Biogeochemical Responses to a Non-Industrial Wood Ash Addition in a South-Central Ontario Forest

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Abstract

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Recovery of forest soils from chronic acidification can be enhanced with the use of non-industrial wood ash (NIWA). Non-industrial wood ash is alkaline and contains high concentrations of macronutrients, but trace metal concentrations must be evaluated to limit risk of metal toxicity following application. Additionally, understanding how different forest ecosystem components respond to NIWA is essential to inform current policy regulating its use as a soil amendment. This study evaluated the response of sugar maple (*Acer saccharum*) sap yield and chemistry, the response of soils beneath maple, American beech (*Fagus grandifolia*) and mixed species canopies, and maple and beech fine roots, foliage, seedling abundance, and understory vegetation abundance and composition to an application of 6 Mg ha^{-1} NIWA. Eight 40 x 40 m plots were established in a hardwood stand in Bracebridge, Ontario and were sampled prior-to and up to two years following application of NIWA (n = 4). Non-industrial wood ash significantly increased organic horizon soil pH and macronutrient (Ca, Mg, and K) concentrations with increases in Mg and K extending to the mineral soils. Significantly higher concentrations of some trace metals (Al, Fe, Mn, Cd, Cu, Pb, Zn) were also observed, but these were restricted to the organic horizons. Sugar maple sap, pH, and sweetness were unaffected by NIWA application, and while increases were observed in nutrient and metal concentrations in sap, the differences were small and variable between years, and all concentrations were consistent with those commonly found in maple sap. Fine root biomass of maple and beech trees was not affected by NIWA application, but higher concentrations of K and Mg were observed in the roots of both species, consistent with higher concentrations observed in the mineral soil horizons beneath both species' canopies. Only significant increases were observed in K in sugar maple foliage. Both critical foliar concentrations and diagnosis and recommendation integrated system (DRIS) norms for sugar maple did not indicate mineral nutrient deficiencies at this site; although this site was acidic and nutrient-poor, this may account for the lack of differences observed, particularly between species. Changes observed in understory vegetation were driven by years rather than between treatments. These results suggest that moderate doses of NIWA can provide significant decreases in soil acidity and increase nutrient availability, with limited increases in metal concentrations that are primarily restricted to the organic horizons.

Keywords: non-industrial wood ash, NIWA, metal toxicity, sugar maple, sap yield, sap sweetness, American beech

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List of Abbreviations

Adenosine triphosphate	ATP
Aluminum	Al
Ammonium chloride	NH_4Cl
Arsenic	As
Basal Area Increment	BAI
Beech Bark Disease	BBD
Below detection limit	BDL
Boron	B
Calcium	Ca
Calcium chloride	$CaCl_2$
Cadmium	Cd
Carbon	C
Carbon dioxide	CO_2
Concentration of regulated Metals	CM
Copper	Cu
Diagnosis and Recommendation Integrated System	DRIS
Diameter at Breast Height	DBH
DW	Dry weight
Exchangeable Cations	EC
FH	Fibrous-humic
Friends of the Muskoka Watershed	FMW
Hydrogen ion	H^+
Inductively Coupled Plasma-Optical Emission Spectrometer	ICP-OES
Industrial Wood Ash	IWA
Iron	Fe
Lead	Pb
Loss on Ignition	LOI
Magnesium	Mg
Manganese	Mn
Ministry of the Environment, Conservation and Parks	MECP
Molybdenum	Mo
Nickel	Ni
Nitric acid	HNO_3
Nitrogen	N
Nitrogen oxides	NO_x
Non-Agricultural Source Material	NASM
Non-Industrial Wood Ash	NIWA
Organic matter	OM
Potassium	K
Selenium	Se
Shannon Diversity	H
Simpson's Diversity	D
Sodium	Na
Species richness	S_r
Sulfur	S

Sulfur dioxide..SO_2
Phosphorous..P
Volume-weighted..VW
Wollastonite..$CaSiO_3$
Zinc..Zn

1. General Introduction

1.1 Global Bioenergy, Biomass, and Soil Amendments

Global change has rapidly altered the boreal and temperate forest environments (Gauthier et al., 2015; Millar & Stephenson, 2015). Acidic deposition has accelerated base cation leaching resulting in nutrient-poor, acidified soils that are less suitable for sustaining sensitive native plant life and biodiversity (Likens et al., 1996; de Vries et al., 2014; Caputo et al., 2016). Nutrient losses are further exacerbated by activities such as harvesting (Phillips & Watmough, 2012). Whilst emissions of sulfur dioxide (SO_2) and nitrogen oxides (NO_x) have been drastically reduced in North America since the 1990's (ECCC, 2022), the legacy effects of acidic deposition are persistent, and natural chemical recovery of soils and surface waters is either slower than expected or not evident (Watmough et al., 2016; Ott & Watmough, 2022). Thus, intervention using soil amendments may be necessary to reverse symptoms of soil acidification.

With international interest shifting away from fossil fuels, there has been increasing focus on bioenergy (Popp et al., 2021). In 2019, biomass was the primary renewable energy consumed globally, accounting for 12% of the renewable energy supply (WBA, 2022), and it is predicted to increase in demand (Titus et al., 2021) and remain as the most used renewable energy source (Popp et al., 2021). Of the global biomass use and supply in 2015, wood accounted for 39%, and of that waste production accounted for 8% (Popp et al., 2021). In the interest of creating a circular economy, biomass residues such as wood ash have been proposed as forest soil amendments with the potential to replenish nutrients and divert waste from landfills (Tan & Lamers, 2021).

1.2 Global Use and Production of Wood Ash

1.2.1 *Global Production of Wood Ash*

Wood ash production has increased with an accelerated demand for bioenergy (Huotari et al., 2015; Titus et al., 2021). In 2013, estimates suggested that globally, 476 million Mg of biomass ash are produced annually (Vassilev et al., 2013) and while management practices and disposal of wood ash vary widely, the most common destination is landfills (Lamers et al., 2018). In Finland only 18% of the wood ash produced in 2004 was used as a forest amendment, and in Sweden, despite producing approximately 300 000 Mg per year, only 5% of that is recycled to forests (Stupak et al., 2007). In Austria, approximately 44% of all biomass ash produced is landfilled with no mention of recycling practices with the remaining ash (Lamers et al., 2018), and in the United States (US) around 90% of wood ash produced is landfilled (Pitman, 2006). In Canada approximately 420,000 Mg of wood ash is produced annually as of 2013 (Elliott & Mahmood, 2015). British Columbia and Quebec have the most number of mills that generate roughly 140,000 and 130,000 Mg per year, respectively (Elliott et al., 2022); it is estimated that 63% of all wood ash produced in Canada is landfilled (Elliott et al., 2022).

1.2.2 *Wood Ash Properties*

Wood ash is created as a by-product from the combustion of woody biomass such as woodchips, sawdust, bark, and softwood (Siddique, 2012) and it can originate from both industrial and non-industrial sources. Industrial wood ash (IWA) is produced primarily from forest and industrial (e.g. pulp and paper mills) residues (Elliott et al. 2022), while non-industrial wood ash (NIWA) is generated from wood-fired ovens, furnaces, and fireplaces in homes and small businesses (Azan et al., 2019). Because industrial sources generally have more consistent combustion conditions and source material (Pitman, 2006), IWA properties may be less variable in nature than NIWA. Ash mass loss and fluctuations in nutrient and metal concentrations have been demonstrated as both temperature- and species-dependent (Misra et al., 1993; Pitman, 2006; Deighton & Watmough, 2020).

Since wood ash properties are primarily determined by the type of feedstock and combustion temperature, the chemical composition of NIWA can vary considerably depending on the source of the ash (Misra et al., 1993; Pitman, 2006). Typically, NIWA pH ranges from 8 – 13 and thus is effective at neutralizing acidity in soils (Augusto et al., 2008). Additionally, not only is NIWA approximately 30% Ca by dry mass, but it contains other macronutrients (magnesium (Mg), potassium (K), and phosphorous (P)) and micronutrients (boron (B), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), and molybdenum (Mo)) that are essential for plant and tree growth (Azan et al., 2019; Deighton & Watmough, 2020). In contrast, other soil amendments such as lime often contain high concentrations of Ca and Mg but lack substantial quantities of other important nutrients (Lundström et al., 2003; Kim et al., 2022) resulting in wood ash as an appealing alternative. Wood ash, however, contains relatively high concentrations of trace metals that need to be evaluated to ensure they do not exceed provincial regulatory guidelines for safe land application.

In Ontario, wood ash is classified as a non-agricultural source material (NASM; Government of Ontario, 2002). Non-agricultural source materials are highly controlled and require strict regulatory approval prior to application as a soil amendment (Hannam et al., 2018). Both the cost and time required to obtain approval may disincentivize initiatives to recycle wood ash because landfilling is an easier and more accessible alternative (Hannam et al., 2018). Nonetheless, analysis of the chemical composition of NIWA is lacking and therefore necessary to ensure metal concentrations do not exceed levels for safe application. Land application guidelines are split into two categories (CM1 and CM2) for the content of regulated metals (CM) for NASMs. Application is mostly unrestricted if metal concentrations fall below CM1 guidelines, restrictions increase between CM1 and CM2, and application is not allowed if any concentrations are above CM2 guidelines (Government of Ontario, 2002). Though the chemical composition of NIWA can be more variable because of the nature of its production, research suggests that amalgamating NIWA from many sources can help dilute any source-specific properties and increase consistency in both nutrient and metal concentrations (Azan et al., 2019; Deighton & Watmough, 2020; Syeda et al., submitted).

Sources and production of NIWA are less studied across Canada, but Azan et al. (2019) estimated that approximately 18,000 Mg are generated each year in Ontario with the majority of it ending up in landfills because of its designation as a waste material (Hannam et al., 2018; Azan et al., 2019). Therefore, increasing accessibility to recycling options for wood ash could have a large impact on reducing the amount sent to landfills while also providing supplementation to nutrient-poor and deficient soils from chronic acidification (Augusto et al., 2008). In an analysis of potential beneficial uses for wood ash (application to agricultural land, forest soil, or use in forest roads or concrete) Gaudreault et al. (2020) found that all scenarios resulted in better environmental indicator scores than landfilling, and wood ash additions have been shown to reduce greenhouse gas emissions in a spruce forest in Sweden (Klemedtsson et al., 2010).

1.3 Effect of Wood Ash on Soils

The neutralization effect of wood ash is well-recognized in the literature and it is often accompanied by increases in base saturation, both of which are positively correlated with ash dose (Demeyer et al., 2001; Pitman, 2006; Augusto et al., 2008; Reid & Watmough, 2014). Decreases in organic horizon acidity are commonly seen shortly after wood ash application (Saarsalmi et al., 2004; Ozolinčius et al., 2007b; Deighton & Watmough, 2020), while reductions in mineral soil acidity tend to materialize over the longer-term (Saarsalmi et al., 2001, 2004). In a similar manner, wood ash increases macronutrient (Ca, Mg, K, P) concentrations (Saarsalmi et al., 2012; Deighton & Watmough, 2020; Arseneau et al., 2021) that can be sustained up to 30 years post-application (Augusto et al., 2008; Saarsalmi et al., 2012). Increases in the abundance of ammonifying, denitrifying organisms in the litter layer (Ozolinčius, et al., 2007b) and in soil microbial biomass (Saarsalmi et al., 2012) may also increase N mineralization and availability (Pitman, 2006).

Elevated trace metal concentrations have also been observed, but these are largely restricted to the organic soil horizons (Augusto et al., 2008) because increases in soil pH reduce the solubility and availability of the metals to biota (Bramryd & Fransman, 1995; Violante et al., 2010). In an experiment conducted by Deighton et al. (2021) there was no evidence to suggest enhanced metal leaching during rain events even following simulated drought conditions and therefore metal mobility may be restricted even with reductions in soil pH that may occur over time. Persistent elevated trace metal concentrations have also been reported as within the normal range of concentrations expected in the soils (Saarsalmi et al., 2012).

1.4 Effects of Wood Ash on Fine Roots

1.4.1 *Fine Root Biomass*

Research has primarily suggested that decreases in fine root biomass occur following wood ash application (Helmisaari et al., 2009) and fertilization treatments (Persson & Ahlström, 1990; Clemensson-Lindell & Persson, 1995). Additionally, decreases in the total length and number of root tips (Ozolinčius et al., 2007b) have accompanied increases in fine root necromass (Clemensson-Lindell & Persson, 1995; Helmisaari et al., 2009). However, Clemensson-Lindell and Persson (1995) reported an increase in specific root length and Helmisaari et al. (2009) have observed deeper fine root distribution following wood ash treatment. Despite the variability in responses of fine root biomass to wood ash and other fertilizer treatments, the observed decreases do not appear to be significant enough to impact tree growth in the long-term (Helmisaari et al., 2009)

1.4.2 *Fine Root Chemistry*

The chemical composition of some elements in fine roots has been closely linked to changes in soil chemistry following ash application. For example, increased concentrations in fine root Ca and Mg corresponded with respective increases in soil concentrations, and decreases in fine root Mn were also correlated with decreases in extractable soil Mn concentrations (Brunner et al., 2004). Higher K concentrations have also been observed in fine roots following wood ash application, though these were not correlated with soil concentrations (Brunner et al., 2004). Additionally, significant increases were observed in Ca, Mg, and K concentrations in sugar maple (*Acer saccharum* Marshall) seedling roots after application of sugar maple, white pine (*Pinus strobus* Linnaeus), and yellow birch (*Betula alleghaniensis* Britton) NIWA (Deighton &

Watmough, 2020). While some increases were observed in metal concentrations in sugar maple seedling roots these were mostly in response to yellow birch NIWA that contained higher than average wood ash metal concentrations (Deighton & Watmough, 2020). In contrast, Brunner et al. (2004) found no increases in root metal concentrations following wood ash application suggesting that while metals may increase in the soils they remain largely unavailable for plant uptake.

1.5 Effects of Wood Ash on Ground Vegetation

Generally, community composition and abundance of ground vegetation is not significantly altered following wood ash addition (Jacobson & Gustafsson, 2001; Arvidsson et al., 2002; Ozolinčius et al., 2007b; Augusto et al., 2008). However, in one particular case, wood ash addition caused a complete substitution of ground vegetation in an originally afforested, drained peatland in Finland 50 years after application (Moilanen et al., 2002). Damage to mosses, lichens, and bryophytes have also been observed (Ozolinčius et al., 2007a; Økland et al., 2022), but these species tend to recover quickly (Jacobson & Gustafsson, 2001). It is important to note that responses of ground vegetation can be driven by a variety of factors, such as soil type, baseline fertility, ash dosage and type, and timeline of the study (Aronsson & Ekelund, 2004; Augusto et al., 2008).

1.6 Sugar Maple

Sugar maple is a keystone species in eastern North American forests and is highly valued for its maple products and sap production. Canadian maple product exports averaged \$512 million annually from 2018 – 2022 and in 2022 maple syrup production was responsible for 78% of the global supply (AAFC, 2023). Reports of sugar maple dieback began as early as the 1950's in the northern portion of its range in the US (Bal et al., 2015), and notable occurrences have been documented since the 1940's in Ontario (Watmough et al., 1999; Tominaga et al., 2008), Quebec (Duchesne et al., 2002, 2005), Pennsylvania (Kolb & McCormick, 1993; Horsley et al., 2000), and New York (Sullivan et al., 2013; Bishop et al., 2015), Vermont (Wilmot et al., 1995*b*; Schaberg et al., 2006), and New Hampshire (Hallett et al., 2006). In many cases these declines have been associated with soils that are particularly susceptible to accelerated base cation leaching and increases in metal concentrations such as aluminum (Al) from acidic deposition (Horsley et al., 2000). Sugar maple also exhibit a high demand for soil calcium (Ca; Horsley et al., 2000; Hallett et al., 2006; Long et al., 2009) and are thus more vulnerable in shallow, coarse-textured soils with lower critical loads of acidity and reduced buffering capacity (Ouimet et al., 2006).

1.6.1 *Effect of soil amendments and wood ash on sugar maple trees*

Sugar maple have exhibited positive responses to soil amendments such as lime or other forms of Ca addition in eastern North America. Research with single doses ranging from 0.5 – 50 Mg ha^{-1} have shown marked decreases in crown dieback, significant increases in foliar nutrient concentrations (Ca, K, Mg, and P), and improvements in crown vigor, basal area increment (BAI), wound closure, and seedling regeneration from 1 – 23 years following application (Wilmot et al., 1996; Long et al., 1997; Juice et al., 2006; Moore & Ouimet, 2006; Huggett et al., 2007; Moore & Ouimet, 2010; Long et al., 2011; Moore et al., 2012; Moore & Ouimet, 2021). While sugar maple has a high demand for Ca, some studies have demonstrated the importance of other base cations (such as Mg) that may be critical to their health noting enhanced positive responses when adding base cation fertilizers in addition to lime (Wilmot et al., 1996; Moore & Ouimet, 2010). Wood ash provides a compelling alternative for treatment of nutrient deficiency in sugar maple because of its high concentrations of Ca accompanied by other essential nutrients.

More specifically, sugar maple also respond positively to wood ash addition and exhibit little risk of metal toxicity. For example, accompanying an increase in soil pH and base cation concentrations, Arseneau et al. (2021) reported a 20% increase in sugar maple mean BAI as well as a decrease in foliar Ca deficiency and excess nitrogen (N) three years following 20 Mg ha^{-1} IWA application. Additionally, Deighton and Watmough (2020) reported increases in foliar Ca and K and root and stem Ca, K, and Mg concentrations in sugar maple seedlings one year following 6 Mg ha^{-1} NIWA application. Increased metal concentrations were noted in the roots and shoots of sugar maple seedlings, but these were not associated with any negative effects on seedling growth (Deighton & Watmough, 2020). Similarly, while no differences were observed in the foliar metal concentrations of sugar maple seedlings, significant increases were observed in some metals in seedling roots four years after application of 4 and 8 Mg ha^{-1} IWA. As a result, the authors recommended doses of less than 8 Mg ha^{-1} to minimize the risk of metal toxicity (Deighton et al., 2021).

To my knowledge there is no literature discussing the effects of wood ash specifically on sugar maple sap, and the response of sap to other soil amendments is quite variable. Moore et al. (2020) reported that liming indirectly improved sap yield in a base-poor stand in Quebec eighteen years after application suggesting that the positive effect of liming on tree growth indirectly led to increased sap yield in the long-term. In New Hampshire, N fertilization (30 kg ha^{-1} $year^{-1}$) was associated with sweeter sap two years after initial application. On the other hand, lime and fertilizer treatments have been found to have no effect on sap yield five years after application in Ontario (Noland et al., 2006) and no effect of various base cation fertilization treatment was found on sap sweetness in Vermont (Wilmot et al., 1995*a*). Given the scale and importance of maple syrup production in Canada, there is a need to determine whether there are benefits or possible risks associated with the application of wood ash to sugarbushes.

1.7 American Beech

American beech (*Fagus grandifolia* Ehrhart) are another dominant species in late-successional hardwood forests (McLaughlin & Greifenhagen, 2012) which have expanded in range by exploiting the dieback of sugar maple in northeastern North America (Duchesne et al., 2005; Duchesne & Ouimet, 2009). Compared with sugar maple, beech trees are more tolerant of low soil pH and base cation concentrations as well as higher concentrations of Al (Park & Yanai, 2009; Duchesne et al., 2013), and they tend to be more resilient to extreme weather events (Nolet & Kneeshaw, 2018). Additionally, American beech seedlings are less sensitive to fluctuating nutrient availability than sugar maple seedlings, which has contributed to their successful and preferential regeneration (Park & Yanai, 2009). However, beech bark disease (BBD) is of increasing concern as it is estimated to spread at a rate of 14.7 – 16.3 km $year^{-1}$ (Morin et al., 2007; Evans & Finkral, 2010) and will likely cover most, if not all of the beech geographical range within the next 50 years (Morin et al., 2007). Beech bark disease is a combination of wounding by a scale insect (the non-native invasive beech scale, *Cryptococcus fagisuga* Lindinger and the native scale insect *Xylococculus betulae* Pergande) and subsequent infection by a canker fungus (*Neonectria spp.*) leading to tree mortality as early as one year following fungal establishment (Cale et al., 2017). Because beech can reproduce vegetatively through root suckering, BBD-induced tree mortality can lead to prolific sprouting (Duchesne et al., 2005; Duchesne & Ouimet, 2009) that may interfere with sugar maple seedling survivorship (Hane, 2003).

1.7.1 *Effect of wood ash on American beech trees*

In the few studies available, American beech have largely shown no response to soil amendments and currently there is no research assessing the response of beech to wood ash treatment. No effect has been found on American beech BAI, crown vigor, mortality or sapling growth following either lime or Ca addition (Long et al., 1997, 2011; Duchesne et al., 2013; Long et al., 2022) and both no effect (Duchesne et al., 2013) and a negative effect (Moore et al., 2008, 2012) have been found on beech regeneration following lime treatment. American beech have, however, exhibited increased growth rates on Al-treated plots (Halman et al., 2015). As a result, it is important to determine whether American beech trees will exhibit any responses to NIWA addition, and whether this addition may favour sugar maple seedlings in areas where beech have expanded in response to sugar maple dieback.

1.8 Research Objectives and Hypotheses

The goal of this thesis was to investigate the effect of non-industrial wood ash applied to a base-poor, sugar maple-dominated stand in Muskoka, Ontario through two separate research chapters. The first research chapter (Chapter 2) aims to understand the short-term (two year) effect of NIWA on sugar maple sap chemistry. Non-industrial wood ash samples were donated by community members of Muskoka, Ontario and were amalgamated prior to application. In this chapter we analyzed a) the chemical composition of NIWA, and how it influenced b) soil chemistry (pH, LOI, nutrients and metal concentrations), c) the quality of sugar maple sap (pH, sweetness, nutrient and metal concentrations), and d) sugar maple foliar nutrient and metal chemistry. The second research chapter (Chapter 3) evaluated the short-term (one year) response of sugar maple and American beech trees to NIWA addition. In this chapter we looked at a) the response of soil chemistry beneath both sugar maple and American beech tree canopies (pH, organic matter, nutrient and metal concentrations), b) variability in maple and beech roots (biomass and nutrient and metal concentrations), c) foliar chemistry (nutrients and metals), and d) the response of understory vegetation (maple and beech seedling and overall species abundance and composition).

1.8.1 *Chapter 2 Hypotheses*

The objective of this research chapter was to determine how NIWA influences soil chemistry beneath sugar maple canopies, maple sap sweetness, and maple sap and foliar chemistry. It was hypothesized that application of an alkaline NIWA to forest soils would immediately (<one year) increase pH and nutrient (Ca, K, Mg, P) concentrations in the organic soil horizons with limited increases in metals (Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, Zn) due to their retention in the organic layer. It was also expected that nutrient concentrations would increase in sugar maple foliage without a corresponding increase in metals. No short-term change was expected in sugar maple sap yield, pH, sweetness, or chemistry.

1.8.2 *Chapter 3 Hypotheses*

The objective of this research chapter was to determine whether sugar maple or American beech respond differently to an application of NIWA by evaluating soil, root, and foliar chemistry, root biomass, and understory vegetation. It was hypothesized that NIWA would immediately (<one year) decrease soil acidity and increase nutrient availability in the organic horizons, roots, and foliage of both species. It was also expected

that metal concentrations would increase in the upper organic soil horizons, but their mobility would be limited in soils with a higher pH and therefore these increases would not be mirrored in the roots or foliage of either species. It was predicted that fine root biomass would decrease following treatment because of increased nutrient availability. Lastly, it was expected that sugar maple seedling abundance would increase as soil acidity decreased and nutrients became more available in the soil, while American beech seedling abundance would not be affected because of their tolerance for more acidic soils that may contain lower concentrations of nutrients.

1.9 Significance of research

With increasing biomass production and a global push to reduce our reliance on fossil fuels and create a circular economy, reducing waste is of paramount importance. Additionally, though acidic deposition has been largely eliminated, forest soils are still exhibiting symptoms of soil acidification, suggesting that natural recovery needs supplementation in the form of soil amendments. Through the use of non-industrial wood ash to base-poor forest soils, both goals can be achieved. Non-industrial wood ash has the potential to fertilize forest soils whilst not compromising forest flora health or the quality of economically and culturally important maple syrup. Nevertheless, wood ash contains trace metals that are regulated for safe application and therefore must first be investigated within these contexts to ensure forest ecosystem health and sugar maple syrup quality. Ultimately, this research will provide valuable information for proper forest and sugarbush management in Ontario and areas of similar forest ecosystems.

2. Sugar Maple (*Acer saccharum*) Sap, Soil, and Foliar Chemistry in Response to Non-Industrial Wood Ash Fertilizer in Muskoka, Ontario

2.1 Abstract

Non-industrial wood ash may be an effective forest soil nutrient supplement but its use in Canada is largely restricted because of unknown concentrations of trace metal contaminants. Sugar maple (*Acer saccharum* Marshall) is particularly sensitive to low soil calcium (Ca) levels, and though maple syrup is of great economic importance in Canada, it is unknown how wood ash could affect sap chemistry. Non-industrial wood ash (NIWA; 6 Mg ha^{-1}) applied to experimental plots in Muskoka, Ontario was rich in Ca (27%) while metal concentrations were well below provincial regulatory limits. One-year post-application, soil pH and base cations (Ca, K, Mg) significantly increased in the litter and FH horizons, and metal concentrations significantly increased in the litter. Sap yield was significantly lower in the control plots in the first year compared to the second-year, post-application, but no other differences were found. In both tapping years sap sweetness remained similar and differences in nutrient and metal concentrations between treatments were generally small and inconsistent. Foliar chemistry remained largely unchanged one year following application except for K, which was twice as high in the treated plots. Therefore, NIWA is unlikely to significantly alter sugar maple sap chemistry indicating it is a viable nutrient supplement that can enhance soil fertility in sugar bushes with no impact on sap sweetness.

2.2 Introduction

Despite reductions in acidic deposition since the Canada-United States Air Quality Agreement in 1991 (Driscoll et al., 2001; ECCC, 2022) the pace of natural chemical recovery in soils and surface waters has been slow, largely due to historic depletion of exchangeable base cation pools in soils and slow mineral weathering rates (Likens et al., 1996; Watmough et al., 2016; Johnson et al., 2018). Sugar maple (*Acer saccharum* Marshall) is particularly sensitive to low concentrations of soil calcium (Ca; Schaberg et al., 2006; St. Clair et al., 2008) and numerous studies have reported decreased vigor, canopy condition, growth rates, and seedling recruitment in sugar maple growing on nutrient depleted soils (McLaughlin & Wimmer, 1999; Driscoll et al., 2001; Duchesne et al., 2002; Sullivan et al., 2013).

Soil amendments including lime, wood ash, and wollastonite have reported beneficial effects on sugar maple (Wilmot et al., 1996; Juice et al., 2006; Moore et al., 2015; Arseneau et al., 2021; Moore & Ouimet, 2021). Increases in soil pH and concentrations of essential macronutrients such as Ca, magnesium (Mg), potassium (K), and phosphorous (P) can persist in the soil and foliage up to 23 years after initial liming treatment (Juice et al., 2006; Long et al., 2011; Moore et al., 2012; Moore & Ouimet, 2021). Soil amendments have led to increased vigor, crown health, growth rate, recruitment of seedlings, and wound closure on sugar maple trees (Wilmot et al., 1996; Houle et al., 2002; Juice et al., 2006; Huggett et al., 2007; Moore & Ouimet, 2010; Long et al., 2011; Deighton & Watmough, 2020; Arseneau et al., 2021).

The impact of neutralizing soil amendments on sugar maple sap production has not been widely studied despite its substantial economic value in Canada and the northeastern

United States. Canada is the largest producer of maple syrup accounting for 78% of global production in 2022 and averaging 79 million kilograms of maple syrup produced annually from 2018 – 2022 (AAFC, 2023). Previous studies evaluating combinations of fertilizer additions have reported mixed results on sap yield and sweetness, defined here as sucrose concentration. For example, Wilmot et al. (1995a) reported that fertilizer additions (primarily Ca, Mg, and K) in northern Vermont did not have an effect on sap sweetness two years after application. Similarly in eastern Ontario, lime, K and P fertilization treatments did not affect sap yield or sap sweetness (Noland et al., 2006). In New Hampshire, N fertilization has increased sap sweetness two years after application, but foliar P was negatively correlated with sweetness and no relationship was observed between Ca addition and sweetness (Wild & Yanai, 2015). On the other hand, Moore et al. (2020) found that liming improved yield and increased sap sweetness up to 20% eighteen years after a single application in Quebec (Moore et al., 2020). Since sap becomes syrup when it is boiled down to approximately 66-67% sucrose and 33-34% water (Ball, 2007) the total volume of maple syrup produced is largely dependent on the initial concentration of sucrose, underlying the importance of evaluating sap sweetness with respect to overall yield.

Wood ash is produced from the combustion of woody biomass such as sawdust, woodchips, bark, and stem wood (Siddique, 2012) and can be classified as either industrial or non-industrial. Industrial wood ash (IWA) is produced from industrial sources, such as the pulp and paper and wood processing industries (Elliott et al., 2022). In contrast, non-industrial wood ash (NIWA) is produced from residential sources (homes or local businesses), using small, wood-fired ovens, furnaces, or fireplaces (Azan et al., 2019).

Because wood ash properties are largely determined by the source material (species and tree parts burned), source origin, and combustion conditions (Pitman, 2006; Deighton & Watmough, 2020), large variations are observed in NIWA chemistry, particularly when produced from species-specific feedstock (Deighton & Watmough, 2020). However, when NIWA is amalgamated from many sources and types of feedstock it exhibits a much greater consistency in chemistry (Syeda et al., submitted).

Wood ash has been used widely in Europe (Lundström et al., 2003; Pitman, 2006) but only sparingly in Canada due to regulation limitations (Hannam et al., 2018). In Ontario, wood ash is currently classified as a non-aqueous, non-agricultural source material (NASM). Non-aqueous, non-agricultural source materials are regulated to ensure soils do not exceed critical levels of metals and can be classified as unrestricted (CM1) or restricted (CM2) based on metal concentrations (Government of Ontario, 2002). As metal concentrations increase, the restrictions on NASM application (e.g. proximity to any water source) become more limiting, to the extent that the material cannot be applied as a NASM if the concentration of any regulated metal is above CM2 guidelines (Hannam et al., 2016).

Each year Ontario alone produces approximately 18,000 Mg of NIWA that could be diverted from landfills (Azan et al., 2019). Wood ash is highly alkaline with a pH of 8.9–13.5 and high concentrations of Ca, K, Mg, and P making it a good candidate for fertilization of acidic, nutrient-depleted soils, but trace metal concentrations such as cadmium (Cd) and zinc (Zn) that are toxic in high concentrations must be monitored (Demeyer et al., 2001). Azan et al. (2019) found that the composition of mixed-hardwood NIWA samples (n = 10) averaged 30% Ca and all metal concentrations were below CM1, except copper (Cu) and Zn that were marginally above CM1 but well below CM2.

Additionally, NIWA samples produced separately from sugar maple, white pine (*Pinus strobus* Linnaeus), and yellow birch (*Betula alleghaniensis* Britton) each contained metal concentrations below CM1 guidelines, except for Cd, Cu, Zn, and selenium (Se) in yellow birch that were above CM1 but below CM2 (Deighton & Watmough, 2020). In south-central Ontario sugar maple trees account for approximately 75% of the species composition in hardwood stands (Tominaga et al., 2008) emphasizing the need to monitor sap yield and chemical properties to evaluate whether NIWA affects overall sap quality.

Considering these knowledge gaps, the objective of this work was to quantify the chemistry of NIWA and its effect on the soils, foliage and sap yield and sugar content of sugar maple trees over the short-term (2 years). It was hypothesized that NIWA would increase the pH and nutrient availability (particularly Ca, Mg, and K) in the organic soil layers (L, FH) and foliage with a limited increase in trace metals due to their low concentrations. It was also expected that there would be no immediate effect on sap yield, pH, chemistry, or sweetness.

2.3 Methodology

2.3.1 *Study Site*

The study forest is in the mixed-wood Great Lakes-St. Lawrence ecozone east of the town of Bracebridge, Ontario, Canada (45°03'45.27" N, 79°08'43.62" W) at an elevation approximately 282 m above sea level (ECCC, 2023a). The average annual temperature is 5.2°C and the average annual precipitation is 1105 mm measured over a 30-year period (1981 – 2010; ECCC, 2023a). The coarse-textured soils are shallow and typically poorly developed podzols and brunisols overlaying Precambrian gneiss and other metamorphic rock (AAFC, 1998). While the soils are acidic (pH_{CaCl_2} 4.1) and nutrient-poor, critical foliar concentrations (Table 2.4; Table 2.5) and diagnosis and recommendation integrated system (DRIS) norms (Table 2.5) indicated that Ca, Mg, K, and P are within the critical limits for sugar maple trees and therefore do not suggest mineral nutrient deficiencies. In 2020, mean basal area in the study site for trees > 10 cm diameter at breast height (DBH, 1.3 m) was 24.9 m^2 ha^{-1}. The forest is uneven-aged and dominated by sugar maple trees (77% of total plot basal area). It is located on a ~100 ha stand operated by Camp Big Canoe that has been preserved since 1968 with no logging or harvesting and strict environmental policies limiting waste and preserving air, water, flora, and fauna quality (Casey, 2021). Water quality in the lake has also been monitored and is similar to other surface waters in the region, with relatively low pH (~5.8 to 6.2 pH units) and Ca concentrations (2.43 mg L^{-1}; Reid & Watmough, 2016). The Camp Big Canoe location is also far from roads and urban areas that eliminates potential road salt or other contamination effects.

2.3.2 *Plot Setup and Experimental Design*

Using a randomized plot design, eight 40 × 40 m plots with a 10 m buffer were established within a 10-ha area at the study forest in early September 2020. Plot areas were selected to satisfy the following conditions: dominated by sugar maple, a minimum 60 m away from any watercourse, and a flat to gentle slope to avoid potential run-off of ash after application (Hannam et al., 2016). One treatment of 6 Mg ha^{-1} NIWA was replicated four times and the remaining four plots were left as controls.

2.3.3 *Field Sampling and Ash Application*

Baseline soil sampling was conducted at the end of September 2020. Samples were collected beneath three sugar maples trees within each plot; each tree was selected to be greater than 10 cm DBH. Three samples were collected from each location within each plot (*n* = 36 per treatment). Grab samples were taken from the litter (L) and fibrous-humic (FH) horizons, and an auger was used to sample the upper mineral (0-10 cm) soil that contained the Ah-horizon (AAFC 1998).

Non-industrial wood ash was contributed by volunteer Muskoka residents and collected by the Friends of the Muskoka Watershed charitable organization (FMW, 2023). At the time of collection ash from all sources was homogenized and sieved (<2 mm) to remove charcoal and large debris such as nails or plastic. In a questionnaire assessing the origins of mixed NIWA donated to the FMW, respondents (*n* = 47) indicated that hardwoods accounted for most of the species burned. Approximately 70% of respondents burned maple species (*Acer* spp.), 50% burned birch (*Betula* spp.), and/or 28% burned oak (*Quercus* spp.). Trunk wood (85%) was the primary part burned, followed by the branches (74%), and/or the bark (70%; Syeda et al., submitted). Once collected and sieved, the ash was stored in a cool, dark environment in large, polyethylene containers prior to application in November 2020. Following approval of the site, ash-handling, and application rate by the Ministry of the Environment, Conservation, and Parks (MECP), ash was weighed and transported to each treatment plot in 10 kg buckets and then applied by hand to ensure a relatively even distribution. Ash was applied at a dose of 6 Mg ha^{-1} (average 2.7% moisture) to the four treatment plots. During application, six sub samples were collected randomly from the ash brought to each plot and kept separate for analysis (*n* = 24).

On February 26, 2021, and February 25, 2022, three sugar maple trees in each plot were tapped for sap collection (*n* = 12 per treatment). Trees selected for tapping had no obvious wounds and were greater than 25 cm DBH to sustain at least one tap. Trees in the control plots averaged 40.7 (± *SD* = 8.3) cm DBH and trees in the treatment plots averaged 49.8 (± *SD* = 7.0) cm DBH. In 2021, one hole was drilled at waist height on an upward angle (approximately 10°) for gravity collection, and to a depth of 3.8 cm on the south side of the tree using a 19/64 drill bit (Perkins et al., 2022). The second year a second hole was tapped using the same method but was located 10 cm on the west side and 15 cm above the tap from the previous year. A 5/16 plastic spile was inserted into the tap hole and 5/16 tubing was attached feeding into a 10 kg bucket with a lid (Perkins et al., 2022). The buckets were then fastened around the tree using metal wiring and S-hooks. Buckets were lined with plastic bags, which were replaced at each sampling to avoid contamination between sap samples for chemical analysis. Sap sampling seasons were from March 20-May 4 in 2021 and March 18-April 27 in 2022. Sap yield was measured at least once per week during the sampling season and occasionally more frequently when yield was high. Yield data were generated using a 2 L graduated cylinder. Sap samples were transferred to 50 mL Falcon tubes and frozen until analysis.

Post-application soil sampling was conducted in July 2021; soils were collected in the same manner as the baseline sampling described above. Foliage samples were also collected from each sugar maple tree where the soil samples were collected. Foliar samples were retrieved using extendable pole pruners from branches receiving direct sunlight and were composited per plot ($n = 4$).

2.3.4 *Laboratory Sample Analysis*

2.3.4.1 Soil and Ash Analysis

Soil and ash samples were oven dried for 24 hours at 110°C. Once dry, L and FH samples were ground using a Wiley Mill and mineral samples were sieved (<2 mm) to prepare for analysis. All samples were analyzed for pH and loss-on-ignition (LOI), and soils were analyzed for exchangeable cations and nutrients (EC; Ca, Mg, K, P, and sodium (Na)), and total metals (aluminum (Al), arsenic (As), boron (B), Cd, Cu, iron (Fe), manganese (Mn), nickel (Ni), Pb and Zn). Ash was additionally evaluated for carbon (C) and N content and total nutrients and metals (Ca, Mg, K, P, Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, Zn).

Soil and ash pH were measured in a 0.01M $CaCl_2$ slurry at a 1:5 ratio. The slurries were shaken for two hours and then rested for one hour prior to taking a reading using an OAKTON pH 510 series multimeter (Oakton Instruments, Vernon Hills, Il, US). The probe was calibrated every 20 samples to ensure continuity. Percent organic matter was determined by LOI (Kalra and Maynard, 1991) using two grams L, FH or five grams of mineral soil or ash. Oven-dry samples were weighed into porcelain crucibles and ashed in a muffle furnace for 8 hours at 450°C. Ash samples were also analyzed for percent C and N content using a CNS combustion analyzer (Elementar vario EL III elemental analyzer, Elementar Americas Inc, Ronkonkoma, NY, US) and EnviroMAT SS-2 standards (SCP Science, Quebec, CA). In Bracebridge, the L, FH and upper and lower mineral soil N content averaged 0.8%, 0.2%, and 0.1%, respectively (Deighton & Watmough, 2020).

Soil exchangeable cations were analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES) with a Perkin Elmer Optima 7000DV (Waltham, MA, US). One gram of L, FH material or five grams of mineral soil were weighed into 50 mL Falcon Tubes before adding 25 mL of 1M ammonium chloride (NH_4Cl) solution, shaking the solution for two hours and resting for one hour. The solution was then filtered through P8 Fast Flow Filter Paper where an additional 25 mL of 1M NH_4Cl was added. Samples were then diluted and refrigerated prior to analysis by ICP-OES. Total metal concentrations in soils and nutrient and metal concentrations in ash were determined using a nitric acid (HNO_3) digestion followed by ICP-OES analysis. Samples were weighed to ~0.2 grams into digiTUBEs (SCP Science, Quebec, CA) and digested on a hot plate at 100°C for eight hours with 2% HNO_3 before digesting at room temperature for another eight hours. The samples were then filtered with P8 Fast Flow Filter Paper, diluted to 25mL with B-pure water and then refrigerated prior to analysis. Soil standards (EnviroMAT SS-1) and blanks were tested periodically to ensure accuracy and a standard curve was created for each analysis with the ICP-OES with elemental standards from SCP Science (SCP Science, Quebec, CA).

2.3.4.2 Foliage Analysis

Foliar samples were dried at 110°C and ground before being analyzed for CN content and total nutrients and metals (Ca, Mg, K, P, Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, Zn). Carbon and N content were determined using a CN combustion analyzer (Elementar vario MICRO cube, Elementar Americas Inc, Ronkonkoma, NY, US) with NIST 1515-SRM apple leaf standards throughout (SCP Science, Quebec, CA). Nutrient and metal concentrations were determined using a nitric acid digestion as described above for soils with NIST 1515-SRM apple leaf standards (recovery was 90-100%; SCP Science, Quebec, CA).

2.3.4.3 Sap Analysis

Sap collected in the 50 mL Falcon Tubes was thawed and each sample was filtered through a 0.45μm nylon filter. Filtered sap was then analyzed for pH using an OAKTON pH 510 series multimeter (Oakton Instruments, Vernon Hills, Il) and sweetness by measuring sucrose concentration on a Brix scale (°Brix ± 0.2% accuracy; Gregory and Hawley, 1983) using a Reed R9500 Brix refractometer, 0 – 32% (Reed Instruments, Wilmington, NC). After filtration the sap was acidified to 2% HNO_3 and measured for nutrient and metal concentrations (Ca, Mg, K, P, Al, As, B, Cd, Cu, Fe, Mn, Na, Ni, Pb, Zn) by ICP-OES.

2.3.5 *Data Analyses*

2.3.5.1 Climate Data

Climate data and weather conditions during the period of study (2021-2022) were retrieved from the Muskoka, ON station (44°58'29.000" N, 79°18'12.000" W) operated by NAV Canada (Ottawa, ON, CA) and were downloaded from the Environment and Climate Change Canada (2023b) portal.

2.3.5.2 Volume-Weighted Calculations

Average volume-weighted (VW) sap pH was calculated using the following equation (Eq. 1):

$$VW_{pH} = -\log\left(\frac{\sum_{i=1}^{\frac{T}{\delta t}}(H_i)}{\sum_{i=1}^{T} Y_i}\right) \quad (1)$$

where H denotes the H^+ concentration of a sample during a particular sampling period (δt) within each sampling season (T) divided by the total volume (L) of sap measured from each individual tree (Y) over the whole sampling season each year. The negative log was then taken to determine the final VW pH.

2.3.5.3 Elemental Flux Calculations

Sap elemental flux (mg) was calculated by multiplying individual elemental concentrations (mg L^{-1}) in each sap subsample by the total volume (L) collected since the previous collection to determine the total elemental flux (mg) per spile for that sampling period. Individual elemental flux values were then summed per tree adapted after Moatar and Meybeck (2005) (Eq. 2):

$$Flux/spile = \sum_{i=1}^{T/\delta t} (C_i V_i)$$

(2)

for a particular sampling period, δt, within each sampling season, T, to get the total flux per spile per season. Further, C_i denotes the concentration (mg L^{-1}) of the element of interest for the respective sampling period and V_i is the volume accumulated (L) for that sampling period (total volume of sap per spile since previous collection date). Flux values for each tree were then averaged per treatment to get the total average elemental flux per treatment per season.

2.3.5.4 Diagnosis and Recommendation Integrated System (DRIS) Calculations

Sugar maple foliar DRIS indices were calculated to determine whether nutrients were lacking (negative) or in excess (positive). Indices were determined by first calculating foliar P, N, Ca, Mg, and K DRIS ratios similar to Casson et al. (2012), where when *A/B* ≥ *a/b* (Eq. 3):

$$f\left(\frac{A}{B}\right) = \left(\frac{A/B}{a/b} - 1\right)\frac{1{,}000}{\mathrm{CV}}$$

(3)

or when *A/B* ≤ *a/b* (Eq. 4):

$$f\left(\frac{A}{B}\right) = \left(1 - \frac{a/b}{A/B}\right)\frac{1{,}000}{\mathrm{CV}}$$

(4)

where *A/B* is the ratio of the two foliar elements being assessed, *a/b* is the foliar ratio norm and CV is the associated coefficient of variation for that norm based on crown position as determined by Lozano and Huynh (1989). Foliar indices were then calculated (Eq. 5):

$$A \text{ index} = \frac{\left[f\left(\frac{A}{B}\right) + f\left(\frac{A}{C}\right) + f\left(\frac{A}{D}\right) + f\left(\frac{A}{E}\right)\right]}{z}$$

(5)

where *A* is defined as the foliar element that the index is being calculated for, *B, C, D,* and *E* refer to the other elements being measured, and *z* is the number of functions used within the nutrient index. The sum of all nutrient indices equals zero so that indices for each element can be compared relative to one another.

2.3.6 *Statistical Analysis*

Statistical analyses were conducted using the R software environment version 4.2.2 (R Core Team, 2022). Soil, sap, and foliar chemistry in the control and treatment plots were compared using a Wilcoxon rank-sum test with a Bonferroni correction to adjust for multiple comparisons (*rstatix* package). Wilcoxon rank-sum tests were used since the primary objective of this paper was to assess treatments effects and model residuals could not be normalized in a repeated measures design. A linear mixed-effects model (*lme4* package) with interactions was used to test for differences in average yield between treatments, while accounting for tree DBH as a covariate, year as a repeated measures factor, and a random effect to control for the differences among replicate trees within the same treatment. A post hoc test was conducted when a significant difference was found using estimated marginal means and pairwise comparisons (*emmeans* package) with a tukey adjustment to test for significant differences between treatment levels each year. Normality of the model residuals was tested using the Shapiro-Wilk normality test (*rstatix* package) and QQ plots (*ggpubr* package), and homogeneity of variances was tested using Levene's test (*car* package). Significance was determined at $p < 0.05$ unless otherwise stated.

2.4 Results

2.4.1 *Non-Industrial Wood Ash Chemistry*

Non-industrial wood ash averaged 27% Ca and 9% K dry weight and an average pH of 13.0 (Table 2.1). Carbon and N content in NIWA were particularly low, with N concentrations low enough to suggest the addition of a N source to NIWA before application (Table 2.1). Mean concentrations of most metals were well below the unrestricted guidelines (CM1) for land application of NASMs in Ontario, Canada (Government of Ontario, 2002), except for Cd and As that fell just below CM1, and Zn and Cu that fell marginally above CM1 but well below restricted levels (CM2; Table 2.1).

Table 2.1. Average pH_{CaCl2}, organic matter, and nutrient and metal concentrations (dw)** of non-industrial wood ash (means ± SE) collected in Muskoka, Ontario and applied to a sugar maple dominated forest in November 2020 (*n* = 24). Non-agricultural source material limits for unrestricted (CM1) and restricted (CM2) use of wood ash in Ontario are included according to the Nutrient and Management Act, 2002†.

	Non-Industrial Wood Ash Properties (*n* = 24)	NASM Limits†		Elemental Additions from NIWA Applied at 6 Mg ha^{-1} (kg ha^{-1})
		CM1	CM2	
pH	13.0 (0.04)			
OM (%)	3.4 (0.3)			
C (%)	8.6 (0.1)			
N (%)	0.1 (0.0)			
Ca (g kg^{-1})	267 (3.0)			1602
K (g kg^{-1})	94.4 (2.9)			566
Mg (g kg^{-1})	19.4 (0.3)			116
Mn (g kg^{-1})	8.8 (0.3)			52.56
P (g kg^{-1})	7.5 (0.1)			45.05
Al (g kg^{-1})	3.8 (0.3)			22.79
Fe (g kg^{-1})	2.2 (0.2)			13.32
Zn (mg kg^{-1})	503 (18.5)	500	4200	3.02
Cu (mg kg^{-1})	164 (9.4)	100	1700	0.99
Cd (mg kg^{-1})	2.9 (0.2)	3	34	0.02
As (mg kg^{-1})	9.9 (2.2)	13	170	0.06
Ni (mg kg^{-1})	9.6 (0.6)	62	420	0.06
Pb (mg kg^{-1})	48.2 (16.1)	150	1100	0.29
B (mg kg^{-1})	265 (5.3)			1.59

*Sulfur concentrations were below the detection limit (BDL) and therefore removed.
**dw, dry weight by mass
†Government of Ontario, 2002.

2.4.2 *Soil Chemistry*

Prior to ash application, pH, organic matter, and average concentrations of nutrients and metals in soil were similar between the control and treatment plots (Figures 2.1, 2.2, and 2.3). One year following NIWA application soil pH was significantly higher in the LFH horizons in the ash treated plots whereas organic matter content was significantly lower in the litter (Figure 2.1). There were many differences in soil macronutrient chemistry between treated and control plots, but these were mostly restricted to the upper organic soil horizons (Figure 2.2). Calcium concentrations were significantly higher in the LFH and upper mineral horizons in the treatment plots and K and Mg exhibited similar patterns, except that significantly higher concentrations were only observed in the FH and upper mineral horizons (Figure 2.2).

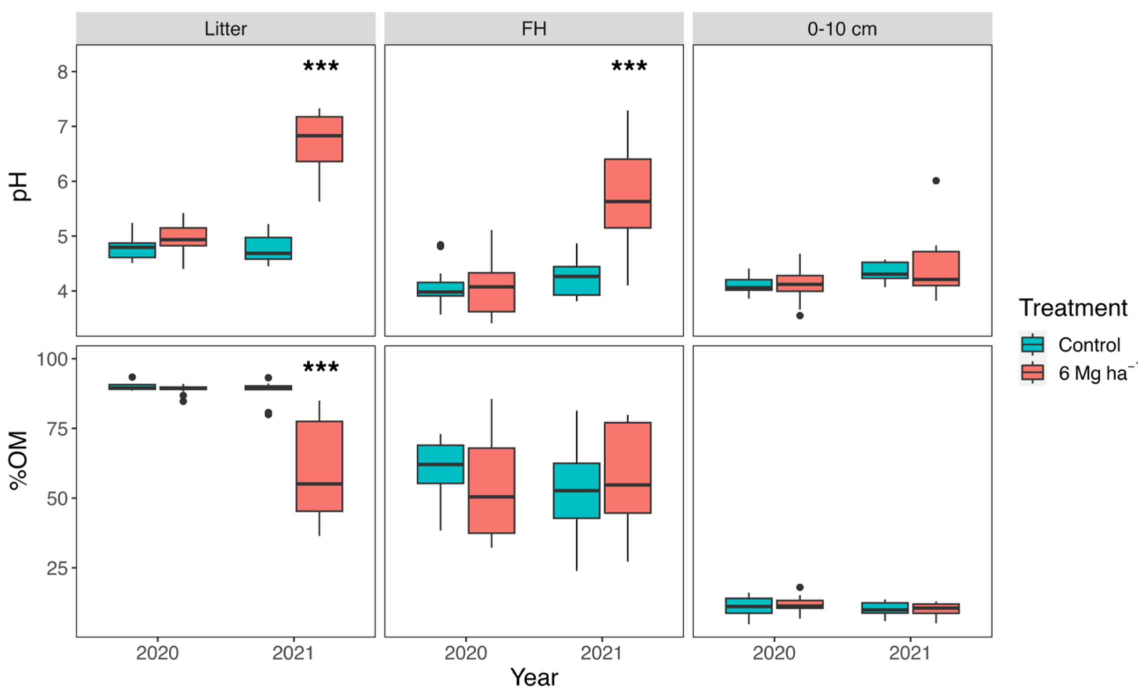

Figure 2.1. Average pH_{CaCl2} and percent organic matter (OM) in the L, FH, and upper mineral soil (0 – 10 cm, Ah) sampled beneath sugar maple trees (n = 12) in the control and NIWA treated (6 Mg ha^{-1}) plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

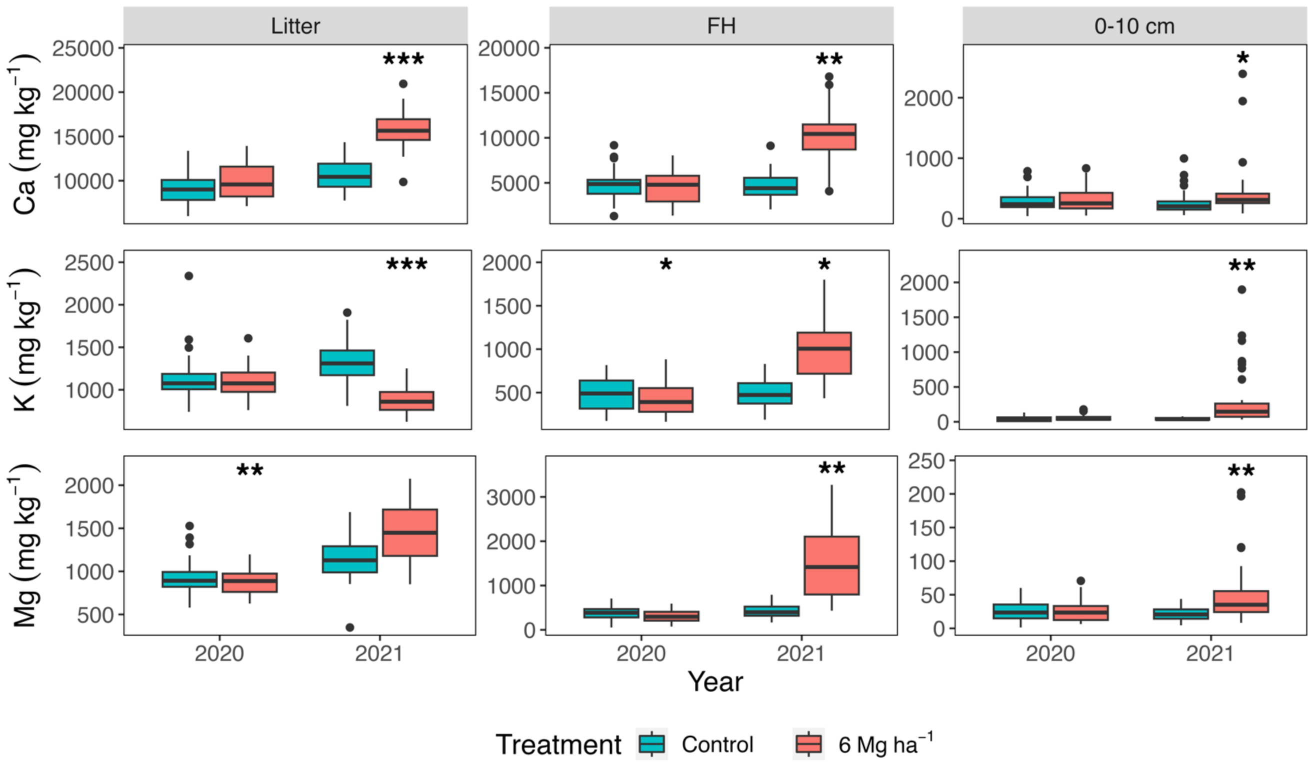

Figure 2.2. Average exchangeable cations in the L, FH, and upper mineral soil (0 – 10 cm, Ah) sampled beneath sugar maple trees (n = 12) prior to (2020) and after (2021) ash application in the control and NIWA treated (6 Mg ha^{-1}) plots. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

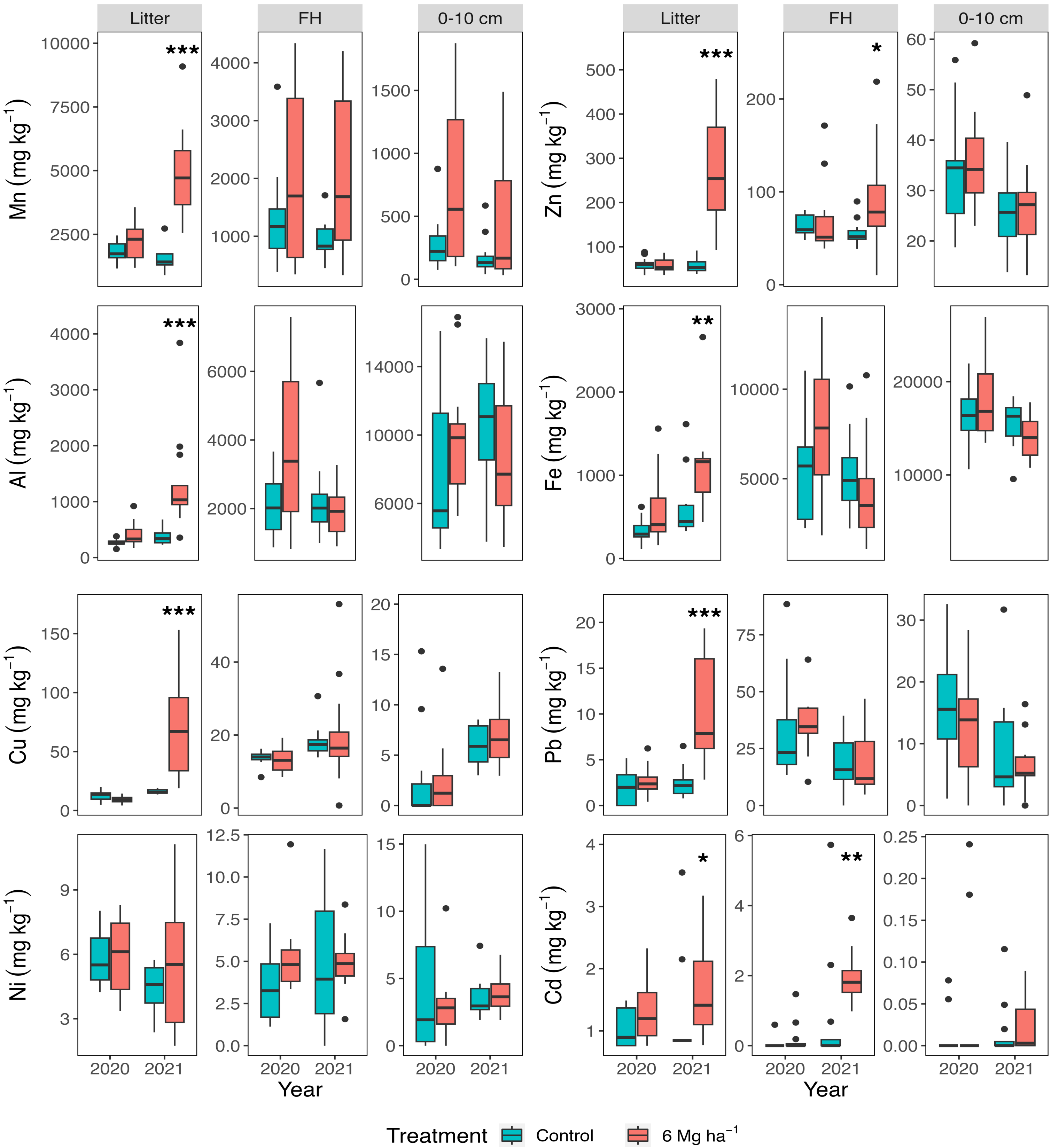

Figure 2.3. Average elemental metal concentrations in the L, FH, and mineral soil (0 – 10 cm, Ah) sampled beneath sugar maple trees (n = 12) prior to (2020) and after (2021) ash application in the control and NIWA treated (6 Mg ha^{-1}) plots. Arsenic and B were below their detectable limits and therefore removed. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

Following NIWA application, concentrations of several metals increased significantly, but these were restricted to the upper organic soil horizons (L and to a lesser extent, FH), with Al, Mn, Cu, Pb, and Zn showing the largest increases in the treated plots compared with controls (Figure 2.3). Concentrations of metals in the upper mineral soil were unaffected by ash application, and all regulated metals remained lower than the maximum allowable concentrations for soils receiving NASM (Figure 2.3; Appendix A-1).

2.4.3 *Climate Data and Sap Yield and Chemistry*

The March 1-April 30 mean air temperatures were 2.7ºC in 2021 and 0.5ºC in 2022 (Figure 2.4). Total precipitation from March 1 – April 30 was similar in both years at 113 mm in 2021 and 128 mm in 2022 (Figure 2.4). In 2021, however, sap yield in the treated plots (54 L) was twice that in the untreated plots (27 L), whereas in 2022, sap yield in both the treated (56 L) and untreated plots (52 L) was almost identical (Figure 2.5). The timing of peak sap flow also differed between seasons with a much earlier peak flow in 2021 compared with 2022, consistent with the earlier warming observed in 2021 (Figure 2.4; Figure 2.5). The linear mixed effects model evaluating the effect of treatment on yield revealed a significant treatment effect ($p < 0.1$) and a significant interaction between treatment and year with no significant effect of tree DBH. Post hoc analysis revealed that there was a significant difference between sap yield in the control plots in 2021 and 2022 ($p < 0.01$; Table 2.2). No significant treatment effect was found on sap pH or sweetness in either sampling year (Table 2.2).

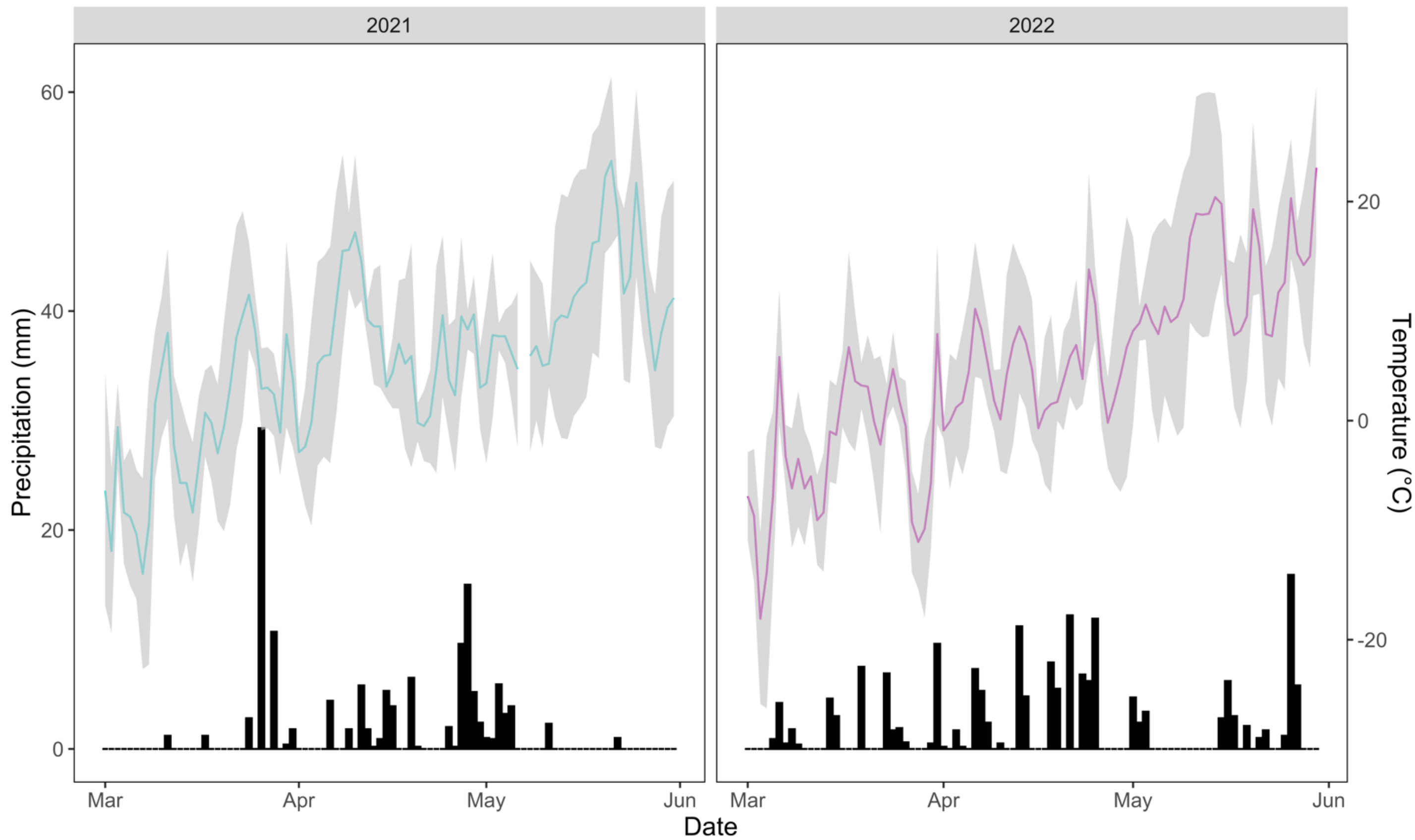

Figure 2.4. Climograph for Muskoka, ON during maple sap sampling seasons in 2021 (March – May) and 2022 (March – April). Bars indicate average daily precipitation (mm), and lines indicate average daily temperature (°C) with minimum and maximum daily temperatures in grey shading. Breaks in the lines indicate missing data (ECCC, 2023b).

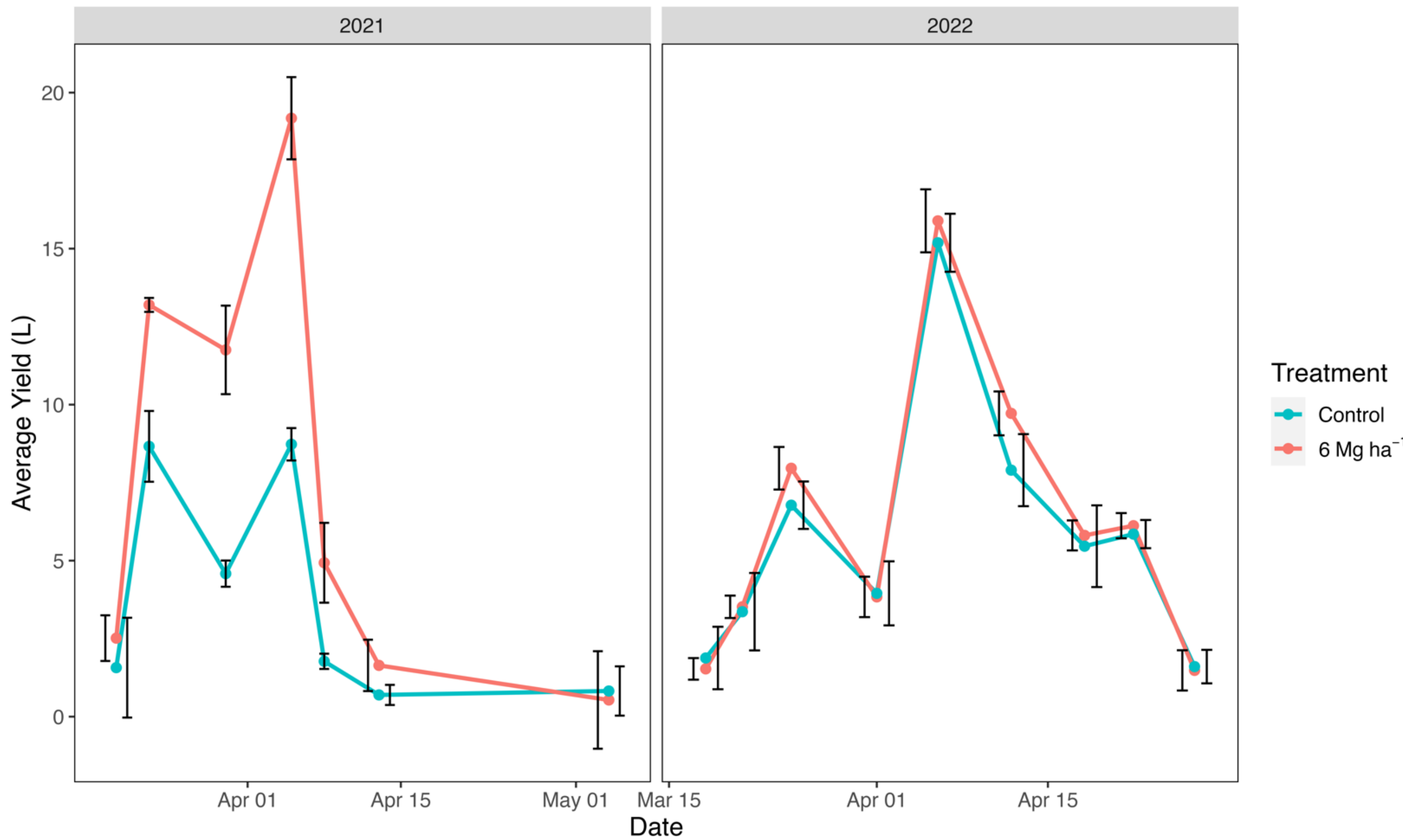

Figure 2.5. Average sap yield (L ± SE) per spile since previous collection (n = 12) in the control and NIWA treated (6 Mg ha^{-1}) plots. Sampling was conducted from March 20-May 4 in 2021 and March 18-April 27 in 2022.

Table 2.2. Average (± SE) volume-weighted pH, °Brix, total yield, and elemental concentrations in sugar maple sap during the 2021 and 2022 sampling seasons in the control and NIWA treated (6 Mg ha^{-1}) plots. Significant differences from control indicated by an asterisk (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$) as determined by a Wilcoxon rank-sum test except for differences in average yield that were determined by a linear mixed effects model to account for DBH and repeated measures in 2021 and 2022 ($p < 0.05$). Also included are the ranges of elemental concentrations in sugar maple sap estimated from sugar maple syrup by accounting for an approximate 50-times concentration during the boiling process.

	2021 Sap		2022 Sap		Range of Concentrations in Sugar Maple Sap†
	Control (*n* = 12)	Treatment (*n* = 12)	Control (*n* = 12)	Treatment (*n* = 12)	
Avg. Total Yield (L)	26.8 (4.6)^{a}	53.8 (5.5)^{ab}	52.0 (5.7)^{b}	55.9 (5.5)^{ab}	
pH	6.50 (0.31)	6.53 (0.35)	7.87 (0.06)	7.95 (0.05)	
°Bx	1.35 (0.07)	1.44 (0.07)	1.37 (0.06)	1.39 (0.05)	
	-----mg L^{-1}-----				-----mg·L^{-1}-----
Ca	64.7 (4.5)	78.8 (3.7)***	52.2 (2.5)	24.9 (1.4)***	5.3 – 80.6
K	60.8 (4.3)	63.0 (4.1)	37.7 (1.4)	63.4 (2.3)***	10.8 – 80.6
Mg	6.0 (0.4)	6.9 (0.3)**	4.2 (0.2)	4.0 (0.2)	0 – 11.5
P	0.4 (0.1)	0.5 (0.1)	0.6 (0.0)	1.6 (0.1)***	0 – 4.7
Na	0.1 (0.0)	0.2 (0.0)	0.2 (0.1)	0.1 (0.0)	0 – 9.84
Mn	5.1 (0.4)	6.0 (0.3)**	4.0 (0.2)	1.2 (0.1)***	0 – 5.0
	-----ug L^{-1}-----				-----ug·L^{-1}-----
Zn	250 (19.3)	321 (25.3)**	282 (11.6)	179 (7.5)***	0 – 2600
Al	28.6 (1.9)	26.1 (1.5)	27.1 (3.4)	29.8 (4.4)	0.2 – 360
Fe	23.2 (5.9)	18.0 (4.3)	18.9 (3.7)	20.1 (3.0)	0 – 1220
Cu	8.7 (1.6)	14.7 (3.0)*	8.8 (1.0)	14.0 (1.2)***	0 – 400
Pb	4.0 (0.5)	2.6 (0.3)*	1.0 (0.1)	0.9 (0.1)	0 – 53.6
Ni	4.6 (0.6)	6.9 (1.0)***	4.1 (0.6)	5.1 (0.3)***	
Cd	1.9 (0.3)	1.8 (0.2)	0.9 (0.1)	0.4 (0.1)***	0 – 980

[*]As and B were below their detection limits and therefore removed.
[†]Review by Mohammed et al., 2022.

Sap nutrient and metal concentrations varied between years, and while some differences were noted in sap chemistry between treated and untreated trees, they were not always consistent between years and in most cases differences between the treated and untreated trees were small (< 30%; Table 2.2). Significantly higher concentrations of Ca, Mg, Mn, Zn, Cu, and Ni were measured in the sap from treated trees in 2021, while in 2022 concentrations of K, P, and Ni were significantly higher in the treated trees (Table 2.2). Further, in 2022 sap concentrations of Ca, Mn, Zn, and Cd were significantly lower in ash treated trees, whereas in 2021 only Pb concentrations were significantly lower in the treated trees compared with controls (Table 2.2). Largely due to much greater sap flow in the treated trees in 2021, significant increases were observed in the mean seasonal elemental flux of most nutrients and metals, but a significantly higher flux was only observed in K and P in 2022 (Table 2.3). In 2021, seasonal fluxes of nutrients and metals in the ash treated trees increased by between 2% and 226% (Table 2.3). Sugar maple sap chemistry is rarely reported, but both sap nutrient and metal concentrations fell within the reported ranges of sugar maple syrup elemental concentrations as gathered by Mohammed et al. (2022) when adjusted for an approximate 50-times concentration during distillation (Table 2.2).

Table 2.3. Annual elemental flux (± SE) in sugar maple sap during the 2021 and 2022 sampling seasons. Significant differences from control indicated by an asterisk (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$) as determined by Wilcoxon rank-sum test.

Element	2021 Sap		2022 Sap	
	Control ($n = 12$)	Treatment ($n = 12$)	Control ($n = 12$)	Treatment ($n = 12$)
	----------mg tree^{-1}----------			
Ca	1525 (292)	3546 (252)***	2717 (407)	1307 (137)*
K	1293 (233)	2684 (258)***	1808 (221)	3291 (352)**
Mg	136 (25.7)	302 (23.1)***	216 (30.4)	212 (24.1)
P	10.1 (2.9)	18.9 (3.4)	29.7 (5.2)	80.4 (14.8)**
Na	3.4 (0.8)	11.1 (4.3)*	9.6 (2.4)	10.5 (4.6)
Mn	127 (29.3)	279 (32.4)**	207 (34.7)	60.9 (7.3)**
	----------ug tree^{-1}----------			
Zn	5528 (1015)	13,096 (1333)***	13,298 (2027)	8437 (836)
Al	622 (111)	1092 (103)**	1265 (205)	1371 (154)
Fe	420 (251)	431 (64.7)*	949 (437)	1133 (314)
Cu	170 (57.1)	555 (95.1)**	364 (96.9)	579 (101)
Pb	92.2 (25.5)	101 (15.1)	48.5 (12.3)	41.7 (9.4)
Ni	91.8 (24.8)	246 (31.8)**	189 (54.3)	238 (26.2)
Cd	48.5 (15.7)	79.4 (9.0)*	47.2 (8.0)	19.6 (4.5)*

*As and B were below their detection limits and therefore removed.

2.4.4 *Foliar Chemistry*

Sugar maple foliar chemistry sampled in the summer of 2021 showed few significant differences between the ash treated and control plots, with the notable exception of K that was almost twice as high in ash treated plots (14,032 mg kg^{-1} in the treated foliage compared with 7,551 mg kg^{-1} in the controls; Table 2.4). Mean concentrations of Ca and several other metals (Mn, Fe, Zn, Al, Ni, and Cd) also tended to be higher in ash treated trees, but for the most part these differences were small and statistically insignificant and remained either within or below critical foliar concentrations reported for sugar maples (Table 2.4; Kolb & McCormick, 1993). Percent C and N were also similar across treatments (Table 2.4). The DRIS values indicated that the nutrient balance in control trees was close to optimum (-20% - +20%) whereas ash treated trees significantly increased the nutrient balance of K while worsening the balance for P (Table 2.5).

Table 2.4. Average (± SE) foliar nutrient and metal concentrations in sugar maple trees (*n* = 4) collected eight months after application of 6 Mg ha^{-1} NIWA to experimental plots in Bracebridge, ON. Significant differences between treatment and control indicated by an asterisk (*, $p < 0.05$) as determined by a Wilcoxon rank-sum test.

Element	Control	Treatment	Critical Foliar Concentrations†
C (%)	46.1 (0.5)	45.3 (0.3)	
N (%)	2.3 (0.1)	2.2 (0.1)	1.6-2.32
Ca (mg kg^{-1})	8970 (1262)	10,345 (562)	5000-21900
K (mg kg^{-1})	7551 (686)	14,032 (1263)*	5500-10400
Mg (mg kg^{-1})	1580 (195)	1556 (36.3)	1100-4000
P (mg kg^{-1})	1360 (142)	1263 (49.2)	800-1800
Mn (mg kg^{-1})	885 (178)	1083 (150)	632-1630
Zn (mg kg^{-1})	26.8 (3.1)	33.3 (2.6)	29-71
Al (mg kg^{-1})	14.3 (1.1)	19.1 (2.5)	32-60
Fe (mg kg^{-1})	49.9 (4.8)	58.3 (8.1)	59-130
Cu (mg kg^{-1})	2.9 (0.5)	2.6 (0.4)	3-9
Ni (mg kg^{-1})	0.3 (0.1)	1.1 (0.7)	
Cd (ug kg^{-1})	0.4 (0.4)	1.1 (0.9)	

*As, B, and Pb were below their detection limits and therefore removed.

†Kolb & McCormick, 1993.

Table 2.5. Average sugar maple foliar concentrations ($n = 4$) based on DRIS eight months after application of 6 Mg ha^{-1} NIWA to experimental plots in Bracebridge, ON with critical foliar concentrations and DRIS norms for reference. Significant differences between treatment and control indicated by an asterisk (*, $p < 0.05$) as determined by a Wilcoxon rank-sum test.

Foliage	Control	Treatment	Critical Foliar Concentrations (%)[†]
Concentrations (%)			
P	0.14	0.13	0.08-0.18
N	2.31	2.99*	1.6-2.23
Ca	0.90	1.03	0.5-2.19
Mg	0.16	0.16	0.11-0.4
K	0.76	1.40*	0.55-1.04
Ratios			DRIS Norms[‡]
P:N	0.06	0.05	0.10
P:Ca	0.15	0.15	0.17
P:Mg	0.87	0.82	1.19
P:K	0.18	0.15*	0.18
N:Ca	2.79	2.95	1.39
N:Mg	15.34	16.25	12.36
N:K	3.15	2.81	1.85
Ca:Mg	5.74	5.69	8.12
Ca:K	1.19	1.01*	1.28
Mg:K	0.21	0.18*	0.15
DRIS Indices			
P	-10.44	-24.09*	
N	18.40	20.73	
Ca	-9.85	-8.89	
K	-8.38	11.47*	
Mg	10.27	0.78	

[†]Kolb & McCormick, 1993.
[‡]Lozano & Huynh, 2008.

2.5 Discussion

2.5.1 *Non-Industrial Wood Ash Chemistry*

Non-industrial wood ash collected from volunteer residents of Muskoka contained very high concentrations of several macronutrients (Ca, Mg, K, and P) while metal concentrations remained low. The pH of the NIWA averaged 13.0, a value that lies at the higher range of those reported in the literature (Demeyer et al., 2001; Pitman, 2006;

Augusto et al., 2008; Deighton & Watmough, 2020). Calcium was the most abundant nutrient, accounting for 27% of the ash by dry weight, followed by K at 9% and Mg at 2%. These values are similar to values reported by Azan et al. (2019) at 30% Ca, 8% K and 2% Mg and Deighton and Watmough (2020) who evaluated ash produced from sugar maple, white pine, and yellow birch separately. Metal concentrations (Cd, Zn, Cu, and Se) in yellow birch ash tended to be elevated above CM1 guidelines, and therefore this species should be avoided or used in small quantities as a NIWA source material (Deighton & Watmough, 2020). Additionally, almost all metal concentrations fell below CM1 guidelines except for Cu and Zn. The CM1 limits for Cu and Zn are 100 mg kg^{-1} and 500 mg kg^{-1}, respectively, and are just below the concentrations reported here at 164 mg kg^{-1} Cu and 503 mg kg^{-1} Zn. Similarly, NIWA collected from Muskoka residents a few years before our ash was collected had concentrations of 101 mg kg^{-1} Cu and 501 mg kg^{-1} Zn, and although they fall slightly above CM1, both concentrations are substantially lower than the CM2 limits and therefore are unlikely to create toxic soil conditions (Azan et al., 2019). In agreement with Syeda et al. (submitted), these results suggest that amalgamating wood ash samples from various species and sources results in metal concentrations that fall safely within provincial regulatory guidelines.

2.5.2 *Soil Responses to Ash Amendments*

Prior to NIWA application no significant differences in soil pH or organic matter content were observed between the treatment and control plots. One year following treatment, soil pH was significantly higher in the upper organic layers, but no significant differences in pH were observed in the mineral layer. Wood ash has a strong neutralizing capacity because of its hydroxide, carbonate and bicarbonate components and its ability to buffer protons in the soil (Demeyer et al., 2001). Consequently, the pH increased in the litter layer by 1.4 units and the FH layer by 1.1 units compared with baseline levels pre-ash application. Such large increases in pH tend to occur when pre-treatment conditions are more acidic as they were here (pH < 5.0, Reid & Watmough, 2014) and are consistent with increases observed by the application of similar doses in other short-term studies (5-6 Mg ha^{-1}, 1 – 5 years; Ozolinčius et al., 2007b; Reid & Watmough, 2014; Deighton & Watmough, 2020). The lack of a significant pH response in the mineral layer 10 months after ash application was also expected. A longer delay before increases in pH are noticeable in the mineral layers is well-documented (Lundström et al., 2003; Ozolinčius et al., 2007b; Moore et al., 2012) because retention of soluble cations in the organic horizons results in slow vertical leaching and thus the neutralizing components take longer to migrate to deeper layers of the soil. For example, Saarsalmi et al. (2001) found no effect on mineral soil pH measured seven years after wood ash fertilization but did see an increase 16 years after application. However, pH increases may be observed as early as five years after application in the upper mineral soil layers and 10 years for lower soil profiles (Saarsalmi et al., 2004).

Organic matter content was significantly lower in the litter of the treatment plots one year following ash application, but no effect on organic matter content was found in the FH or mineral horizons. Decreases in organic matter are not always observed in ash application studies (Fritze et al., 1994; Saarsalmi et al., 2001; Deighton & Watmough, 2020) and those found here are most likely the result of some residual ash, which is low in organic matter content, remaining on the uppermost layer of soil during sampling the following year.

Soil Ca, Mg, and K concentrations in the organic and upper mineral soil horizons also tended to increase while metal concentrations remained relatively low. Short-term and sustained increases in Ca and Mg in the organic horizons following ash application are commonly reported in the literature (Saarsalmi et al., 2001, 2004; Augusto et al., 2008; Reid & Watmough, 2014; Arseneau et al., 2021). While the increases observed here were the most significant in the litter (Ca) and FH (Ca and Mg) layers, Ca and Mg concentrations were also higher in the upper mineral horizon one year after application, similar to other studies (Saarsalmi et al., 2001, 2004; Ozolinčius et al., 2007b; Augusto et al., 2008; Deighton & Watmough, 2020; Arseneau et al., 2021). On the other hand, K was significantly lower in the litter layer whilst increasing in the FH and upper mineral soil. Increases in K concentrations in the organic horizons are most commonly observed in the short-term (1 – 5 years post-application; Ozolinčius et al., 2007b; Augusto et al., 2008) but have been observed to persist in deeper soil profiles over longer time periods (6 – 16 years; Bramryd & Fransman, 1995; Saarsalmi et al., 2004; Augusto et al., 2008). Low K concentrations in the litter layer are likely due to the high solubility of K and its displacement off soil exchange sites by other cations such as Ca and Mg (Ohno, 1992; Reid & Watmough, 2014). Other research, however, has found no significant differences between control and treated plots in the forest floor or upper mineral soil K three years after 20 Mg ha^{-1} wood ash application in sugar maple stands in Quebec (Arseneau et al., 2021).

Increases in all detectable metal concentrations were noted in the litter layer of the treatment plots one year following wood ash application, but only Cd and Zn increased in the FH layer, and none were observed in the upper mineral horizon. Increases in the litter horizon are to be expected due to the slow weathering rate of the ash observed in this study and are consistent with other findings (Hansen et al., 2018; Deighton et al., 2021). Whilst metal mobility will likely be further restricted due to the decrease in acidity of the upper soil horizons (Augusto et al., 2008; Violante et al., 2010), Deighton et al. (2021) found no evidence of enhanced metal leaching with wood ash treatment even following simulated drought conditions that led to a reduction in soil pH suggesting metal mobility may not be of concern even in more acidic soils.

2.5.3 *Sap Yield and Chemistry*

The optimal January-May mean temperature for peak sap volume is 1ºC (Rapp et al., 2019) and average monthly temperatures were similar in both sampling seasons. The first winter following ash application (2021) the January-May mean temperature was 0ºC whereas in 2022 it was -2ºC. Additionally, earlier sap collection corresponded with a higher March mean temperature (-1ºC in 2021 and -3ºC in 2022). Sap production is highly influenced by climate variability (Marvin & Erickson, 1956; Kim & Leech, 1985) as sugar maple sap is exuded when temperatures fluctuate between freezing at night and thawing during the day alternating between negative and positive pressures in the xylem tissue (Tyree, 1983; Cirelli et al., 2008). Therefore, sap production and total yield depend largely on temperatures during the production season and the preceding months (Rapp et al., 2019), but if the observed differences in yield were due to differences in climate then more variability between years would have been expected. In this case, sap yield in the treatment plots was double that of the controls the first year following NIWA application, and it was similar to the yields of the treated and control trees in 2022. While it is important to mention that this effect was not significant, it is possible that nutrient supplementation the first-year enhanced maple sap production in a year where production in Ontario was the lowest it had been since at least 2018 (AAFC, 2023). This would also correspond with the overall increases seen in nutrient concentrations in 2021. In an experiment conducted by Mengel and Haeder (1977), the rate of phloem sap exudation in castor bean (*Ricinus communis*) receiving a high K treatment (1.0 mM) was approximately double those that had received a lower dose (0.4 mM) and this did not cause a dilution effect on other compounds such as sucrose. These results were attributed to increased carbon dioxide (CO_2) assimilation leading to better allocation of ATP necessary for phloem loading ultimately resulting in a higher osmotic pressure in the plant (Mengel & Haeder, 1977). However, if elevated foliar K concentrations and CO_2 assimilation increase osmotic pressure, then it cannot explain the differences in sap yield the first year as no leaves were present between NIWA application and the first sampling season. Therefore, it seems possible that increased nutrient concentrations may alter the osmotic potential of water in the tree and thus influence sap flow (Shabala & Shabala, 2011).

No effect was observed on sap pH or sap sweetness in either year following ash application. Sap pH can vary seasonally, acidifying as the season progresses (Jones & Alli, 1987; Fromard et al., 1995; Lagacé et al., 2015), but this process appears to be more related to natural fluctuating concentrations of Ca, Mg, and malate (Schill et al., 1996; Schell, 1997) and tree developmental stage than to other factors such as temperature (Pramsohler et al., 2022). Sap pH in this experiment became slightly more acidic towards the end of both seasons but these decreases were not significant and average values were consistent with the reported range of 6.5 to 8.5 depending on the time of season (Jones & Alli, 1987; Clément et al., 2010; Lagacé et al., 2015). Sap sugar content exhibits a negative linear relationship with the May-October mean temperature of the previous growing season (Rapp et al., 2019), which in this study averaged 14.5ºC in 2020 and 15.9ºC in 2021. The previous seasons' May-October mean precipitation was less predictive of sap sugar content than temperature (Rapp et al., 2019), but was similar in both years with 3.6 mm in 2021 and 3.1 mm in 2022. The lack of changes observed here in the sugar concentration of maple sap are similar to a study conducted in Ontario where lime and fertilizer treatments were found to have no effect on sap sweetness (Noland et al., 2006) and is supported by the lack of correlation found between sweetness and base cation concentrations in Vermont (Wilmot et al., 1995a). In contrast, in a long-term study, liming was found to improve sap sweetness up to 20% eighteen years after application in Québec (Moore et al., 2020) and N additions have been shown to increase sap sweetness two years after application in New Hampshire (Wild & Yanai, 2015). Overall, research on the effect of wood ash on sap sweetness is limited. Sugar content in sap varies seasonally and is positively correlated with sweetness in previous years (Wilmot et al., 1995a) because it is driven by non-structural carbohydrate production in previous growing seasons (Muhr et al., 2016). Thus, sweetness is likely influenced by the availability of stored carbon in trees, and if wood ash application can increase carbon sequestration it may result in increased sap sweetness over the long term. It is also important to note that when sap yield was nearly double the first year following application, no dilution effect was observed on sap sweetness between the control and treated plots, similar to the results obtained by Mengel and Haeder (1977). Ultimately, this study provides evidence that application of wood ash does not negatively impact sweetness.

Differences in sap nutrient concentrations between treated and untreated trees were noted but these were not consistent between years and most differences were small. In both control and treated trees Ca, Mg, and K were present in the greatest concentrations in both years, as found previously (Yuan et al., 2013; Lagacé et al., 2015; Mohammed et al., 2022). When comparing sap between treatments each year, significantly higher concentrations were observed in Ca (+22%) and Mg (+15%) the first year following application, but the second-year concentrations were surprisingly lower in Ca (-52%) and Mg (-5%) and significantly higher in K (+68%) and P (+167%). These patterns are consistent with the seasonal fluxes of each cation in both years. Similarly, eighteen years after liming, Moore et al. (2020) found significant increases in Ca (5 Mg ha^{-1} treatment) and Mg (2 and 5 Mg ha^{-1} treatments) but not K in the sap. An antagonistic relationship between K and Mg uptake in plants is also well recognized in the literature (Moore & Ouimet, 2006; Xie et al., 2021) and thus it is possible that high concentrations of Mg in the first year prevented significant increases in K despite its increase in flux; the subsequent leveling of Mg concentrations then allowed for greater increases in K concentrations the following year (Xie et al., 2021), though the influence of this mechanism on sap is not well understood. Previous research has also noted that cation concentrations can vary considerably over the season (Lagacé et al., 2015).

Some differences in sap metal concentrations between treated and control trees were observed, but these were also inconsistent between years. Seasonal fluctuations of metal concentrations in maple syrup are common (Mohammed et al., 2022), and are often attributed to factors such as climate variability and microbial activity (Lagacé et al., 2015). When average maple syrup metal concentrations reported in a review by Mohammed et al.

(2022) were adjusted to sap concentrations it was also evident that the sap metals observed here, even after NIWA treatment, are at the lower end of the reported range and therefore do not pose a concern. These results highlight the natural variability of sap composition and suggest that NIWA application may increase essential nutrient concentrations without raising the toxicity from metals.

2.5.4 *Foliar Chemistry*

In general, sugar maple foliar base cation and metal concentrations were similar between control and treated plots one year after application. No significant differences were observed between Ca, Mg, or P in the control and treated plots, and all foliar concentrations for these nutrients were within the healthy range for sugar maple trees (Kolb & McCormick, 1993; Bal et al., 2015). Increased foliar concentrations of nutrients such as Ca, Mg, and P may take longer to see due to retention in existing organic matter (Augusto et al., 2008; Reid & Watmough, 2014). However, since critical foliar concentrations and DRIS norms did not indicate mineral nutrient deficiencies for sugar maple in the control plots, it is also possible that this accounts for the lack of effect seen following the treatment, particularly in Ca and Mg. Only foliar K concentrations were significantly higher in the treated plots which is consistent with the more immediate availability of K from wood ash application (Reid & Watmough, 2014). Foliar K is essential for tree health as deficiencies have been linked with reduced photosynthetic capacity (Xie et al., 2021) which may ultimately impact sap sweetness through a reduction in stored carbon (Wong et al., 2003). Healthy sugar maple foliar K concentrations range from 5.5-10.4 g kg^{-1} (Kolb & McCormick, 1993) and while average concentrations in the control plots remained within this range at 7.6 g kg^{-1}, treated trees averaged higher at 14.0 g kg^{-1}. Reported DRIS indices in the control plots also show foliar nutritional balances within the optimal range for sugar maples (-20% - +20%; Lozano & Huynh, 1989). While indices generally improved in the treatment plots they only did so significantly for K while worsening significantly for P. Increasing P deficiencies found here and in other studies after liming (Moore & Ouimet, 2006) and ash amendments (Arseneau et al., 2021) are likely due initially to the reduced bioavailability of P in more acidic soils and are potentially exacerbated over the long-term through P precipitation in the form of insoluble hydroxyapatite as soil pH and Ca concentrations rise (Penn & Camberato, 2019). In comparison, application of a similar dose of dolomitic lime (5 t ha^{-1}) showed improved DRIS indices in Ca and Mg slowly over time, but decreases in K and P in sugar maple ten years after application (Moore & Ouimet, 2006). These results lend further evidence to the suggestion that the effect of NIWA on Ca and Mg concentrations also materializes over the longer term. A study conducted by Arseneau et al. (2021) found no significant differences in N, P, K, or Ca concentrations in sugar maple foliage three years after application of 20 Mg ha^{-1} wood ash, but they did find a significant increase in Mg in the treated plots. Therefore, surges in K concentrations appear to occur in the short-term (1-5 years; Augusto et al., 2008) and given that Arseneau et al. (2021) also found no significant differences in forest floor or upper mineral soil K concentrations in either the control or treated plots as observed here, it is likely that higher K concentrations would not pose a risk. Future research may also consider periodic monitoring for P deficiencies.

No significant changes were observed in foliar metal concentrations, and all values remained within healthy ranges for sugar maple trees except for Al, Fe, and Cu that were either just at or below the healthy range, but these were consistent between treatments. It was noted that both Zn and Cd tended to be higher in the foliage of ash treated trees, similar to the study conducted by Deighton and Watmough (2020), where significant increases in sugar maple seedlings treated with NIWA were reported. However, these increases in Cd and Zn were small and remained within the range of values found in healthy sugar maple.

2.6 Conclusions

This study evaluated the effect of applying 6 Mg ha^{-1} NIWA to a base-poor, sugar maple-dominated forest stand in central Ontario, Canada. Wood ash is not routinely used as a soil amendment in Canada due to concern over metal toxicity, however, all metals from our NIWA samples did not exceed provincial regulatory thresholds that would have restricted application to soils. Non-industrial wood ash showed a positive fertilization effect on soils, increasing organic horizon pH and base cations in the organic and upper mineral horizons with metal concentration increases being restricted to the litter layer and remaining below regulatory guidelines. Additionally, NIWA application may provide a short-term (<1 year) surge in sap yield whilst not compromising sap pH or sweetness and increasing nutrient concentrations without the risk of metals exceeding common concentration ranges found in sugar maple syrup. The mechanisms behind the positive response in sap volume are unclear but are the focus of continued study. Lastly, changes in foliar chemistry were limited, aside from significant increases in K concentrations. These results suggest that the application of NIWA does not have a negative effect on sugar maple sap and may supplement essential nutrients for its production and sweetness.

3. Contrasting Sugar Maple (*Acer saccharum*) and American Beech (*Fagus grandifolia*) Responses to Non-Industrial Wood Ash Fertilizer in Muskoka, Ontario

3.1 Abstract

Non-industrial wood ash (NIWA) can be used as a forest soil amendment to supplement acidified soils that exhibit chronic nutrient depletion. Use of NIWA may help restore forest soil pH and nutrient reserves but its use in Ontario, Canada is highly restricted because of metal contaminants that could be toxic in high concentrations. Sugar maple (*Acer saccharum* Marshall) have declined in recent decades and American beech (*Fagus grandifolia* Ehrhart) have subsequently expanded since they are less sensitive to more acidic, nutrient-depleted soils, but it is unknown how they will respond to NIWA treatment. Non-industrial wood ash that was high in macronutrient (calcium (Ca), magnesium (Mg), potassium (K), and phosphorous (P)) and low in metal concentrations was applied at a rate of 6 Mg ha^{-1} to experimental plots in Bracebridge, Ontario. One-year post-application, soil chemistry beneath maple, beech and mixed canopies responded similarly with large significant increases in pH, nutrient and metal concentrations in the organic horizons, and significant increases in K and Mg in the mineral soil horizons. Both maple and beech fine root biomass was similar between the control and treated plots, while fine root K and Mg concentrations of both species were significantly higher in the treated plots. Foliar responses of both species were muted, but K was significantly higher in treated sugar maple trees. Understory vegetation abundance and composition was also consistent between the control and treated plots, but some differences were noted between years. Both maple and beech seedling abundance were also unaffected by NIWA application. Overall, NIWA can

provide a significant boost in pH and macronutrient concentrations in forest soils one year following application and may be an ideal alternative for nutrient supplementation.

3.2 Introduction

Chronic acidification of forest ecosystems caused by decades of acid deposition has compromised North American and European soils (Likens et al., 1996; Driscoll et al., 2001; de Vries et al., 2014). Acidic deposition has led to soil base cation depletion, increased metal mobility, and an overall decline in tree health and biodiversity (Driscoll et al., 2001; de Vries et al., 2014). Even though atmospheric deposition of sulfur (S), and more recently nitrate (NO_3-N), has been significantly reduced since the 1990's (ECCC, 2022), the supply of acid-neutralizing base cations through natural chemical weathering is often not adequate to replace what has been leached in acid-sensitive areas such as the Canadian Shield (Driscoll et al., 2001; Ouimet et al., 2006; Watmough et al., 2016; Johnson et al., 2018). Soil acidification is further exacerbated on harvested sites where base cations and other essential nutrients are removed from the system altogether (Akselsson et al., 2007; Thiffault et al., 2011). Thus, the application of wood ash is a possible remediation strategy to help speed up the recovery process and mitigate leaching of base cations.

Wood ash is generated as a by-product of wood combustion and can effectively buffer acidic soils because of its hydroxide, bicarbonate, and carbonate compounds (Demeyer et al., 2001) as well as high concentrations of base cations such as calcium (Ca), potassium (K), magnesium (Mg), and nutrients such as phosphorous (P; Pitman, 2006; Deighton & Watmough, 2020). Wood ash is not specifically regulated in Ontario as it is classified within a broad scope of other materials as a non-aqueous, non-agricultural source material (NASM; Government of Ontario, 2002; Hannam et al., 2016). Application of NASMs is primarily restricted by the content of regulated metals (CM) that fall under two categories: CM1 and CM2. The CM1 guidelines for application are largely unrestricted if all metal concentrations fall below regulated levels, but restrictions increase if concentrations fall between CM1 and CM2, and application is not allowed above CM2 (Government of Ontario, 2002; Hannam et al., 2016). In Ontario, approximately 18,000 Mg of non-industrial wood ash (NIWA) are generated and landfilled in Ontario each year (Azan et al., 2019) and therefore updated management and application guidelines could help re-purpose this waste as a remediation technique for acidified forest soils. Research suggests that the amalgamation of NIWA helps maintain high levels of macronutrients while limiting metal concentrations mostly below CM1 and never above CM2 guidelines (Azan et al., 2019; Deighton & Watmough, 2020).

Sugar maple (*Acer saccharum* Marshall) and American beech (*Fagus grandifolia* Ehrhart) are dominant species in the Great Lakes St. Lawrence Forest region. Sugar maple is a keystone species in eastern North America and is highly valued for its many products (AAFC, 2023). However, maple trees are particularly sensitive to acidic deposition and low Ca concentrations in soil (Sullivan et al., 2013). Sugar maple decline has been reported since the 1950's with reductions observed in vigor, canopy condition, growth, and recruitment (Westing, 1966; Adams and Hutchinson, 1992; Duchesne et al., 2002; Schaberg et al., 2006; St.Clair et al., 2008; Sullivan et al., 2013). Conversely, American beech trees are more tolerant of low soil pH, low base cation concentrations, and higher concentrations of phytotoxic aluminum (Al) that is mobilized in acidic soils (Cronan & Schofield, 1990; Park & Yanai, 2009; Stephanson & Coe, 2017) and reportedly have expanded in some areas at the expense of sugar maple (Duchesne et al., 2005; Duchesne & Ouimet, 2009; Nolet & Kneeshaw, 2018). On the other hand, American beech trees are increasingly threatened by beech bark disease (BBD), an insect-fungus complex that eventually causes tree mortality (Stephanson & Coe, 2017). Although BBD has been reported in Ontario as early as the 1960's, large-scale infection has only largely occurred since 2000-2010 (Cale et al., 2017). Trees that are heavily infected with BBD grow 40% slower, develop cankers that jeopardize the integrity of the bark and leave the tree vulnerable to further infection, and may experience heavy crown mortality (Cale et al., 2017). Infection ultimately leads to overall tree mortality with estimates as high as 61-81% over a seven year period within the "killing front" stage of infection (Cale et al., 2017).

In contrast to sugar maple, which typically responds positively to soil amendments including lime, wood ash, and wollastonite ($CaSiO_3$), the response of American beech trees to amendments is often more muted (Moore et al., 2008; Long et al., 2011). This may be because American beech trees are typically less sensitive to acidic soils and Ca deficiencies compared with maple (Duchesne et al., 2005; Duchesne & Ouimet, 2009; Halman et al., 2015). For example, lime and $CaSiO_3$ studies have indicated that treatments have no measurable effect on American beech basal area increment (BAI), crown vigor, or mortality (Long et al., 1997, 2011; Duchesne et al., 2013; Long et al., 2022). In contrast, base rich soil amendments have resulted in increased BAI in sugar maple as early as one year after treatment, as well as increased soil, foliar and fine root macronutrient concentrations, increased regeneration, and reduced crown dieback and overall mortality (Wilmot et al., 1996; Juice et al., 2006; Huggett et al., 2007; Moore et al., 2008, 2012; Long et al., 2011, 2022; Arseneau et al., 2021). American beech regeneration also appears to be stimulated by stressors such as base-poor soils and increased soil Al concentrations (Duchesne et al., 2005; Duchesne & Ouimet, 2009; Duchesne et al., 2013 Sullivan et al., 2013; Halman et al., 2015) and regeneration has been shown to decline following treatment with lime (Moore et al., 2008). The response of American beech regeneration to amendment with wood ash, however, is less studied. There is also little research on the differences between species in fine root biomass and base cation and metal concentrations. Increased nutrient concentrations in fine roots may stimulate root production (Adams & Hutchinson, 1992; Juice et al., 2006) but have also been associated with a decrease in biomass (Persson & Ahlström, 1990; Clemensson-Lindell & Persson, 1995; Helmisaari et al., 2009) as well as total length and number of fine root tips (Ozolinčius et al., 2007b). Given the differences in the response of each species to base poor and acidic soil conditions (Halman et al., 2015) fine root responses may be species specific (Dijkstra et al., 2001) and therefore more research is necessary to understand the effect of NIWA addition.

To address these knowledge gaps, this work aimed to quantify the short-term (one year) response of both sugar maple and American beech trees to the application of 6 Mg ha^{-1} NIWA. It was hypothesized that the NIWA would increase organic soil pH, as well as increase macronutrient concentrations in the soil organic layers, roots, and foliage of both species. It was predicted that there would be a corresponding increase in metal concentrations in the organic soil layers beneath both species and in mixed soil samples, but these would not translate to the deeper layers of soil, roots, or foliage due to their low concentrations and retention in the organic horizons. It was hypothesized that fine root biomass would decrease within ash-amended plots as a result of increased nutrient availability, and that sugar maple seedling abundance would increase in the understory, but American beech seedling abundance would be unaffected.

3.3 Methodology

3.3.1 *Study Area*

The study area is located within the Great Lakes-St. Lawrence ecozone in an uneven-aged mixed-wood forest east of the town of Bracebridge, Ontario, Canada (45°03'45.27" N, 79°08'43.62" W). The site is approximately 282 m above sea level, the average annual temperature is 5.2°C and the average annual precipitation is 1105 mm measured over a 30-year period (1981 – 2010; Government of Canada, 2023). Total annual precipitation was slightly below the 30-year average but was similar between years (884 mm in 2021 and 806 mm in 2022; ECCC, 2023b). Underlain by gneiss rock the shallow soils are acidic (approximately 4.1 pH) and typically poorly developed podzols and brunisols with coarse sandy loam soils (AAFC, 1998). The study forest is located on ~100 ha owned by Camp Big Canoe since 1968 and has been preserved with no logging and environmental policies limiting waste and preserving air, soil, water, and flora and fauna quality (Casey, 2021). The study forest is dominated by sugar maple trees (79% of total plot basal area) but also contains primarily American beech, ironwood (*Ostrya virginiana* (Miller) K. Koch), yellow birch (*Betula alleghaniensis* Britton), white ash (*Fraxinus americana* Linnaeus), and black cherry (*Prunus serotina* Ehrhart). Lake water has a low pH (~5.8 to 6.2 pH units) and low Ca concentrations (2.43 mg L^{-1}; Reid & Watmough, 2016) similar to other surface waters in the region.

3.3.2 *Experimental Design and Sampling*

3.3.2.1 Plot Setup and Experimental Design

Eight 40 x 40 m plots with a 10 m buffer were established within a 10-ha area at the study forest using a randomized plot design in September 2020. Plots were randomly located but were selected to contain at least five mature sugar maple and American beech trees greater than 10 cm diameter at breast height (DBH) and were at least 60 m away from any watercourse to avoid runoff after application.

3.3.2.2 Ash Application and Field Sampling

Non-industrial (residential) wood ash was donated by volunteer residents of Muskoka and collected by the Friends of the Muskoka Watershed (FMW, 2023). At collection the ash was sieved (<2 mm) to remove charcoal and large debris and then processed with a magnet to remove other waste (e.g. nails and screws) before being stored in large, polyethylene containers prior to application. The NIWA was weighed into 10 kg buckets and then transported to four replicate plots to be distributed evenly by hand at a rate of 6 Mg ha^{-1} in November 2020. Six subsamples were randomly collected from the ash at each plot to be analyzed individually (n = 24).

At the end of September 2020 baseline soil sampling was conducted prior to application at each of the eight plots. In each plot, soils were sampled beneath the canopies of three mature sugar maple trees, three mature American beech trees, and four mixed canopy locations. Tree canopies sampled beneath were selected to be greater than 10 cm DBH and the mixed canopy locations were randomly selected within the plot. Four samples were collected at each canopy location within a plot (n = 160 per treatment). Grab samples were collected from the litter (L) and fibrous-humic (FH) horizons, and a steel Dutch auger was used to sample the upper (0 – 10 cm mostly containing the Ah horizon) and lower (11 – 20 cm mostly containing the Bm horizon) soil horizons (AAFC, 1998).

Post-treatment soil sampling was conducted in the same manner as baseline soil sampling in July 2021. Additionally, two sets of root samples were collected for analysis of elemental concentrations as well as measurement of root biomass. For elemental analysis the largest root was found at the base of each tree and followed out approximately 0.5 m where the litter layer was removed and a 10 x 10 cm section of the fine roots was cut on either side of the large root. Fine roots were sampled to a depth of 5 cm and both samples were combined per tree for nutrients and metals. For root mass estimates, two root cores were taken from the FH layer beneath each tree with a cylindrical soil corer (depth = 5 cm, surface area = 118 cm^2), kept separate for analysis and then averaged. All roots were washed prior to drying for analysis. Extendable pole pruners were used to collect foliage samples from each sugar maple and American beech tree where soil samples were collected. When possible, foliar samples were taken from branches receiving direct sunlight and were composited per species per plot ($n = 4$ per treatment). Vegetation surveys were also conducted in June 2021 and 2022 to compare sugar maple and American beech seedling abundance as well as overall species abundance and composition between the control and treated plots. Twenty-four surveys recording species abundance were taken using 1 x 1 m quadrats within each plot (total 24 m^2 per plot). Understory vegetation was included as vascular plants with a DBH < 1 cm and less than 2 m in height. Mean precipitation from April-October in 2021 was 3.22 mm and in 2022 was 2.56 mm (ECCC, 2023b).

3.3.3 *Laboratory Sample Analyses*

3.3.3.1 Soil and Ash Analyses

Soil and ash samples were oven-dried at 110°C for 24 hours. Litter and FH samples were then ground using a Wiley Mill and mineral samples were sieved (< 2 mm). All samples were analyzed for pH and loss-on-ignition (LOI), and soils were analyzed for exchangeable cations (Ca, Mg, K, sodium (Na), P), and total metals (Al, arsenic (As), boron (B), cadmium (Cd), copper (Cu), iron (Fe), manganese (Mn), nickel (Ni), lead (Pb), and zinc (Zn). Ash was also measured for carbon (C) and nitrogen (N) content and total nutrients and metals (Ca, Mg, K, P, Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, and Zn).

Soil and ash pH were measured using an OAKTON pH 510 series multimeter (Oakton Instruments, Vernon Hills, Il, US). Samples were shaken for two hours in a 0.01M $CaCl_2$ slurry at a 1:5 ratio and then rested for one hour. The probe was recalibrated every 20 samples. Loss-on-ignition was measured to determine percent organic matter (OM) of each sample (Kalra and Maynard, 1991). Two grams of oven dried L, FH or five grams of mineral soil or ash were weighed into porcelain crucibles and ashed in a muffle furnace at 450°C for eight hours. Percent C and N content in the ash were measured using a CNS combustion analyzer (Elementar vario EL III elemental analyzer, Elementar Americas Inc, Ronkonkoma, NY, US) and EnviroMAT SS-2 standards (SCP Science, Quebec, CA).

Soil exchangeable cations were measured by weighing one gram of L, FH or five grams of mineral soil into 50 mL Falcon Tubes and adding 25 mL 1 M ammonium chloride (NH_4Cl). The solution was then shaken for two hours and rested for one hour before filtering through P8 Fast Flow Filter Paper where an additional 25 mL of 1 M NH_4Cl was added. Samples were diluted prior to analysis with a Perkin Elmer Optima 7000DV inductively coupled plasma optical emission spectrometer (ICP-OES; Waltham, MA, US). Acid extractable metal concentrations in soils and elemental concentrations in the ash were measured using a nitric acid (HNO_3) digestion. Approximately 0.2 grams of each sample were weighed into digiTUBEs (SCP Science, Quebec, CA) and combined with 2.5 mL HNO_3 (67 – 70% w/w; VWR Chemicals, PA, US). Samples were then digested on a hot plate for eight hours at 100°C before digesting at room temperature for another eight hours. Samples were then filtered through P8 Fast Flow Filter Paper, diluted to 25 mL with B-pure water, and then refrigerated prior to analysis. Soil (EnviroMAT SS-1) and ash (EnviroMAT SS-2) standards (SCP Science, Quebec, CA) and blanks were included periodically to test accuracy and a standard curve was created for each analysis with the ICP-OES with elemental standards from SCP Science (SCP Science, Quebec, CA)

3.3.3.2 Root Mass and Chemical Analyses

Root samples were dried at 110°C for 24 hours. Roots collected for total elemental concentrations (Ca, Mg, K, P, Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed using a HNO_3 digestion in the same manner as described above. For fine root mass measurements all roots < 1 mm in diameter were separated for each sample, weighed on an analytical balance, and then averaged per tree.

3.3.3.4 Foliar Analyses

Foliar samples were dried at 110°C for 24 hours and then ground using a coffee grinder. Samples were analyzed for CN content using a CN combustion analyzer (Elementar vario MICRO cube, Elementar Americas Inc, Ronkonkoma, NY, US) with NIST 1515-SRM apple leaf standards throughout (SCP Science, Quebec, CA). Nutrient and metal concentrations (Ca, Mg, K, P, Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, and Zn) were determined using a HNO_3 acid digest as described above with NIST 1515-SRM apple leaf standards (recovery was 90-100%; SCP Science, Quebec, CA).

3.3.4 *Statistical Analyses*

Statistical analysis was conducted using R software environment version 4.2.2 (R Core Team, 2022). Soil, root, and foliar chemistry were compared between treatments within years using a Wilcoxon rank-sum test with a Bonferroni correction to adjust for multiple comparisons (*rstatix* package). Wilcoxon rank-sum tests were used since the primary objective of this chapter was to determine differences between treatments, not years, and soil, root, and foliar chemistry model residuals could not be normalized in a repeated measures design. Significance was determined at $p < 0.05$ unless otherwise stated.

3.3.4.1 Understory Vegetation Statistics

Shannon Diversity (H), Simpson's Diversity (D), and species richness (S_r) were calculated for each plot using species abundance data. Shannon's Diversity index was calculated using (Eq. 3.1):

$$H = -\sum_{i=1}^{s} p_i \ln(p_i)$$

(3.1)

where p_i is the proportion of one species and s is the total number of species. Simpson's diversity index was calculated using (Eq. 3.2):

$$D = 1 - (\sum n(n-1)/N(N-1))$$

(3.2)

where n is the number of individuals of a single species and N is the number of individuals in the total population. Lastly, richness was defined as the number of different species per 24 m^2 based on the total area surveyed per plot. A generalized linear model (*stats* package) was used to compare species abundance, H, D, and S_r between treatments across years. Tukey's HSD with a Bonferroni correction (*multcomp* package) was then used for pairwise comparisons on variables where significant effects were found.

3.4 Results

3.4.1 *Non-Industrial Wood Ash Chemistry*

Non-industrial wood ash averaged a pH of 13.0 and was characterized by high concentrations of macronutrients (26.7% Ca, 9.4% K, and 1.9% Mg). All regulated metal concentrations were substantially lower than CM2 guidelines and all concentrations remained below CM1 except for Zn and Cu that slightly exceeded CM1 limits for unrestricted NASM application (Table 3.1; Government of Ontario, 2002).

Table 3.1. Average pH_{CaCl2}, LOI, and nutrient and metal concentrations (dw)** of non-industrial wood ash (means ± SE) donated by the residents of Muskoka, Ontario and applied to a mixed-wood forest in November 2020 (*n* = 24). Non-agricultural source material limits for unrestricted (CM1) and restricted (CM2) use of wood ash in Ontario are included according to the Nutrient and Management Act, 2002†.

	Non-Industrial Wood Ash Properties (*n* = 24)	NASM Limits†		Elemental Additions from NIWA Applied at 6 Mg ha^{-1} (kg ha^{-1})
		CM1	CM2	
pH	13.0 (0.04)			
OM (%)	3.4 (0.3)			
C (%)	8.6 (0.1)			
N (%)	0.1 (0.0)			
Ca (g kg^{-1})	267 (3.0)			1602
K (g kg^{-1})	94.4 (2.9)			566
Mg (g kg^{-1})	19.4 (0.3)			116
Mn (g kg^{-1})	8.8 (0.3)			52.56
P (g kg^{-1})	7.5 (0.1)			45.05
Al (g kg^{-1})	3.8 (0.3)			22.79
Fe (g kg^{-1})	2.2 (0.2)			13.32
Zn (mg kg^{-1})	503 (18.5)	500	4200	3.02
Cu (mg kg^{-1})	164 (9.4)	100	1700	0.99
Cd (mg kg^{-1})	2.9 (0.2)	3	34	0.02
As (mg kg^{-1})	9.9 (2.2)	13	170	0.06
Ni (mg kg^{-1})	9.6 (0.6)	62	420	0.06
Pb (mg kg^{-1})	48.2 (16.1)	150	1100	0.29
B (mg kg^{-1})	265 (5.3)			1.59

*Sulfur concentrations were below their detectable limit (BDL) and therefore removed.
**dw, dry weight by mass
†Government of Ontario, 2002

3.4.2 *Soil Chemistry*

Prior to ash application, soil pH, percent organic matter, and mean exchangeable base cation concentrations were similar between the control and treatment plots for sugar maple, American beech, and mixed canopy sample locations (Figures 3.1-3.6). For all canopy locations the average soil pH ranged from 4.5-4.8 in the L, 3.7-4.0 in the FH, 3.7-4.1 in the upper horizon and 4.0-4.3 in the lower horizon prior to application. Additionally, before application percent organic matter averaged 89%, 59%, 12%, and 10% in the L, FH, upper, and lower horizons, respectively. Exchangeable base cation concentrations (Ca, K, Mg) were also similar amongst baseline soil samples, although a few differences were noted. For example, Ca concentrations were slightly higher in the treatment plots (9247 mg kg^{-1}) than in the controls (7602 mg kg^{-1}) in the litter beneath American beech trees, and Mg was slightly lower under sugar maple in the litter (880 mg kg^{-1}) of treatment plots compared with controls (1057 mg kg^{-1}) prior to treatment. Potassium was also slightly lower in the FH layer of the treated plots (346 mg kg^{-1} compared to 477 mg kg^{-1} in the controls) beneath sugar maple trees.

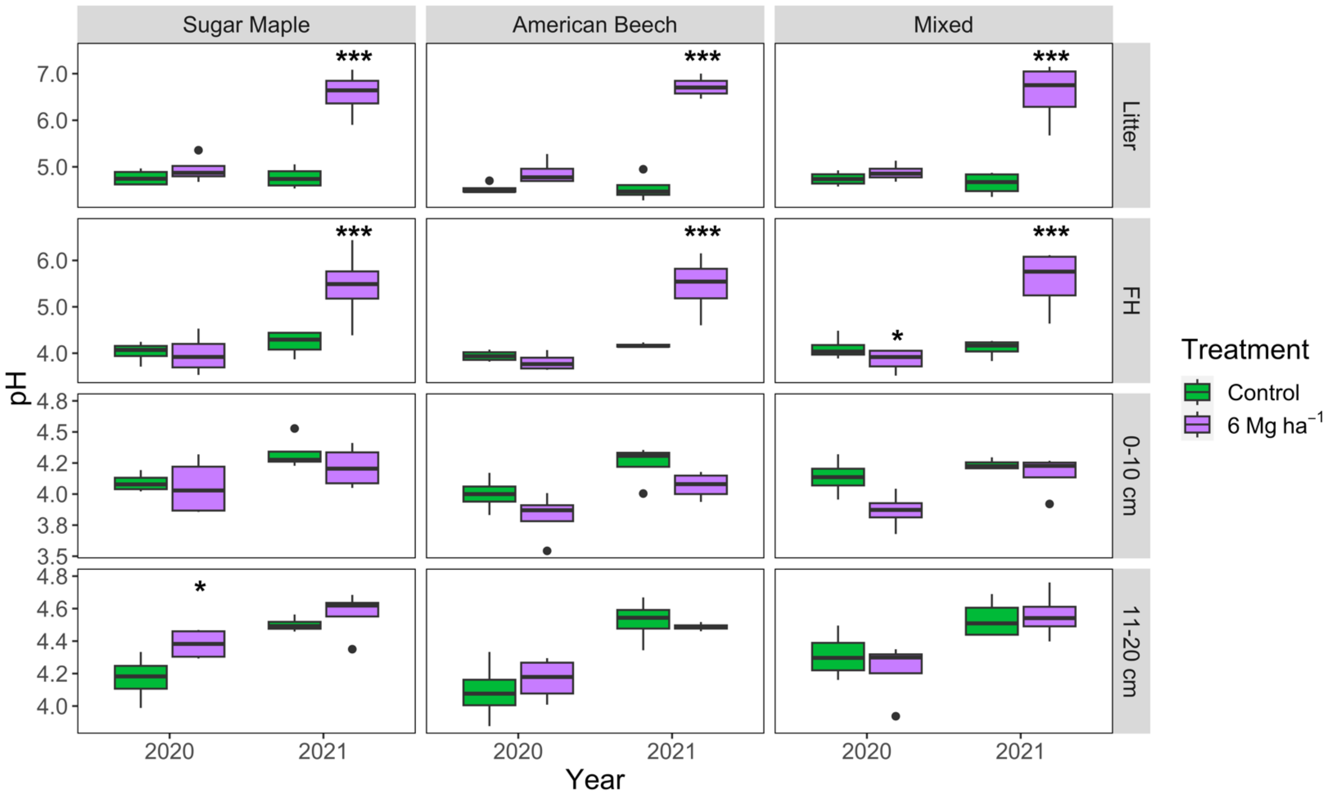

Figure 3.1. Control and treated L, FH, and mineral soil (0 – 10 cm, 11 – 20 cm) pH of sugar maple (*n* = 12), American beech (*n* = 12), and mixed (*n* = 16) samples during baseline (2020) and post-treatment (2021) sampling. Significant differences between control and treatment indicated by an asterisk as determined by Wilcoxon rank-sum tests (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

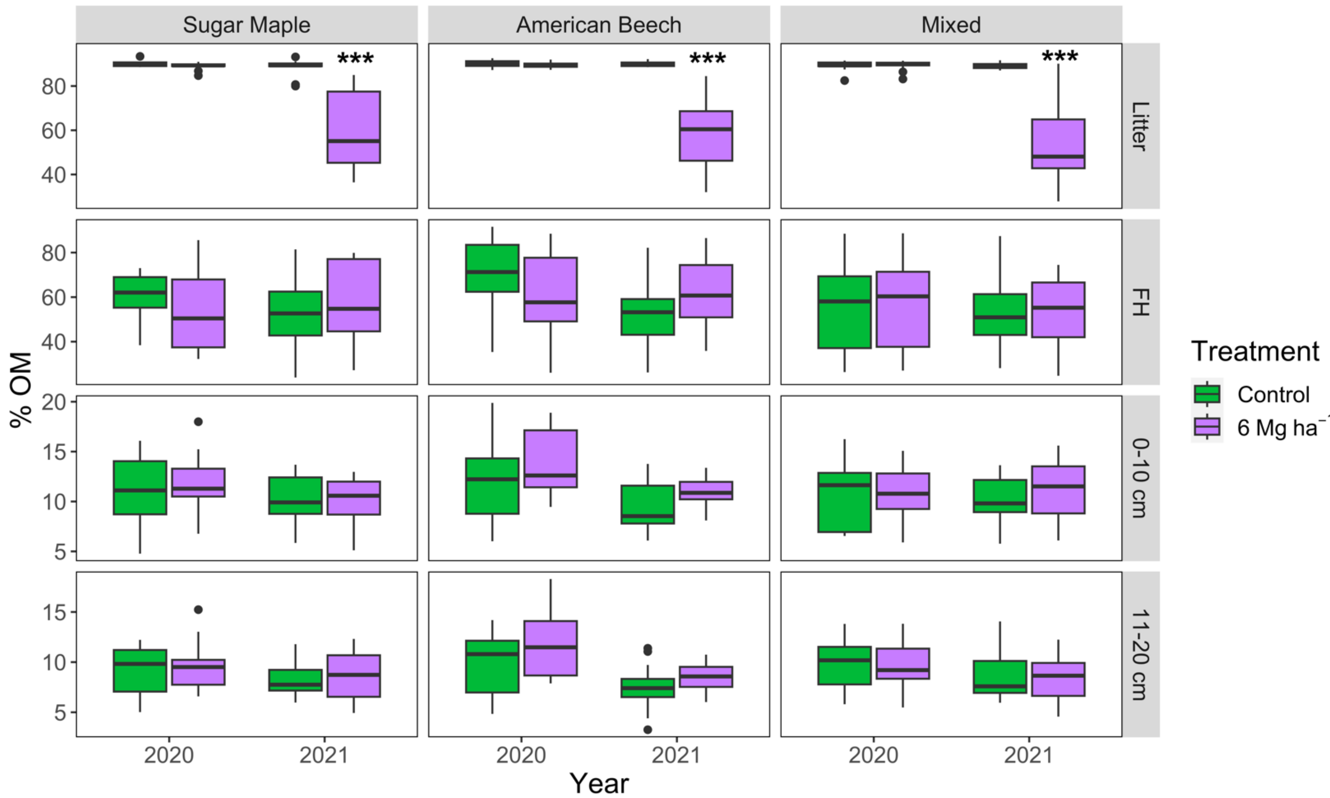

Figure 3.2. Average percent organic matter (OM) in the control and treated L, FH, and mineral soil (0 – 10 cm, 11 – 20 cm) beneath sugar maple (*n* = 12), American beech (*n* = 12), and mixed (*n* = 16) samples during baseline (2020) and post-treatment (2021) sampling. Significant differences between control and treatment indicated by an asterisk as determined by Wilcoxon rank-sum tests (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

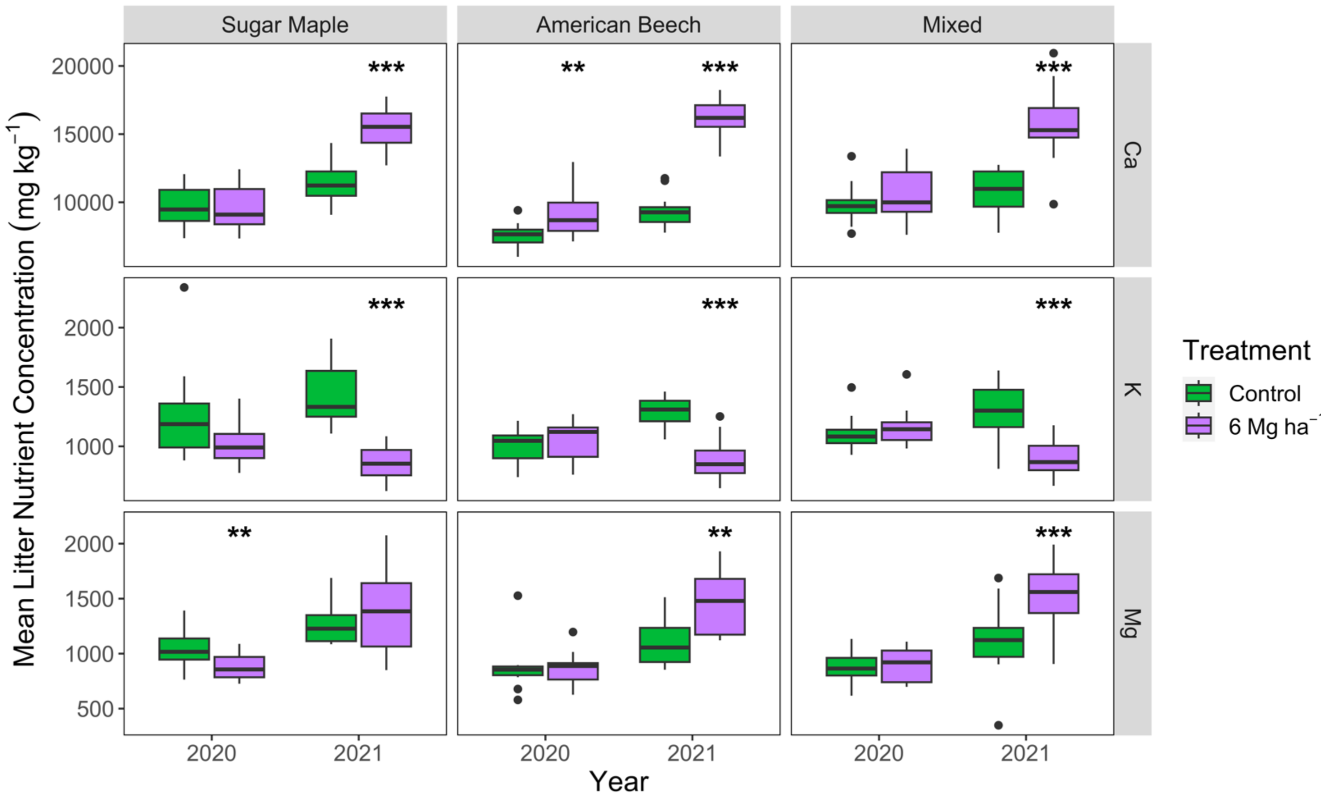

Figure 3.3. Exchangeable base cations in the L layer sampled beneath mature sugar maple (n = 12) and American beech (n = 12) trees as well as mixed soil samples (n = 16) in the control and ash treated plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

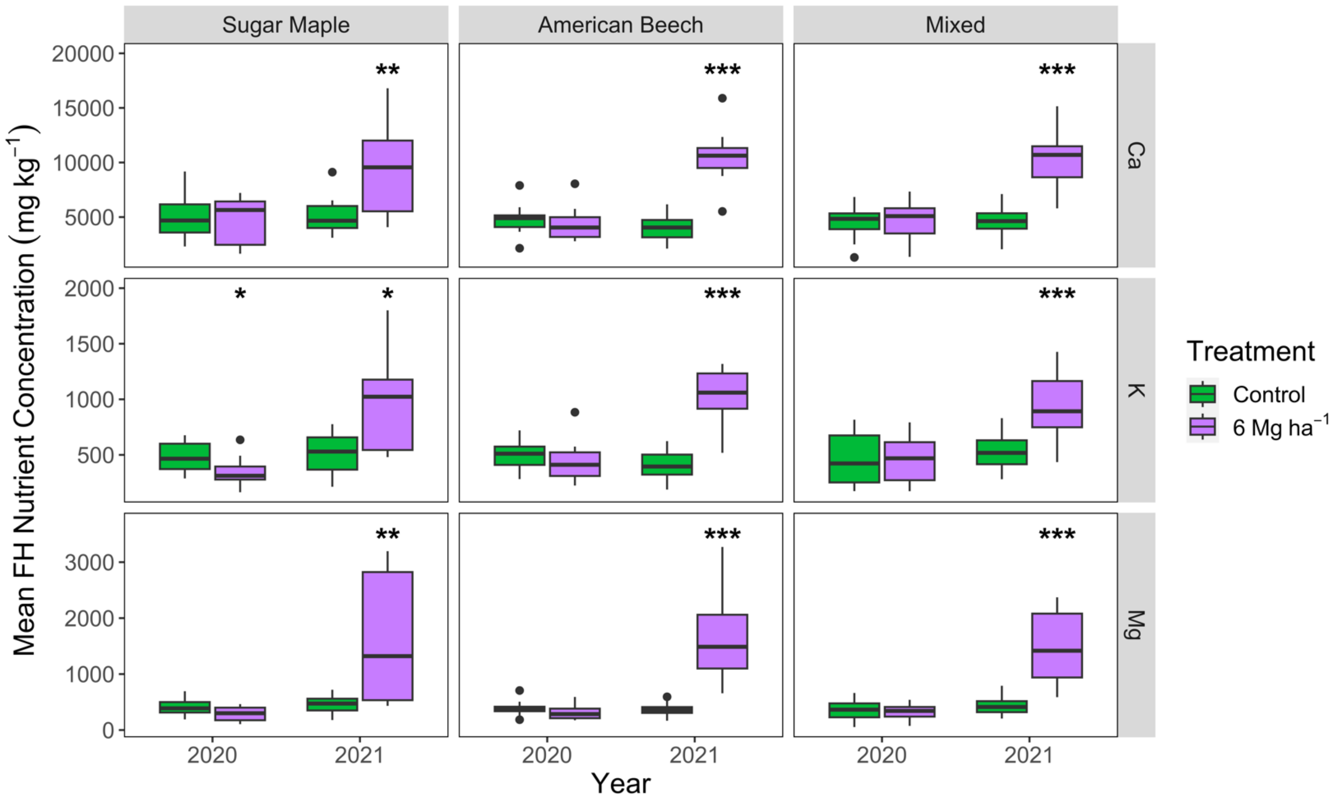

Figure 3.4. Exchangeable base cations in the FH layer sampled beneath mature sugar maple (n = 12) and American beech (n = 12) trees as well as mixed soil samples (n = 16) in the control and ash treated plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

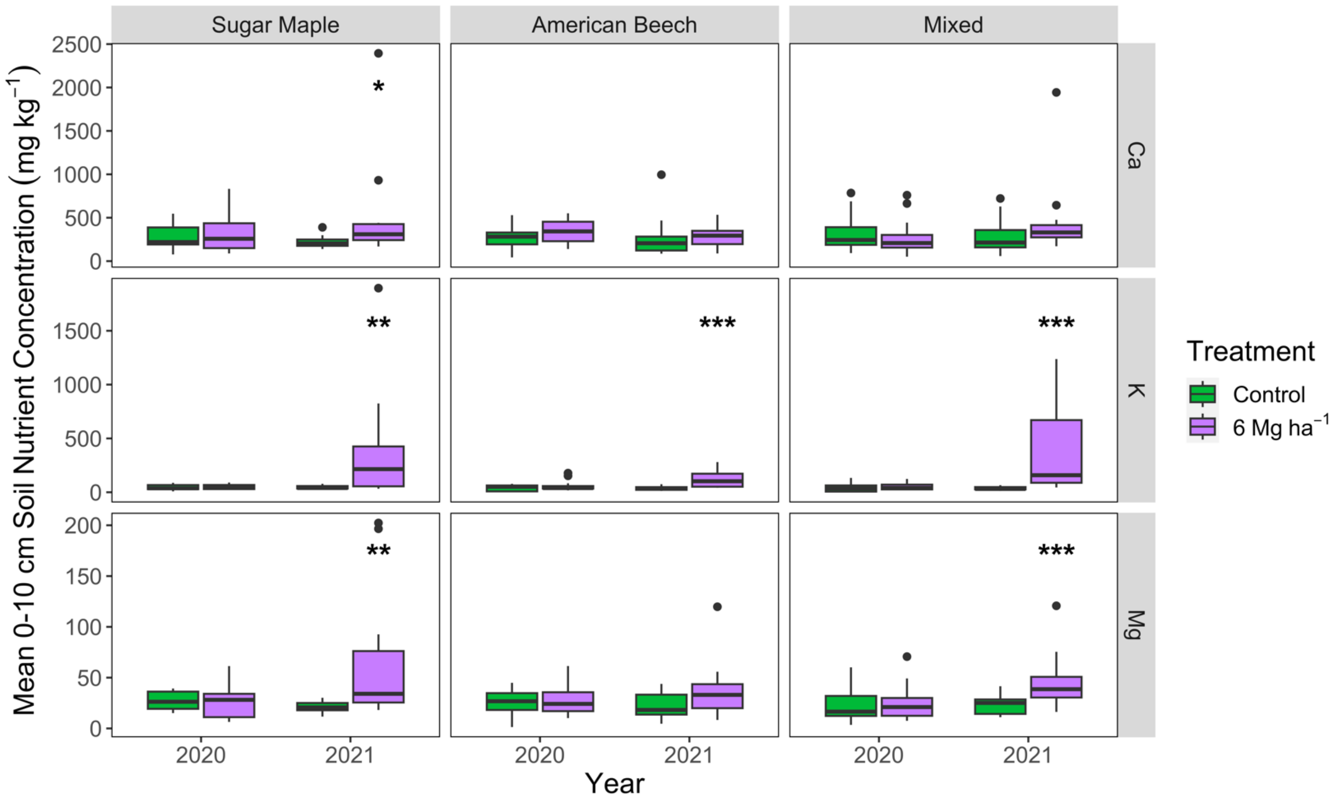

Figure 3.5. Exchangeable base cations (mean ± SE) in the upper soil horizon (0 – 10 cm, Ah) sampled beneath mature sugar maple (n = 12) and American beech (n = 12) trees as well as mixed soil samples (n = 16) in the control and ash treated plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

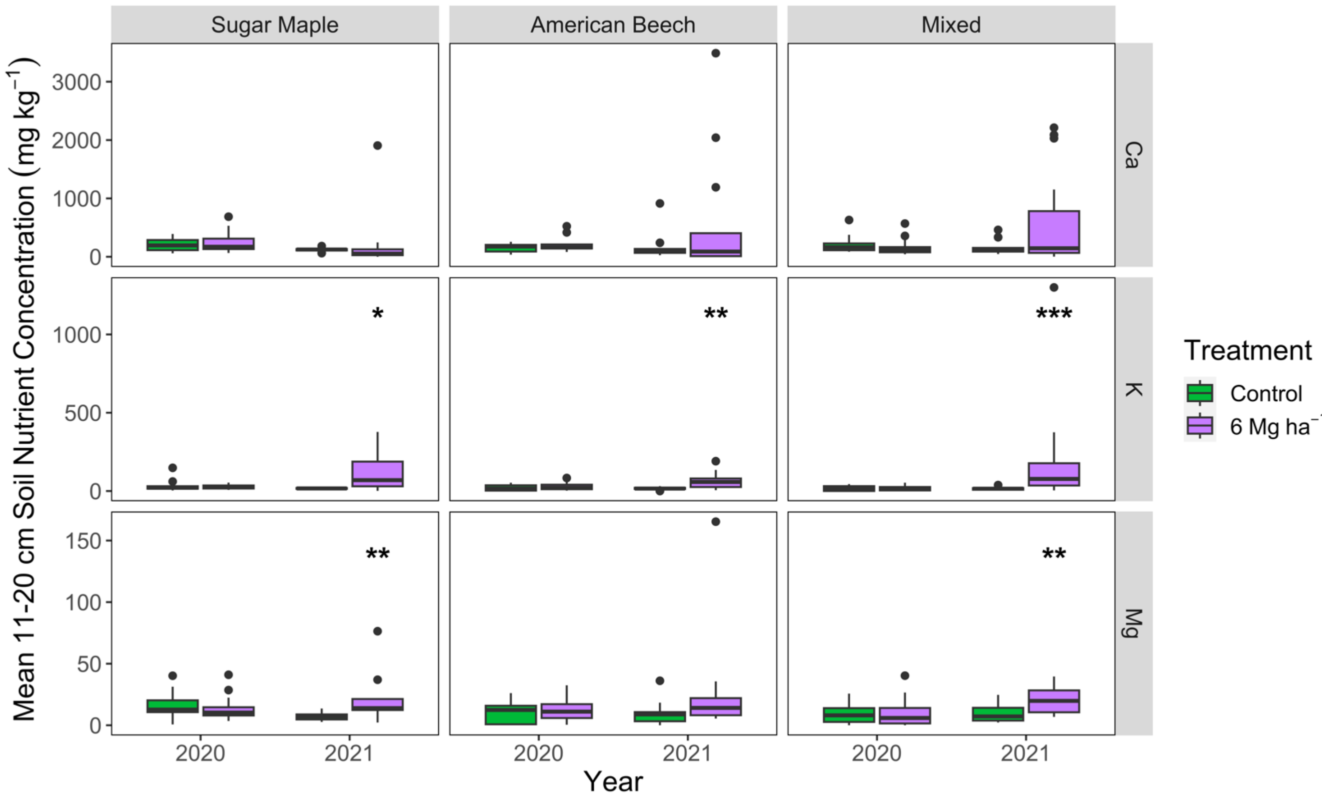

Figure 3.6. Exchangeable base cations (mean ± SE) in the lower soil horizon (11 – 20 cm, Bm) sampled beneath mature sugar maple (*n* = 12) and American beech (*n* = 12) trees as well as mixed soil samples (*n* = 16) in the control and ash treated plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

One year after ash application pronounced changes in the chemistry of the L and FH horizons were observed in all canopy locations, with more moderate responses in the mineral soils (0 – 10 cm and 11 – 20 cm; Figure 3.1-3.6, Table 3.2, Appendix A-2). There was a large, significant increase in pH (up to 2 pH units) in the litter of all ash-treated plots, and organic matter content in the litter was approximately 30% lower than in untreated plots (Figure 3.1, Figure 3.2).

Following application (July 2021), Ca was approximately 30-50% higher in the treatment plots, significantly so in the LFH layers of all three canopy locations (Figure 3.3, 3.4). Almost all increases in Mg in the LFH layers of the treated plots were significant with the most pronounced increases observed in the FH layer and significant increases extending through both soil horizons beneath sugar maples and in the mixed soil samples (Figure 3.3, 3.4). Potassium was significantly lower in the treated plots at each canopy location in the litter layer but was significantly higher in the FH and mineral layers (Figures 3.3-3.6).

One year after application, concentrations of most metals (Al, Fe, Mn, Cd, Cu, Ni, Pb, and Zn) in the litter layer were significantly higher (4 to 10 times) in the ash treated plots compared with untreated plots, but there were few significant differences in metal concentration between treated and untreated plots in deeper soil layers (Table 3.2, Appendix A-2).

Table 3.2. Average elemental metal concentrations (± SE) of the L and FH sampled beneath mature sugar maple (n = 12) and American beech (n = 12) trees and in mixed soil samples (n = 16) in the control and ash treated plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

	Pre-App		Post-App	
	Control	Treatment	Control	Treatment
------------------------------------ Sugar Maple Soils ------------------------------------				
Litter				
Al (mg kg^{-1})	267 (17.5)	410 (65.7)	375 (41.5)	1311 (263)***
Fe (mg kg^{-1})	332 (41.2)	586 (126)	608 (114)	1113 (162)**
Mn (mg kg^{-1})	1803 (124)	2210 (210)	1510 (136)	4925 (517)***
Cd (mg kg^{-1})	0.4 (0.2)	1.1 (0.2)*	0.3 (0.2)	1.2 (0.2)**
Cu (mg kg^{-1})	12.6 (1.2)	9.4 (1)	16.1 (0.5)	68.7 (12.0)***
Ni (mg kg^{-1})	0.9 (0.2)	0.9 (0.3)	4.4 (0.3)	5.4 (0.9)
Pb (mg kg^{-1})	2.0 (0.5)	2.6 (0.5)	2.5 (0.5)	10.3 (1.7)***
Zn (mg kg^{-1})	60.0 (4.6)	58.6 (4.6)	57.3 (4.5)	271 (38.9)***
FH				
Al (mg kg^{-1})	2124 (274)	3646 (644)	2284 (351)	1979 (236)
Fe (mg kg^{-1})	5593 (819)	7739 (1128)	5371 (645)	4212 (835)
Mn (mg kg^{-1})	1312 (249)	2003 (449)	918 (102)	2077 (408)
Cd (mg kg^{-1})	BDL	0.2 (0.1)	0.7 (0.5)	1.0 (0.2)**
Cu (mg kg^{-1})	13.7 (0.6)	13.2 (1)	18.2 (1.3)	20.1 (4.2)
Ni (mg kg^{-1})	3.6 (0.7)	5.3 (0.7)	4.9 (1.2)	4.9 (0.5)
Pb (mg kg^{-1})	32.6 (6.5)	35.6 (3.8)	18.1 (3.4)	19.3 (4.3)
Zn (mg kg^{-1})	63.0 (3.3)	69.5 (11.8)	55.6 (4.1)	92.9 (16.6)*
------------------------------------ American Beech Soils ------------------------------------				
Litter				
Al (mg kg^{-1})	246 (33.2)	275 (31.5)	279 (13.4)	1557 (214)***
Fe (mg kg^{-1})	276 (65.6)	337 (66.3)	393 (19.8)	1160 (161)***
Mn (mg kg^{-1})	1245 (108)	1701 (166)*	1151 (82.0)	4792 (498)***
Cd (mg kg^{-1})	0.3 (0.2)	0.8 (0.2)	0.3 (0.3)	1.0 (0.2)***
Cu (mg kg^{-1})	10.6 (1.2)	7.4 (0.8)*	15.3 (0.6)	82.0 (12.6)***
Ni (mg kg^{-1})	0.9 (0.2)	1.3 (0.4)	4.2 (0.4)	4.8 (0.8)
Pb (mg kg^{-1})	0.8 (0.3)	2.5 (0.8)*	2.8 (0.6)	13.7 (4.0)**
Zn (mg kg^{-1})	46.7 (5.3)	48.1 (3.2)	53.3 (5.3)	320 (45.4)***
FH				
Al (mg kg^{-1})	1491 (291)	2078 (486)	2082 (252)	2009 (318)
Fe (mg kg^{-1})	2885 (581)	4993 (960)	4692 (658)	4650 (934)
Mn (mg kg^{-1})	818 (141)	763 (122)	857 (202)	1205 (268)
Cd (mg kg^{-1})	BDL	BDL	0.8 (0.2)	0.7 (0.1)
Cu (mg kg^{-1})	11.1 (0.7)	11.6 (0.5)	18.5 (2.3)	16.4 (2.1)
Ni (mg kg^{-1})	2.7 (0.6)	4.5 (0.4)*	7.2 (1.4)	4.8 (0.3)
Pb (mg kg^{-1})	16.1 (3.6)	28.0 (4.1)	23.8 (7.4)	17.0 (2.9)
Zn (mg kg^{-1})	51.3 (3.8)	50.2 (1.5)	49.5 (6.7)	72.5 (7.1)**

------------------------------------- Mixed Soils -------------------------------------				
Litter				
Al (mg kg^{-1})	319 (33.7)	294 (29.1)	375 (33.6)	2116 (370)***
Fe (mg kg^{-1})	414 (63.3)	382 (79.2)	527 (53.3)	1265 (157)***
Mn (mg kg^{-1})	1617 (99.8)	1851 (102.9)	1597 (165)	6162 (565)***
Cd (mg kg^{-1})	0.4 (0.2)	0.9 (0.1)	0.3 (0.3)	1.6 (0.2)***
Cu (mg kg^{-1})	12.8 (0.8)	7.5 (0.8)***	16 (0.4)	97.4 (12.7)***
Ni (mg kg^{-1})	1.3 (0.2)	0.9 (0.2)	4.2 (0.3)	6.9 (0.9)*
Pb (mg kg^{-1})	1.5 (0.6)	2.6 (1)	2.2 (0.3)	13.1 (1.7)***
Zn (mg kg^{-1})	59.5 (4.1)	50.1 (2.9)	55.1 (3.7)	349 (42.7)***
FH				
Al (mg kg^{-1})	2096 (340)	2226 (380)	2746 (350)	2661 (509)
Fe (mg kg^{-1})	4738 (590)	5387 (907)	6358 (819)	5399 (898)
Mn (mg kg^{-1})	1310 (126)	1235 (247)	1326 (335)	2051 (290)*
Cd (mg kg^{-1})	0.1 (0)	BDL	1.9 (0.4)	1.0 (0.2)
Cu (mg kg^{-1})	11.8 (1.0)	12 (0.7)	21.1 (1.5)	20.0 (2.5)
Ni (mg kg^{-1})	3.2 (0.4)	4.5 (0.4)*	8.4 (1.2)	5.0 (0.4)*
Pb (mg kg^{-1})	29.9 (3.4)	32.1 (5.4)	38.3 (5.1)	23.1 (4.3)*
Zn (mg kg^{-1})	62.3 (4.9)	56.3 (4.9)	58.9 (5.3)	92.7 (12.1)**

*As and B were below their detectable limits and therefore removed.

3.4.3 *Fine Root Biomass and Chemistry*

Fine root biomass was similar in all canopy locations and no significant differences were observed between the control and treated plots collected beneath sugar maple or American beech trees (Table 3.3). Concentrations of base cations and P in fine roots tended to be higher in the ash treated plots, but this was only significant for K and Mg for both tree types and P in beech fine roots (Figure 3.7). Only B root concentrations were significantly higher in the treated plots of both species, whereas all other metal concentrations were similar or were lower in trees receiving ash (Table 3.4).

Table 3.3. Average fine root mass (mg cm^{-2} ± SE) beneath control and treated sugar maple (*n* = 24) and American beech trees (*n* = 24) in the 2021 sampling season. Significant differences to control indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$).

Treatment	Sugar Maple	American Beech
Control	1034 (95.2)	1133 (196)
6 Mg ha^{-1}	939 (69.4)	1343 (265)

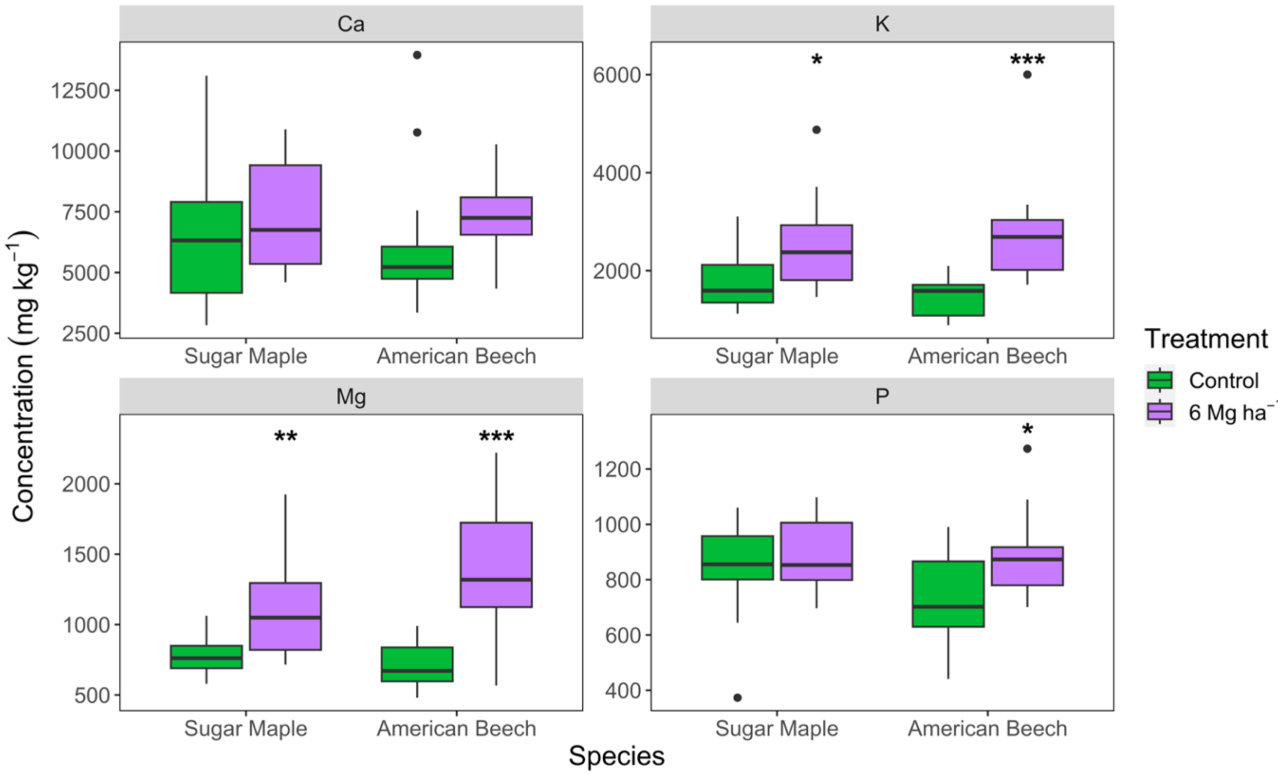

Figure 3.7. Average root nutrient concentrations beneath mature sugar maple (n = 12) and American beech (n = 12) trees during the 2021 sampling season. Significant differences between treatment and control indicated by an asterisk (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$) as determined by a Wilcoxon rank-sum test.

Table 3.4. Average metal concentrations (± SE) in roots beneath mature sugar maple (n = 12) and American beech (n = 12) trees in the 2021 sampling season. Significant differences to control indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

Element	Sugar Maple		American Beech	
	Control	Treatment	Control	Treatment
Al (mg kg^{-1})	848 (178)	879 (163)	470 (92.5)	556 (170)
Mn (mg kg^{-1})	438 (53.3)	577 (106)	253 (37.0)	339 (61.8)
Fe (mg kg^{-1})	1270 (204)	1159 (183)	651 (177)	814 (228)
Zn (mg kg^{-1})	84.9 (28.1)	47.2 (4.9)	48.3 (9.3)	34.8 (4)
Pb (mg kg^{-1})	36.8 (20.6)	7.5 (1.1)**	21.9 (11.2)	9.7 (3.1)
B (mg kg^{-1})	2.8 (1.1)	7.1 (1.7)*	3.7 (1)	9.8 (1.8)**
Cu (mg kg^{-1})	6.1 (1.3)	4.0 (0.7)	3.2 (1.3)	3.7 (0.7)
Ni (mg kg^{-1})	3.3 (0.6)	2.5 (0.2)	2.2 (0.2)	2.1 (0.2)

*As and Cd were below their detectable limits and therefore removed.

3.4.4 *Foliar Chemistry*

There were very few differences in foliar chemistry in ash treated trees one year after application. The notable exception was K that was significantly higher in sugar maple foliage (14,032 mg kg^{-1} in the treated plots compared to 7,551 mg kg^{-1} in the controls). Potassium concentrations in foliage also increased by approximately 30 % in American beech, (12,852 mg kg^{-1} in the treated plots compared to 9,838 mg kg^{-1} in the controls; Figure 3.8), although this difference was not significant. Foliar concentrations of Ca, Mg, and P as well as most metals in sugar maple (Al, Mn, Fe, Zn, Ni, and Cd) and American beech (Mn, Zn, Cu, and Ni) also tended to be higher in the treated plots, but these differences were small and insignificant (Figure 3.8, Table 3.5). Percent C and N were also similar across treatments (Table 3.5).

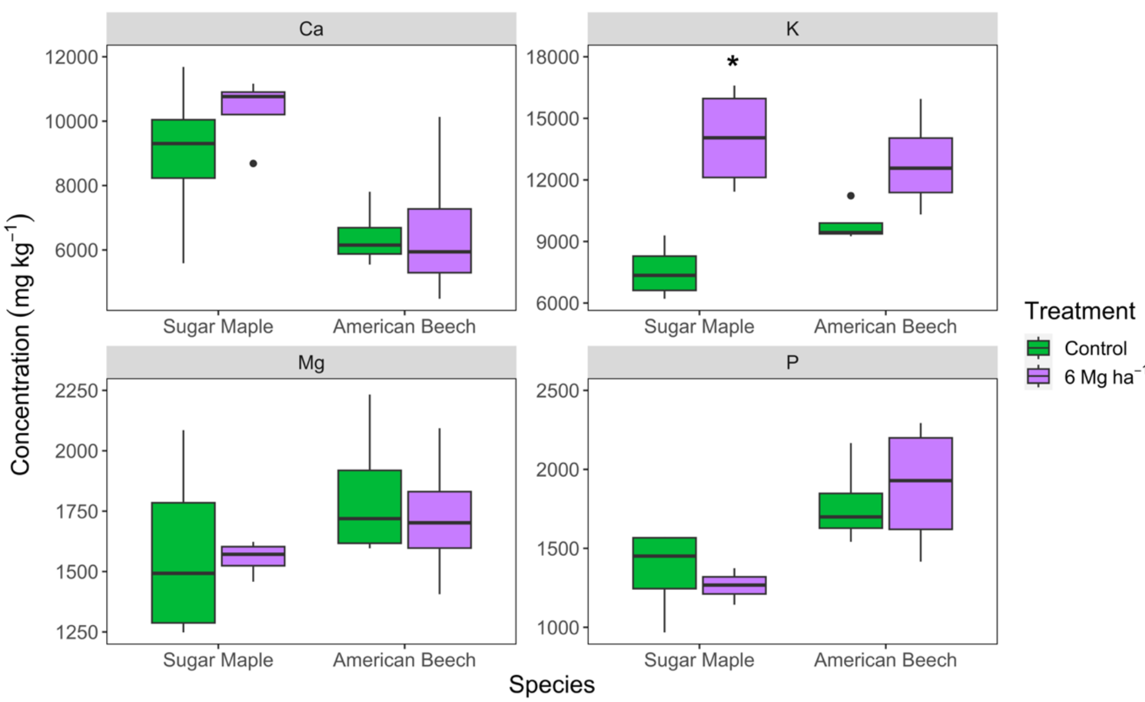

Figure 3.8. Average foliar nutrient concentrations in mature sugar maple (n = 4) and American beech (n = 4) trees during the 2021 sampling season. Significant differences between treatment and control indicated by an asterisk (*, $p < 0.05$) as determined by a Wilcoxon rank-sum test.

Table 3.5. Foliar C, N, and metal concentrations (mean ± SE) in mature sugar maple (n = 4) and American beech (n = 4) trees in the 2021 sampling season. No significant differences were found.

Element	Sugar Maple		American Beech	
	Control	Treatment	Control	Treatment
C (%)	46.1 (1.0)	45.3 (0.4)	47.0 (0.0)	46.5 (0.6)
N (%)	2.3 (0.6)	2.2 (0.2)	2.7 (0.5)	2.8 (0.1)
Al (mg/kg)	14.3 (1.1)	19.1 (2.5)	17.4 (1.2)	15.2 (2.2)
Mn (mg/kg)	885 (178)	1083 (150)	420 (51.5)	447 (96.7)
Fe (mg/kg)	49.9 (4.8)	58.3 (8.1)	61.6 (4.9)	58.1 (2.8)
Zn (mg/kg)	26.8 (3.1)	33.3 (2.6)	27.6 (3.2)	30.4 (4.1)
Cu (mg/kg)	2.9 (0.5)	2.6 (0.4)	6.4 (0.7)	7.2 (2.0)
Ni (mg/kg)	0.3 (0.1)	1.1 (0.7)	3.8 (0.6)	4.3 (1.1)

*As, B, Cd, and Pb were below the detection limit and therefore removed.

3.4.5 *Understory Vegetation Diversity, Richness, and Abundance*

Understory vegetation of vascular plants was similar both years after application. A generalized linear mixed effects model indicated that a significant main effect of year was found on Shannon's Diversity (H), Simpson's Diversity (D), and species richness (S_r). However, no main effect of treatment or interaction effect was found on any of these indices (Table 3.6). Higher values for H and D indicate greater diversity and therefore communities tended to be more diverse in the ash treated plots in both years, as well as in both the control and ash treated plots in 2022 compared to 2021. Species richness was also higher in the treatment plots compared to the controls each year and in both the control and treated plots in 2022 compared to 2021 (Table 3.6).

Table 3.6. Diversity indices per treatment (± SE) one year (2021) and two years (2022) after 6 Mg ha^{-1} NIWA treatment. Richness is measured as number of individuals per 24 m^2. Significant differences are indicated with letters as determined by a post-hoc pairwise Tukey HSD with Bonferroni correction (*, $p < 0.05$).

	2021		2022	
Diversity Indices	Control	Ash	Control	Ash
Shannon-Weiner Diversity (H)	1.10 (0.16)^{a}	1.49 (0.20)^{ab}	1.91 (0.09)^{bc}	2.29 (0.13)^{c}
Simpson Diversity (D)	0.44 (0.08)^{a}	0.58 (0.08)^{ab}	0.75 (0.05)^{ab}	0.85 (0.04)^{b}
Richness (S_r)	15.0 (0.96)^{a}	22.0 (1.31)^{ab}	21.0 (0.65)^{bc}	25.0 (0.85)^{c}

Species abundance also remained similar between treatments each year. Results of a generalized linear mixed effects model revealed a significant main effect of year and a significant interaction effect. In 2022, total abundance was significantly lower in the control plots but higher in the treatment plots compared with 2021 (Figure 3.9). Sugar maple seedling abundance was approximately 50% lower in both control and treated plots in 2022, whereas American beech seedling abundance was ~30% higher in the control plots in 2022 compared with 2021, but almost identical in the ash treated plots between years.

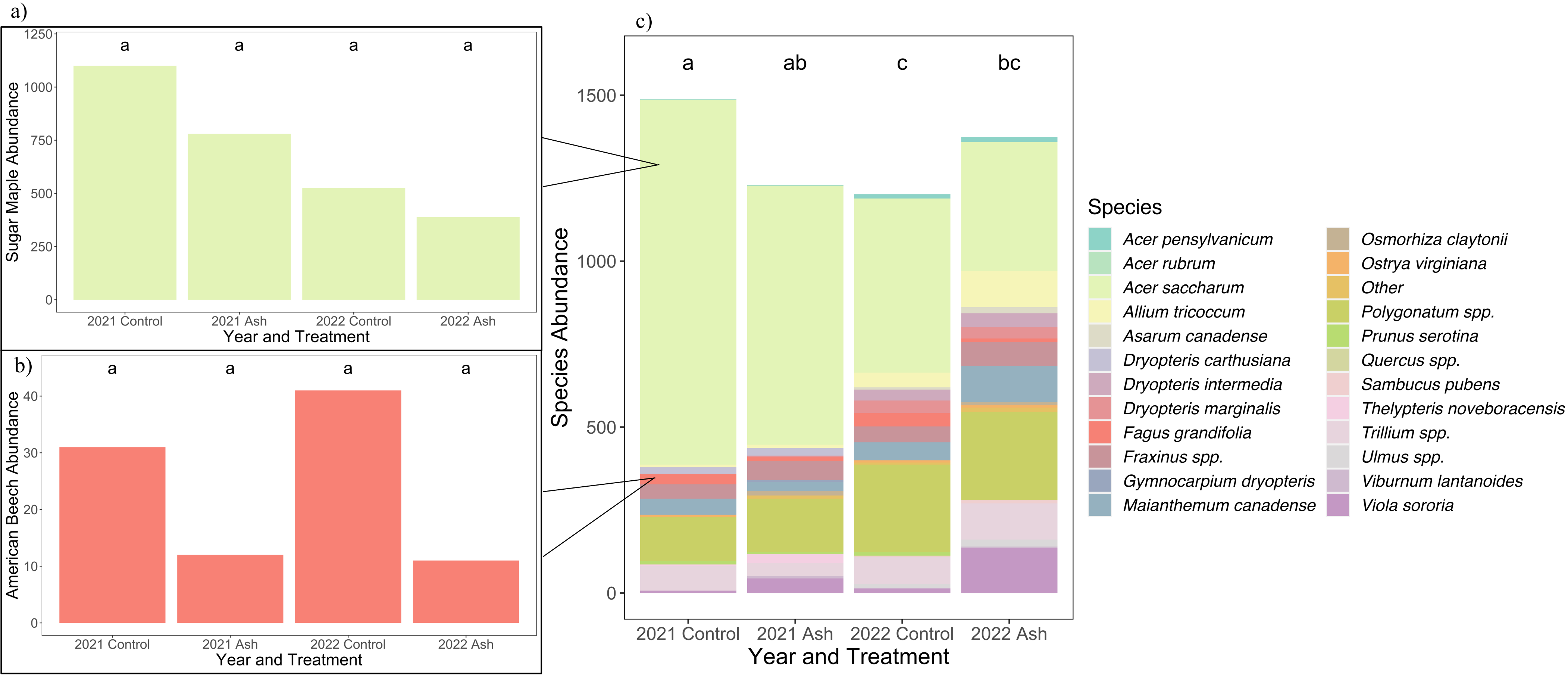

Figure 3.9. Species abundance data one year (2021) and two years (2022) after 6 Mg ha^{-1} NIWA addition for a) sugar maple, b) American beech, and c) total species per plot. Species with less than two individuals per plot were grouped into other and include: *Betula spp., Convolvulus spp., Corylus cornuta, Cypripedium spp., Tilia americana, Rubus idaeus, Sambucus canadensis*. Significant differences in total species abundance per plot are indicated by letters ($p < 0.05$).

3.5 Discussion

3.5.1 *Non-Industrial Wood Ash Chemistry*

The chemical composition of the NIWA obtained in this study is consistent with previous research and is highly alkaline averaging a pH of 13, a value at the upper end of the 8 – 13 range commonly reported for wood ash (Pitman, 2006; Augusto et al., 2008; Azan et al., 2019; Deighton & Watmough, 2020; Deighton et al., 2021). It is also common to find low concentrations of N and organic matter due to volatilization during combustion (Pitman, 2006). Concentrations of Ca, K, and Mg (27%, 9%, and 2%, respectively) were at the higher end of the range of concentrations reported previously (Fritze et al., 1994; Saarsalmi et al., 2004; Pitman, 2006; Azan et al., 2019; Deighton & Watmough, 2020; Deighton et al., 2021). Concentrations of most metals in NIWA also remained below CM1 limits. Only Cu (164 mg kg^{-1}) and Zn (503 mg kg^{-1}) fell above their CM1 limits (100 and 500 mg kg^{-1}, respectively) but they remained substantially lower than CM2 (1700 and 4200 mg kg^{-1}, respectively; Government of Ontario, 2002). These concentrations are consistent with those found in other NIWA samples (Azan et al., 2019; Deighton & Watmough, 2020) and are within the ranges reported for other wood ash sources (Saarsalmi et al., 2004; Pitman, 2006; Augusto et al., 2008). Both Cd and As approached their CM1 limits, but are still similar to values reported previously (Pitman, 2006; Azan et al., 2019; Deighton & Watmough, 2020) and are well below CM2 concentrations.

3.5.2 *Soil Chemistry*

Prior to ash application, control and treated plots had similar soil characteristics. Soils were acidic ranging from a pH of 3.7 – 4.8 across all horizons, and both exchangeable base cation and total metal concentrations were low. Differences in soil chemistry below trees can be species-specific (Ott & Watmough, 2021) and optimal soil chemical properties for survivorship of sugar maple and American beech are often contrasted in the literature. Sugar maple prefer less acidic soils and higher concentrations of base cations such as Ca, while beech is usually inversely related to these properties and often tolerates much higher concentrations of Al (Dijkstra, 2001). In this study, however, few differences were noted in the pre-treatment soils beneath maple and beech trees and in the mixed soil samples.

The first year following ash application soil chemistry responded similarly across all three canopy locations. The most pronounced changes were observed in the organic horizons where soil acidity and organic matter decreased, and base cation and metal concentrations increased. Significant increases in organic horizon soil pH are attributed to the acid neutralizing capacity of the NIWA as a result of its alkalinity (Pitman, 2006; Augusto et al., 2008) and increases of 0.5 – 1.5 units are commonly seen in the organic horizon pH for up to 30 years post-application (Fritze et al., 1994; Saarsalmi et al., 2001, 2004; Ozolinčius et al., 2007b; Saarsalmi et al., 2012; Arseneau et al., 2021). Slow vertical leaching of soluble cations (due to retention in the organic horizons) typically delays the migration of the neutralization effects to deeper layers of the soil. Therefore, increases in mineral soil pH are not often observed shortly after application (<3 years; Ozolinčius et al., 2007b; Arseneau et al., 2021). Significant decreases in acidity in the upper and lower mineral soil pH have been observed as early as 5 and 10 years after application, respectively (Saarsalmi et al., 2004). On the other hand, organic matter content decreased significantly in the litter layer at all three canopy locations from approximately 90% to as low as 53%. No effect on organic matter (Deighton & Watmough, 2020; Deighton et al., 2021) as well as a decrease in organic matter from wood ash (Saarsalmi et al., 2004) and lime (Moore et al., 2008) have been noted in the literature. Decreases in organic matter over the long-term (10 year) have been observed with high doses of lime (20 Mg ha^{-1}; Moore et al., 2008) and while it is possible that decreased acidity in the soil accelerates organic matter breakdown over time (Pitman, 2006) this result is not always the case (Moore et al., 2015). Given that the soil in this study was sampled shortly after application it is most likely that the decreases noted here are a result of NIWA that has a low organic matter content remaining on the surface of the soil and being incorporated into the litter samples during sampling.

Post-application, exchangeable Ca concentrations in the soil were significantly higher in the treated litter and FH layer of all three canopy locations (+35% and +87% beneath sugar maple, +71% and +166% beneath American beech, and +46% and +121% in mixed soil samples, respectively). Whilst sugar maple trees tend to respond positively to higher calcium concentrations, American beech trees often do not respond at all (Long et al., 1997; Halman et al. 2013; Long et al., 2022). Similar results were observed for Mg, except the increased concentrations were not significant beneath sugar maple trees in the litter layer likely because baseline concentrations were lower to begin with. Short-term (1 – 3 years) increases in Ca and Mg concentrations in the organic soil horizons after wood ash application are common (Long et al., 1997; Ludwig et al., 2002; Hansen et al., 2018; Deighton & Watmough, 2020; Arseneau et al., 2021) and higher concentrations of both elements have persisted for 10 – 30 years post-application (Saarsalmi et al., 2001, 2004, 2012; Long et al., 2022). Increases in Ca and Mg concentrations also extended to the mineral soils in the treatment plots where Ca was significantly higher in the upper mineral soil beneath sugar maple trees and Mg was significantly higher in both the upper and lower mineral soil horizons beneath sugar maple trees and in mixed soil samples. Faster movement of Mg compared with Ca through the soil profiles has been observed after liming (Long et al., 1997; Moore et al., 2008) and wood ash application (Saarsalmi et al., 2004; Ozolinčius et al., 2007b) and therefore support the conclusion that the solubility and plant availability of macronutrients in the wood ash goes in the order K > Mg > Ca (Ozolinčius et al., 2007b). Thus, the high affinity of base cations such as Ca and Mg for soil exchange sites over other macronutrients such as K results in increased concentrations that not only persist for longer in the organic horizons but lead to sustained increases in deeper soil profiles over longer periods of time (Long et al., 2022). Interestingly, Dijkstra (2001) and Dijkstra et al. (2003) found that sugar maple tree litter is more susceptible to base cation leaching than American beech trees, possibly explaining the significantly higher concentrations of Ca in the upper mineral horizon and Mg in the upper and lower mineral horizons observed beneath sugar maple trees in the treated plots, but not beneath American beech. Additionally, Duchesne and Ouimet (2009) found that soil Ca and Mg concentrations on sites where American beech were present were about half those on the sites where American beech were absent, suggesting decomposition and regulation of organic acid concentrations may occur at a slower rate in soils beneath American beech trees (Dijkstra et al., 2001). Potassium behaved similarly except for in the litter layer of the treated plots where concentrations were significantly lower at all three canopy locations; this is likely due to its displacement off soil exchange sites by Ca and Mg (Long et al., 1997; Moore et al., 2008; Reid & Watmough, 2014). Significantly higher concentrations of K were observed in the FH, upper, and lower mineral horizons for all three canopy locations. This is consistent with other wood ash research with similar doses (Jacobson et al., 2004; Saarsalmi et al., 2004, 2014).

While some differences were observed in baseline soil metal concentrations, these differences were small. Following application, significantly higher concentrations were observed in almost all metals in the litter layer of the treatment plots at all three canopy locations. However, these increases were largely retained in the litter as significantly higher concentrations were observed in far fewer metal concentrations in the FH layer, and none in the mineral horizons. Increases in metal concentrations in the organic horizons were expected shortly after application and are consistent with other findings (Hansen et al., 2018; Deighton & Watmough, 2020). In the FH layer, only significantly higher concentrations of Cd were observed beneath sugar maple trees, but higher concentrations of Zn were observed at all three canopy locations. Significantly higher concentrations of Mn were also observed in the FH layer in the mixed soil samples. In some cases Zn has been observed to increase in wood ash treated plots (Deighton & Watmough, 2020; Deighton et al., 2021) but the risk of Zn toxicity is low as metal mobility has not been found to increase after application of doses ranging from 3 to 30 Mg ha^{-1} (Hansen et al., 2018) and metal mobility is typically limited at high pH (Kicińska et al., 2022) and in forest soils with significant amounts of organic matter (Augusto et al., 2008; Violante et al., 2010). Lower organic matter content in the litter layer, combined with the retention of metals in the organic horizons, is likely a result of ash that is low in organic matter remaining on top of the soil at the time of sampling.

3.5.3 *Fine Root Biomass and Chemistry*

Fine root biomass was not significantly different between the control and treated plots for either sugar maple or American beech trees following NIWA application. Fine root biomass in this study was similar to the average root biomass of beech, maple and yellow birch reported by Safford (1974). The response of fine roots to soil amendments is variable, and previous studies have reported a significant decrease in live root biomass (Persson & Ahlström, 1990; Helmisaari et al., 2009) with a significant increase in specific root length (Clemensson-Lindell & Persson, 1995) or no effect on biomass with a decrease in total root length and number of root tips (Ozolinčius et al., 2007b) following lime or wood ash application.

Potassium and Mg concentrations in fine roots increased significantly in the treated plots beneath sugar maple and American beech trees whilst no differences were observed in Ca, though concentrations in the treated plots were slightly higher. A significant increase was also observed in P concentrations beneath American beech trees. Higher K and Mg concentrations in the roots reflect soil chemistry changes, particularly in the mineral horizons. It is interesting that significant increases were not observed in fine root Ca concentrations taken from the FH layer given that 35-53% of sugar maple and 32-60% of American beech fine root biomass exists in the forest floor (Yanai et al., 2008). Large increases in Ca, Mg, K, and P concentrations are commonly observed in fine roots following wood ash application (Augusto et al., 2008). However, these results are somewhat in contrast to those reported by Brunner et al. (2004) that suggest that Ca and Mg soil concentrations are significantly related to fine root concentrations, whilst K is not.

Enhanced heavy metal concentrations were not observed in the roots of the treated plots beneath either sugar maple or American beech trees, and these results are consistent with other findings (Brunner et al., 2004). Some research even suggests that concentrations of Al, Fe, and Mn decrease significantly in fine roots following application (Brunner et al., 2004; Augusto et al. 2008). Significant decreases were observed in Pb concentrations beneath sugar maple trees, and although not significant, decreases were observed in Fe, Zn, Cu, and Ni concentrations beneath sugar maple trees and in Zn and Pb concentrations beneath American beech. Fine root concentrations of B were also significantly higher beneath both species in the treated plots. Boron is an essential plant nutrient required for cell wall formation and membrane function (Lehto et al., 2010) and is one of the few metals that increases in availability with increases in pH (Padbhushan & Kumar, 2017). Given that soil B concentrations reported here were below their detectable limit it is unlikely that concentrations pose a risk of phytotoxicity.

3.5.4 *Foliar Chemistry*

Foliar chemical responses were relatively muted between the control and treated plots and were consistent between both species. Only K significantly increased in sugar maple foliage, though it was slightly higher in American beech foliage. These results are in contrast to those reported by others where Ca and Mg concentrations significantly increased in sugar maple foliage one year and 30 years after liming (Long et al., 1997, 2022) and four years after wood ash application (Arseneau et al., 2021). These doses were much larger (22.4 Mg ha^{-1} lime and 20 Mg ha^{-1} wood ash, respectively) than the one applied in this study (6 Mg ha^{-1}) and as a result significant increases in foliar base cation concentrations may take longer to translate from the soil (Augusto et al., 2008; Reid & Watmough, 2014). There is relatively little research on the concentration of nutrients and metals in American beech foliage, especially in response to treatments such as lime or wood ash, but the concentrations reported here are in line with those reported by Park and Yanai (2009). Similarly, C, N and metal concentrations did not differ significantly between the control and treated plots for either species. These results are not surprising since the metal concentrations added to the soil are quite low and their mobility is mostly restricted to the litter horizon. These results also do not translate directly from fine root concentrations, aside from increases in K in sugar maple roots and foliage, unlike those reported in Park and Yanai (2009). The responses of both sugar maple and American beech were more similar than expected based on differing tolerances to acidic soils (Halman et al., 2015). However, these results are consistent with those found by Wills et al. (2023), who evaluated foliar responses of both species to a gradient of soil acidification. They hypothesized that the observed deviation from the expected response to acidic soils may be controlled by other factors such as a low threshold of foliar base cation concentrations or by regulation elsewhere, such as in the roots (Wills et al., 2023).

3.5.5 *Understory Diversity and Composition*

Sugar maple and American beech seedling abundance exhibited similar patterns in the control and treated plots each year. Decreases were observed in sugar maple seedling abundance in both the control and treated plots from 2021 to 2022, while American beech abundance was slightly higher in the control plots in 2022 compared to the controls in 2021 and was almost identical in the ash treated plots each year. For both species, seedling abundance was lower in the ash treated plots each year, but these differences were not significant. The responses of maple and beech seedlings to soil amendments such as lime and wood ash are variable in the literature. Liming applications have resulted in increased sugar maple regeneration (Long et al., 1997; Juice et al., 2006) with both a decreased effect (Moore et al., 2008, 2012) and no effect on American beech regeneration (Duchesne et al., 2013). From a forest management perspective, particularly in sugar maple bushes, research has suggested that the combination of liming and understory cleaning (evenly distributing saplings and providing 1 m for crown expansion; Moore et al., 2012) or releasing (removal of all competitors within 1 m of crown perimeters; Duchesne et al., 2013) are necessary to give sugar maple seedling regeneration a competitive advantage over American beech. It is also important to consider that aboveground beech biomass tends to decline at increasing rates corresponding with BBD stand infection duration (Cale et al., 2017) and it is not clear how the dynamics of the sugar maple-American beech dichotomy will respond to this additional environmental pressure, particularly in the case of wood ash application.

Overall, understory vegetation exhibited significant differences from one to two years post-application, but these differences were not observed between treatments within years. Although not significant, all three indices measured (H, D, and S_r) indicated greater diversity and richness in the ash treated plots compared to the controls both years. Additionally, species abundances of herbs such as wild leek (*Allium tricoccum*), trillium (*Trillium* sp.), and violet (*Viola sororia*) appeared to be much greater in the ash treated plots in 2022 than in 2021 and compared to the control plots both years. While it does appear that the statistical differences in composition are driven more by year than by treatment, increases in the abundance of herbs and grasses following wood ash application are commonly found in the literature, while decreases are often seen in mosses and shrubs (Augusto et al., 2008). Other research on sandy soils have found no significant differences when evaluating the H of understory vegetation prior to, one and two years after wood ash application (Ozolinčius et al., 2007b) as well as on plant community composition or abundance (Arvidsson et al., 2002; Jacobson et al., 2004), but drastic changes have been reported in species composition in a drained peatland up to 50 years following application (Moilanen et al., 2002). Therefore, the response of understory vegetation to wood ash addition may depend on soil type. Furthermore, even when no significant changes are found in vascular plants, immediate visible damage and reductions have been observed in bryophytes, lichens, and mosses (Ozolinčius et al., 2007a; Økland et al., 2022) but recovery of these species from the visible damage has been observed within 3 – 5 years (Jacobson & Gustafsson, 2001). Initial damage to bryophytes, lichens, and mosses may be a result of how the wood ash is prepared for application. It is possible that loose ash causes an initial shock to the non-vascular plants because of its high pH, whereas this effect was not observed when wood ash was first pelletized (Jacobson & Gustafsson, 2001) or stabilized with water and then crushed (Arvidsson et al., 2002). Ultimately, these results suggest that wood ash does not significantly alter species composition in the short-term if applied in similar doses.

3.6 Conclusions

Non-industrial wood ash samples were high in macronutrient concentrations and low in metal concentrations, with almost all metals remaining below provincial guidelines. Soil chemistry responded similarly at all three canopy locations with the most pronounced changes observed in the litter and FH layers, notably with K and Mg migrating to deeper soil horizons. Increased metal concentrations were restricted to the litter layer and the observed decrease in litter organic matter concentration was likely a result of ash remaining on the soil at the time of sampling. Fine root biomass was unchanged in both sugar maple and American beech trees, but root Mg and K concentrations increased significantly. Foliar responses were limited in both species, though K increased significantly in sugar maple trees. Understory vegetation was also not affected by NIWA treatment, and observed differences appeared to be driven by year rather than by treatment. Overall, this evidence suggests that NIWA provides an effective alternative as a soil amendment that can not only fertilize forest soils but also divert waste from landfills.

4. General Conclusions

The main goal of this thesis was to evaluate the use of NIWA as a forest soil amendment (at a rate of 6 Mg ha^{-1}) to counteract the legacy effects of acidic deposition in south-central Ontario. This was addressed in two main ways: first, by evaluating the chemical composition of NIWA, and second by assessing its effect on sugar maple sap, forest soils, sugar maple and American beech tree roots and foliage, and understory vegetation. Aside from its acid-neutralizing capacity and high concentrations of macronutrients, the most important component to assess in NIWA was its concentrations of regulated metals. Ultimately, NIWA that has been amalgamated from many sources (including different harvesting locations and ovens) contains metal concentrations that fall well-within provincial regulatory guidelines for safe land application. Certain species, such as yellow birch (Deighton & Watmough, 2020), should be avoided to maintain these low concentrations. Once applied, NIWA is effective at neutralizing soil acidity in a short period of time (<1 year) while also increasing concentrations of essential macronutrients. Increased metal concentrations in the organic soil horizons were observed following addition, but there was no evidence to suggest that these concentrations are toxic to forest flora or will be mobile enough in the future to be a cause for concern. Increases were observed in nutrient concentrations in sugar maple foliage and in maple and beech roots, while fine root biomass and root nutrient concentrations were unaffected by ash application. There were also no significant changes in understory vegetation. Lastly, NIWA does not appear to significantly alter sugar maple sap yield, sweetness, or chemistry, suggesting it can safely be used as a forest soil amendment or fertilizer in sugarbushes.

Sugar maple syrup production is a massive industry in Canada. Given the impact of climate change on the beginning and length of the production season (Duchesne et al., 2009; Houle et al., 2015) and changes in the distribution of sugar maple (Matthews & Iverson, 2017; Jain et al., 2021) as well as its optimal climate range (Rapp et al., 2019), proper management practices are essential to sustain or potentially increase current production levels in Canada. It is well recognized that sugar maple trees have a greater need for nutrients (most notably Ca) relative to other species, and therefore nutrient deficiencies from acidic deposition should be addressed to ensure healthier trees that grow larger and live longer. Additionally, if NIWA increases tree growth in the longer term as suggested in other studies (Pitman, 2006; Augusto et al., 2008; Huotari et al., 2015; Reid & Watmough, 2016), then application could also result in sweeter sap. Larger and healthier trees produce more sap, and if sugar concentrations can potentially increase (i.e., through carbon sequestration) then total maple syrup yields may also increase substantially. Ultimately, gaining a better understanding of whether NIWA can influence sugar maple yields on nutrient-poor soils and/or increase sugar maple sweetness over the long-term will be necessary to take advantage of the full potential of maple syrup production in future climate scenarios and could be beneficial for future management practices of this cultural and economic resource in North America.

Furthermore, few differences were found in the short-term (one year) response of sugar maple and American beech trees to NIWA application. Soils beneath the canopies of both species responded positively to NIWA addition with increases in nutrient concentrations, and similar increases were also observed between species in the roots, however only sugar maple foliage exhibited significant increases in K. Generally, sugar maple responds positively to fertilizer or amendments while American beech do not often exhibit any changes. While this site was acidic and nutrient-poor, both critical foliar concentrations and DRIS indices for sugar maple did not indicate mineral nutrient deficiencies, which may account for the limited response observed in both maple and beech trees. Nonetheless, these results suggest that NIWA addition can enhance nutrient availability for both species; increased nutrient availability is essential for maple tree health because of its greater demand for nutrients, but it may also increase beech resistance to the scale insect that initiates beech bark disease (BBD, Cale et al., 2015; Stephanson & Coe, 2017) and thus potentially delay or even prevent infection for some trees. These implications could help inform decisions regarding ongoing BBD management.

The results presented in this thesis suggest that NIWA addition is an effective fertilizer or amendment for neutralizing acidified soils as well as replenishing nutrients that have been lost over the previous few decades. By focusing on different components such as the soils, tree roots and foliage, sap and understory vegetation, as well as two common species (sugar maple and American beech) that are prominent in northeastern north American forests, this thesis provided insights into how NIWA cycles through forest ecosystems. Not only are the risks of metal toxicity limited in the short-term through retention in organic matter, but application may be beneficial enough to both species to influence future forest management practices. There are, however, limitations to this study that can be considered in future research. Longer-term responses of sugar maple sap to NIWA addition should be considered and would allow for more conclusive results on whether sap yield or sap sweetness can be influenced by catering to favourable soil conditions for sugar maple. Additionally, the relationship between increased nutrient availability and disease-resistance could be further explored in the context of American beech exposure to BBD. Lastly, metal mobility could be monitored over a longer time span to ensure that increases observed in the organic soil do not translate to deeper soil horizons, groundwater, or tree components (i.e., roots and foliage). Overall, these results suggest that amalgamated NIWA can be safely applied at a rate of 6 Mg ha^{-1} as a forest soil amendment or fertilizer.

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6. Appendices

A-1. Maximum allowable soil metal concentrations for NASM application.

Metals	Maximum Concentration (mg/Kg soil, dw*)[†]
As	14
Cd	1.6
Cu	100
Ni	32
Pb	60
Zn	220

[†]Nutrient and Management Act, 2002.
*dw, dry weight

A-2. Average elemental metal concentrations (± SE) in the mineral soil sampled beneath mature sugar maple (*n* = 12) and American beech (*n* = 12) trees and in mixed soil samples (*n* = 16) in the control and ash treated plots prior to (2020) and after (2021) ash application. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

	Pre-App Control	Treatment	Post-App Control	Treatment
------ Sugar Maple Soils ------				
0-10 cm				
Al (mg kg^{-1})	8096 (1372)	9965 (1059)	10458 (1030)	8666 (1137)
Fe (mg kg^{-1})	16326 (931)	18029 (1197)	15496 (730)	14070 (654)
Mn (mg kg^{-1})	326 (79.8)	765 (190)	180 (45)	458 (140)
Cd (mg kg^{-1})	BDL	BDL	BDL	BDL
Cu (mg kg^{-1})	2.5 (1.4)	2.5 (1.1)	6.0 (0.6)	6.8 (0.8)
Ni (mg kg^{-1})	4.5 (1.6)	3.0 (0.7)	3.6 (0.4)	3.8 (0.4)
Pb (mg kg^{-1})	16.0 (2.4)	12.3 (2.5)	8.5 (2.6)	6.5 (1.3)
Zn (mg kg^{-1})	33.8 (3.2)	36.3 (2.9)	25.4 (2)	26.7 (2.7)
11-20 cm				
Al (mg kg^{-1})	10509 (1437)	12949 (650)	13800 (1113)	12776 (1094)
Fe (mg kg^{-1})	15060 (944)	18051 (1028)*	13438 (743)	14605 (965)
Mn (mg kg^{-1})	190 (38.9)	745 (209)*	155 (50.9)	310 (106)
Cd (mg kg^{-1})	BDL	BDL	BDL	BDL
Cu (mg kg^{-1})	0.5 (0.4)	1.1 (0.5)	6.7 (0.9)	7.1 (0.8)
Ni (mg kg^{-1})	2.7 (0.9)	4.6 (0.8)	3.7 (0.3)	3.6 (0.3)
Pb (mg kg^{-1})	3.7 (1.4)	5.9 (1.1)	0.1 (0.1)	0.5 (0.3)
Zn (mg kg^{-1})	29.1 (2.8)	41.8 (4.4)**	24.4 (2)	23.8 (2.5)
------ American Beech Soils ------				
0-10 cm				
Al (mg kg^{-1})	7615 (1101)	8498 (1134)	8966 (934)	9025 (1205)
Fe (mg kg^{-1})	14412 (1397)	19173 (1477)*	14332 (1141)	14526 (775)
Mn (mg kg^{-1})	175 (60.0)	152 (41)	149 (40.2)	105 (19.7)
Cd (mg kg^{-1})	0.1 (0.1)	BDL	BDL	BDL
Cu (mg kg^{-1})	0.8 (0.5)	1.0 (0.4)	3.1 (0.5)	3.0 (0.4)
Ni (mg kg^{-1})	4.0 (1.4)	3.2 (1.3)	2.8 (0.3)	3.1 (0.3)
Pb (mg kg^{-1})	18.3 (5.8)	19.7 (3.6)	5.9 (1.1)	6.7 (1.4)
Zn (mg kg^{-1})	25.7 (3.1)	27.0 (1.5)	20.6 (1.7)	20.2 (1.7)
11-20 cm				
Al (mg kg^{-1})	9895 (1047)	11562 (1123)	11970 (1065)	12806 (973)
Fe (mg kg^{-1})	13095 (1254)	18714 (1472)*	12182 (1178)	15139 (993)
Mn (mg kg^{-1})	129 (30.3)	129 (26.6)	107 (28.3)	98.9 (25.8)
Cd (mg kg^{-1})	BDL	BDL	BDL	BDL
Cu (mg kg^{-1})	0.1 (0.1)	0.3 (0.2)	2.9 (0.7)	5.2 (1.6)
Ni (mg kg^{-1})	2.6 (0.8)	3.7 (0.8)	2.7 (0.3)	3.1 (0.3)
Pb (mg kg^{-1})	7.3 (3.2)	6.9 (1.6)	0.6 (0.4)	0.3 (0.2)
Zn (mg kg^{-1})	24.3 (2.5)	25.9 (2.1)	18.2 (1.2)	19 (1.7)

------------------------------------- Mixed Soils -------------------------------------				
0-10 cm				
Al (mg kg^{-1})	7654 (681)	7944 (892)	9807 (616)	9385 (958)
Fe (mg kg^{-1})	15955 (753)	16647 (939)	15254 (677)	14706 (812)
Mn (mg kg^{-1})	320 (60.9)	314 (80.8)	372 (190)	285 (68.2)
Cd (mg kg^{-1})	BDL	BDL	BDL	BDL
Cu (mg kg^{-1})	1.5 (0.9)	0.9 (0.3)	4.1 (0.7)	3.6 (0.7)
Ni (mg kg^{-1})	3.7 (1.2)	4.3 (1.3)	4.2 (0.5)	4.1 (0.4)
Pb (mg kg^{-1})	15.6 (5.1)	19.5 (3.2)	5.7 (1.4)	6.0 (1.1)
Zn (mg kg^{-1})	31.9 (3.2)	30.6 (1.9)	27.0 (2.1)	23.3 (2.1)
11-20 cm				
Al (mg kg^{-1})	11843 (555)	11713 (833)	14297 (968)	13514 (984)
Fe (mg kg^{-1})	17048 (996)	18145 (1300)	12714 (811)	14876 (761)
Mn (mg kg^{-1})	243.2 (43.9)	280 (67.8)	360 (250)	163 (34.7)
Cd (mg kg^{-1})	BDL	BDL	BDL	BDL
Cu (mg kg^{-1})	0.4 (0.4)	0.8 (0.6)	4.2 (0.6)	5.4 (1.1)
Ni (mg kg^{-1})	2.5 (0.6)	3.6 (0.6)	4.0 (0.4)	4.2 (0.6)
Pb (mg kg^{-1})	4.2 (1.2)	6.4 (1.4)	0.6 (0.6)	1.0 (0.7)
Zn (mg kg^{-1})	30.7 (2)	30.0 (2.0)	23.5 (1.4)	22.0 (1.5)

*As and B were below their detectable limits and therefore removed.