of single and/or multiple treatments over time. This type of work is needed to determine the longevity of treatment effects and their net impact on soil chemistry and tree tissue concentrations. In turn, this information can be used to (i) compare the effectiveness of different amendment types or formulations, (ii) further calibrate nutrient budget models, (iii) estimate required inputs to meet nutrient management goals, (iv) assess the economics of amendment use, and (v) develop cost-effective management regimes related to site type.
5. Regularly integrate acquired knowledge and understanding of forest soil conditions into new or existing tools and guidelines designed to promote sustainable forest management planning across the province.

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