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Article

Sugar maple sap, soil, and foliar chemistry in response to non-industrial wood ash fertilizer in Muskoka, Ontario

Shelby M. Conquer[a], **Norman D. Yan**[b,c], **and Shaun A. Watmough**[a]

[a]School of the Environment, Trent University, Peterborough, ON K9L 0G2, Canada; [b]Friends of the Muskoka Watershed, P.O. Box 416, Bracebridge, ON P1L 1T7, Canada; [c]Department of Biology, York University, Toronto, ON M3J 1P3, Canada

Corresponding author: **Shelby M. Conquer** (email: shelbyconquer@trentu.ca)

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 $\mathrm{Mg \cdot ha^{-1}}$) 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, significant increases were observed in the treated plots in the soil pH and base cations (Ca, K, and Mg) in the surface soil horizons, and metal concentrations in the litter. Sap yield in the control plots was significantly lower in the first-year post-application than in the second year, 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 1 year following application, except for K that was twice as high in the treated plots. Ultimately, NIWA is unlikely to significantly alter sugar maple sap chemistry, indicating that it is a viable nutrient supplement that can enhance soil fertility in sugar bushes with no impact on sap sweetness.

Key words: sugar maple (*Acer saccharum* Marshall), non-industrial wood ash (NIWA), sap yield, sap sweetness, metal toxicity

1. Introduction

Despite reductions in acidic deposition since the Canada–United States Air Quality Agreement in 1991 (Driscoll et al. 2001; Environment and Climate Change Canada 2016), historic depletion of exchangeable base cation pools and slow weathering rates have slowed the pace of natural chemical recovery in soils and surface waters (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 has suffered decreased vigor, canopy condition, growth rates, and recruitment on these nutrient-depleted soils (McLaughlin and Wimmer 1999; Driscoll et al. 2001; Duchesne et al. 2002; Sullivan et al. 2013).

Soil amendments including lime, wood ash, and wollastonite have shown beneficial effects on sugar maple (Wilmot et al. 1996; Juice et al. 2006; Moore et al. 2015; Arseneau et al. 2021; Moore and 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 and 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 and Ouimet 2010; Long et al. 2011; Deighton and 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 to 2022 (AAFC 2022). Previous studies evaluating combinations of fertilizer additions have produced mixed results on sap yield and sweetness, defined here as sucrose concentration. For example, Wilmot et al. (1995) reported that fertilizer additions (primarily Ca, Mg, and K) in northern Vermont did not have an effect on sap sweetness 2 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 been shown to increase sap sweetness 2 years after application, but foliar P was negatively correlated with sweetness and no relationship was observed between Ca addition and sweetness (Wild and Yanai 2015). On the other hand, Moore et al. (2020) found that liming improved yield and increased sap sweetness up to 20%, 18 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 softwood, sawdust, woodchips, and bark (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). 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 and Watmough 2020), large variations are observed in NIWA chemistry, particularly when produced from species-specific feedstock (Deighton and 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. In press).

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-agricultural source material (NASM). Non-agricultural source materials are regulated to ensure that 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 an NASM if the concentration of any regulated metal is above CM2 guidelines (Hannam et al. 2016).

Each year, Ontario alone produces approximately 18 000 tonnes 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 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 and 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 is 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 and foliage with a limited increase in 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. Materials and methods

2.1. 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 2023*a*). 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 2023*a*). The 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 (Tables 4 and 5) and diagnosis and recommendation integrated system (DRIS) norms (Table 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 $m^2 \cdot ha^{-1}$. 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 $mg \cdot L^{-1}$; Reid and Watmough 2016). The Camp Big Canoe location was considered ideal for an ash application study based on these characteristics, as well as its distance from roads and urban areas that eliminates potential road salt or other contamination effects.

2.2. Plot set-up and experimental design

Using a randomized plot design, eight 40 × 40 m plots with a 10 m buffer were established within a 10 ha perimeter 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 ash after application (Hannam et al. 2016). One treatment of 6 $Mg \cdot ha^{-1}$ NIWA was replicated four times and the remaining four plots were left as controls.

2.3. Field sampling and ash application

Baseline soil sampling was conducted at the end of September 2020. Three samples were collected beneath three sugar maples trees within each plot ($n = 72$); each tree was selected to be greater than 10 cm DBH. 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 (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. In press). Once collected and sieved, the ash was stored in a cool, dark environment in large, polyethylene containers prior to application in November 2020. Following site, ash-handling, and application approval 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 relatively even distribution. Ash was applied at a dose of 6 $\text{Mg}\cdot\text{ha}^{-1}$ (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 26 February 2021 and 25 February 2022, three sugar maple trees in each plot were tapped for sap collection ($n = 24$). 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 ($\pm SD = 8.3$) cm DBH and trees in the treatment plots averaged 49.8 ($\pm SD = 7.0$) 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 at a distance of 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 with each sampling to avoid contamination between sap samples for chemical analysis. Sap sampling seasons were from 20 March to 4 May 2021 and 18 March to 27 April 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 ($n = 4$).

2.4. Laboratory sampling analysis

2.4.1. Soil and ash analysis

Soil and ash samples were oven dried for 24 h 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, sodium (Na)), and total metals (aluminum (Al), arsenic (As), boron (B), Cd, Cu, iron (Fe), manganese (Mn), nickel (Ni), lead (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, and Zn).

Soil and ash pH were measured in a 0.01 M $CaCl_2$ slurry at a 1:5 ratio. The slurries were shaken for 2 h and then rested for 1 h 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 2 g L, FH, or 5 g of mineral soil or ash. Oven-dry samples were weighed into porcelain crucibles and ashed in a muffle furnace for 8 h at 450 °C. Ash samples were also analyzed for percent C and N content using a CNS combustion analyzer (Elementar vario EL III elementar analyzer, Elementar Americas Inc., Ronkonkoma, NY, US) and EnviroMAT SS-2 standards (SCP Science, Quebec, CA). In Bracebridge, the LFH and upper and lower mineral soil N content averaged 0.8%, 0.2%, and 0.1%, respectively (Deighton and 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 and FH material or 5 g of mineral soil were weighed into 50 mL Falcon Tubes before adding 25 mL of 1 M ammonium chloride (NH_4Cl) solution, shaking the solution for 2 h and resting for 1 h. The solution was then filtered through P8 Fast Flow Filter Paper where an additional 25 mL of 1 M 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 g into digiTUBEs (SCP Science, Quebec, CA) and digested on a hot plate at 100 °C for 8 h with 2% HNO_3 before digesting at room temperature for another 8 h. The samples were then filtered with P8 Fast Flow Filter Paper, diluted to 25 mL 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).

2.4.2. 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, and 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).

2.4.3. Sap analysis

Sap collected in the 50 mL Falcon Tubes was thawed and each sample was filtered through a 0.45 µm 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, and Zn) by ICP-OES.

2.5. Data analyses

2.5.1. 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 (2023*b*) portal.

2.5.2. Volume-weighted calculations

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

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

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.

2.5.3. 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):

$$(2) \qquad Flux/spile = \sum_{i=1}^{T/\delta t} (C_i V_i)$$

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.

2.5.4. Diagnosis and recommendation integrated system 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 $A/B \geq a/b$ (eq. 3):

$$(3) \qquad f\left(\frac{A}{B}\right) = \left(\frac{A/B}{a/b} - 1\right)\frac{1000}{\text{CV}}$$

or when $A/B \leq a/b$ (eq. 4):

$$(4) \qquad f\left(\frac{A}{B}\right) = \left(1 - \frac{a/b}{A/B}\right)\frac{1000}{\text{CV}}$$

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):

$$(5) \qquad A\,\text{index} = \frac{\left[f\left(\frac{A}{B}\right) + f\left(\frac{A}{C}\right) + f\left(\frac{A}{D}\right) + f\left(\frac{A}{E}\right)\right]}{z}$$

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.

2.6. 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 treatment effects and model

Table 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-industrial wood ash properties	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

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

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.

3. Results

3.1. Non-industrial wood ash chemistry

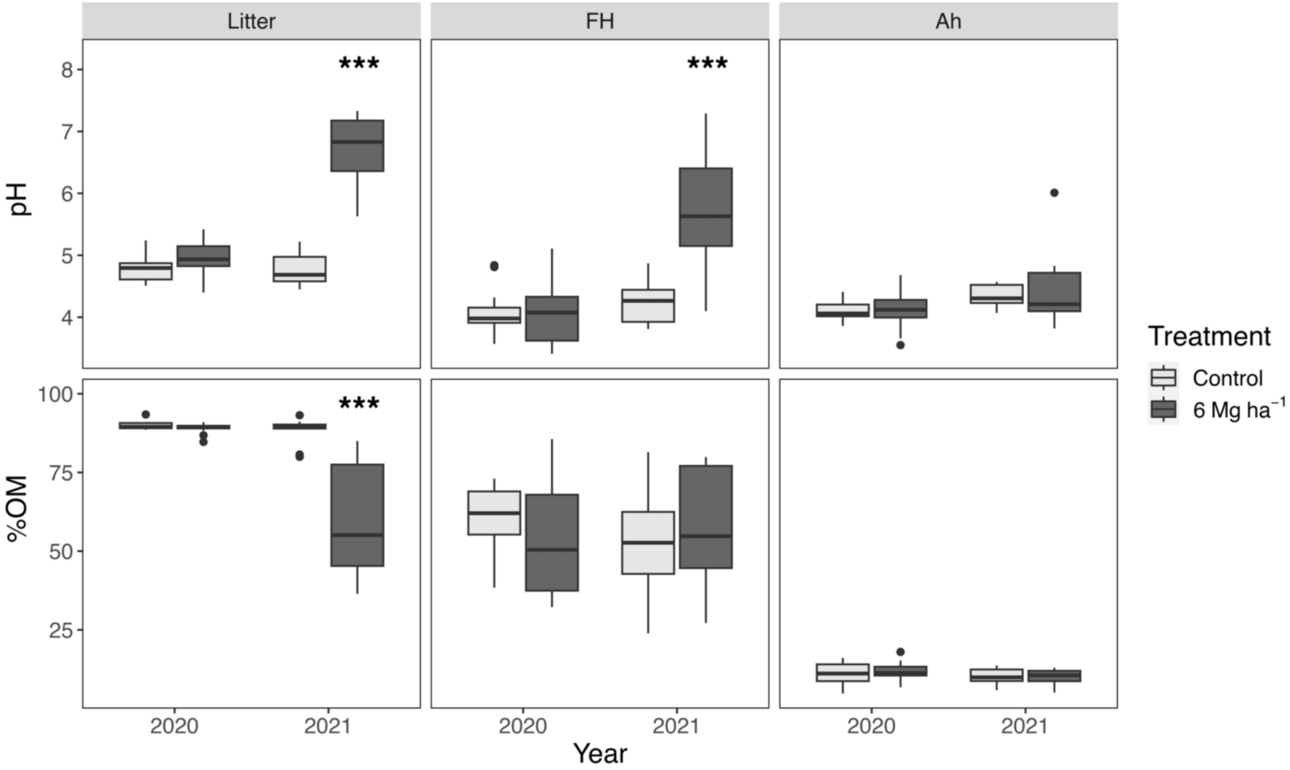
Non-industrial wood ash averaged 27% Ca and 9% K dry weight and an average pH of 13.0 (Table 1). Carbon and N content in NIWA were particularly low, with N concentrations low enough to suggest the addition of an N source to NIWA before application (Table 1). Mean concentrations of most metals were well below the unrestricted guidelines (CM1) for land application of non-agricultural source materials 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 1).

3.2. Soil chemistry

Prior to ash application, pH, organic matter, and average concentrations of nutrients and metals were similar between the control and treatment plots (Figs. 1–3). 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 (Fig. 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 (Fig. 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 (Fig. 2).

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 (Fig. 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 (Fig. 3; Appendix A, Table A1).

Fig. 1. Average pH_{CaCl2} and %organic matter (OM) in the L, FH, and upper mineral soil (Ah) sampled beneath sugar maple trees ($n = 24$) 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 ($*$, $p < 0.05$; $**$, $p < 0.01$; $***$, $p < 0.001$).



3.3. Climate data and sap yield and chemistry

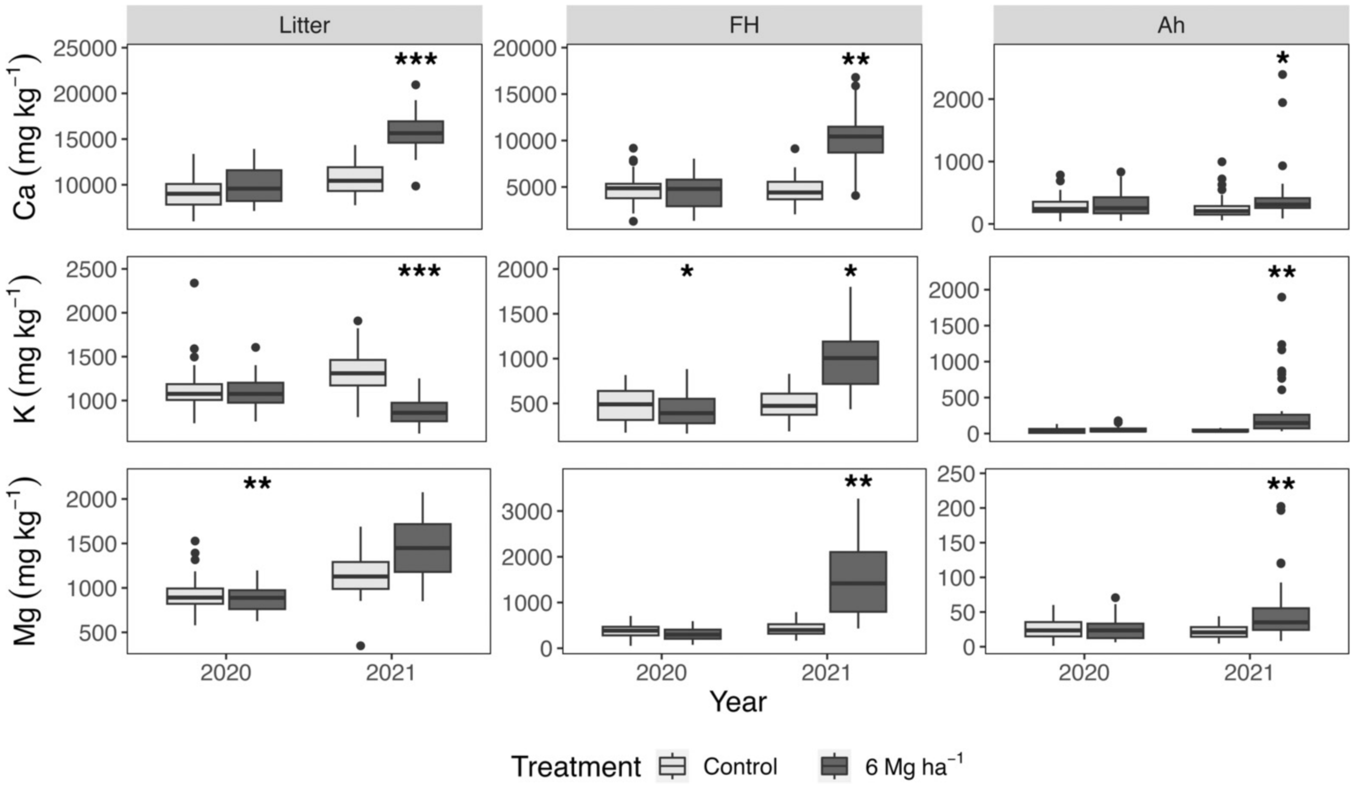
The 1 March–30 April mean air temperatures were 2.7 °C in 2021 and 0.5 °C in 2022 (Fig. 4). Total precipitation was similar in both years from 1 March to 30 April at 113 mm in 2021 and 128 mm in 2022 (Fig. 4). In 2021, however, sap yield in the treated plots (54 L) was twice that in the untreated plots (27 L), whereas in 2022, sap yield in both the treated (56 L) and untreated plots (52 L) was almost identical (Fig. 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 (Figs. 4 and 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). No significant treatment effect was found on sap pH or sweetness in either sampling year (Table 2).

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). 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). 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). 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 3). In 2021, seasonal fluxes of nutrients and metals in the ash-treated trees increased by between 2% and 226% (Table 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).

3.4. 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 (14 032 mg·kg^{-1} in the treated foliage compared with 7551 mg·kg^{-1} in the controls; Table 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 insignificant and remained either within or below crit-

Fig. 2. Average exchangeable cations in the L, FH, and upper mineral soil (Ah) sampled beneath sugar maple trees ($n = 24$) prior to (2020) and after (2021) ash application in the control and NIWA treated (6 $\mathrm{Mg{\cdot}ha^{-1}}$) plots. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (∗, $p < 0.05$; ∗∗, $p < 0.01$; ∗∗∗, $p < 0.001$).



ical foliar concentrations reported for sugar maples (Table 4; Kolb and McCormick 1993). Percent C and N were also similar across treatments (Table 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 5).

4. Discussion

4.1. 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 and 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 and 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 $\mathrm{mg{\cdot}kg^{-1}}$ and 500 $\mathrm{mg{\cdot}kg^{-1}}$, respectively, and are just below the concentrations reported here at 164 $\mathrm{mg{\cdot}kg^{-1}}$ Cu and 503 $\mathrm{mg{\cdot}kg^{-1}}$ Zn. Similarly, NIWA collected from Muskoka residents a few years before our ash was collected had concentrations of 101 $\mathrm{mg{\cdot}kg^{-1}}$ Cu and 501 $\mathrm{mg{\cdot}kg^{-1}}$ Zn, and although they fall slightly above CM1, both concentrations are substantially lower than the CM2 limits and are therefore unlikely to create toxic soil conditions (Azan et al. 2019). In agreement with Syeda et al. (In press), these results suggest that amalgamating wood ash samples from various species and sources results in metal concentrations that fall safely within provincial regulatory guidelines.

4.2. 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

Fig. 3. Average elemental metal concentrations in the L, FH, and upper mineral soil (Ah) sampled beneath sugar maple trees ($n = 24$) 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 ($*$, $p < 0.05$; $**$, $p < 0.01$; $***$, $p < 0.001$).

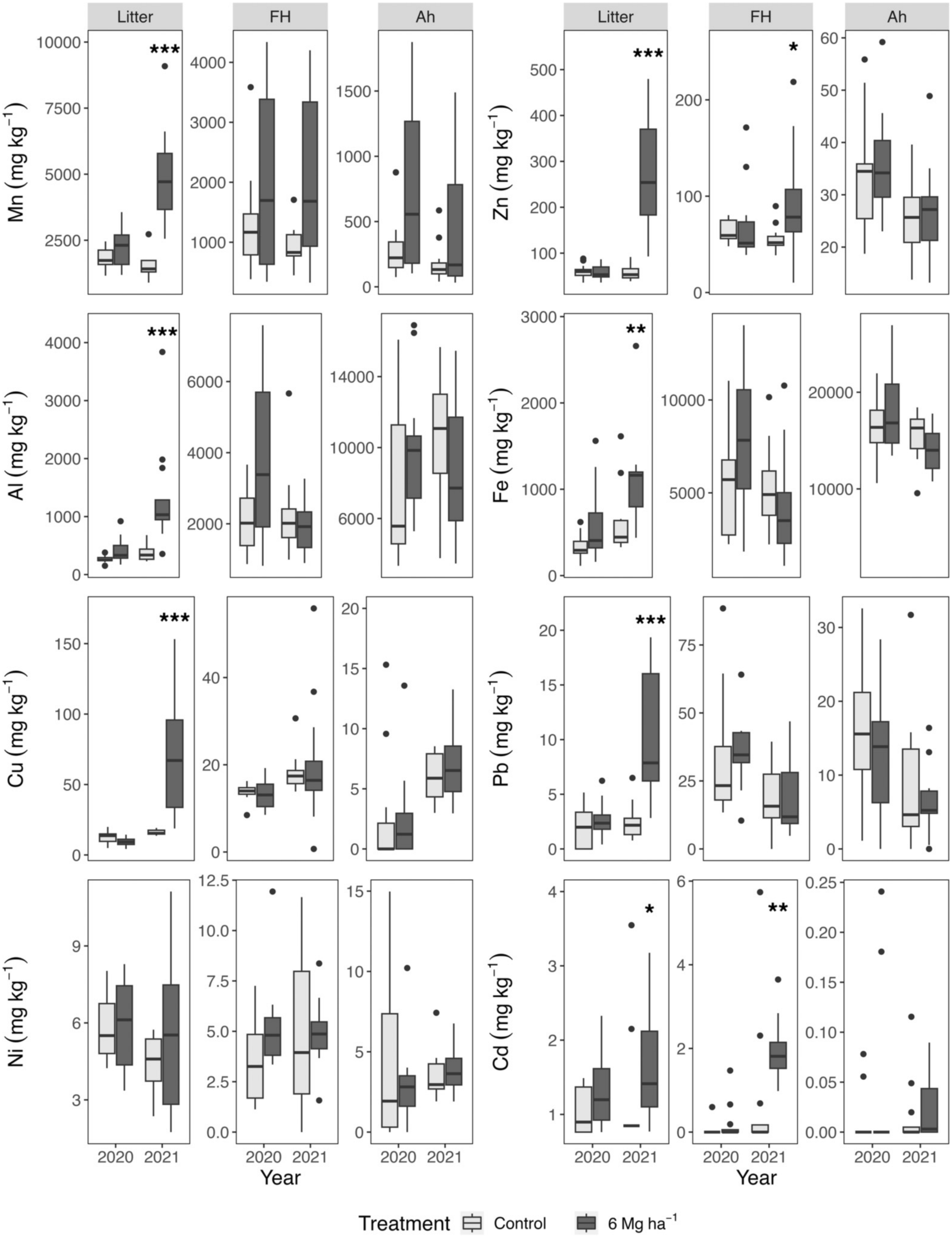


Fig. 4. Climograph for Muskoka, ON during maple sap sampling seasons in 2021 (March–May) and 2022 (March–April). Bars indicate average daily precipitation (mm) and lines indicate average daily temperature (°C) with minimum and maximum daily temperatures in grey shading. Breaks in the lines indicate missing data (ECCC 2023*b*).

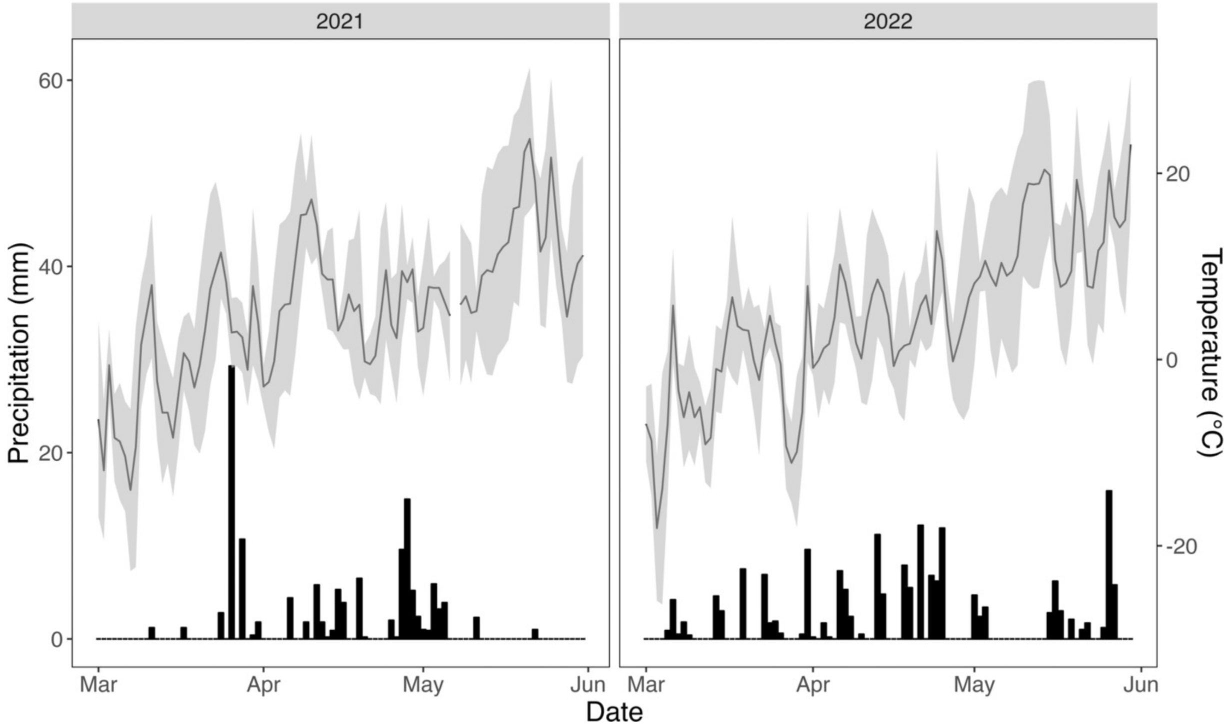


Fig. 5. Average sap yield (L ± SE) per spile since previous collection ($n = 24$) in the control and NIWA-treated (6 Mg·ha^{-1}) plots. Sampling was conducted from 20 March to 4 May in 2021 and 18 March to 27 April in 2022.

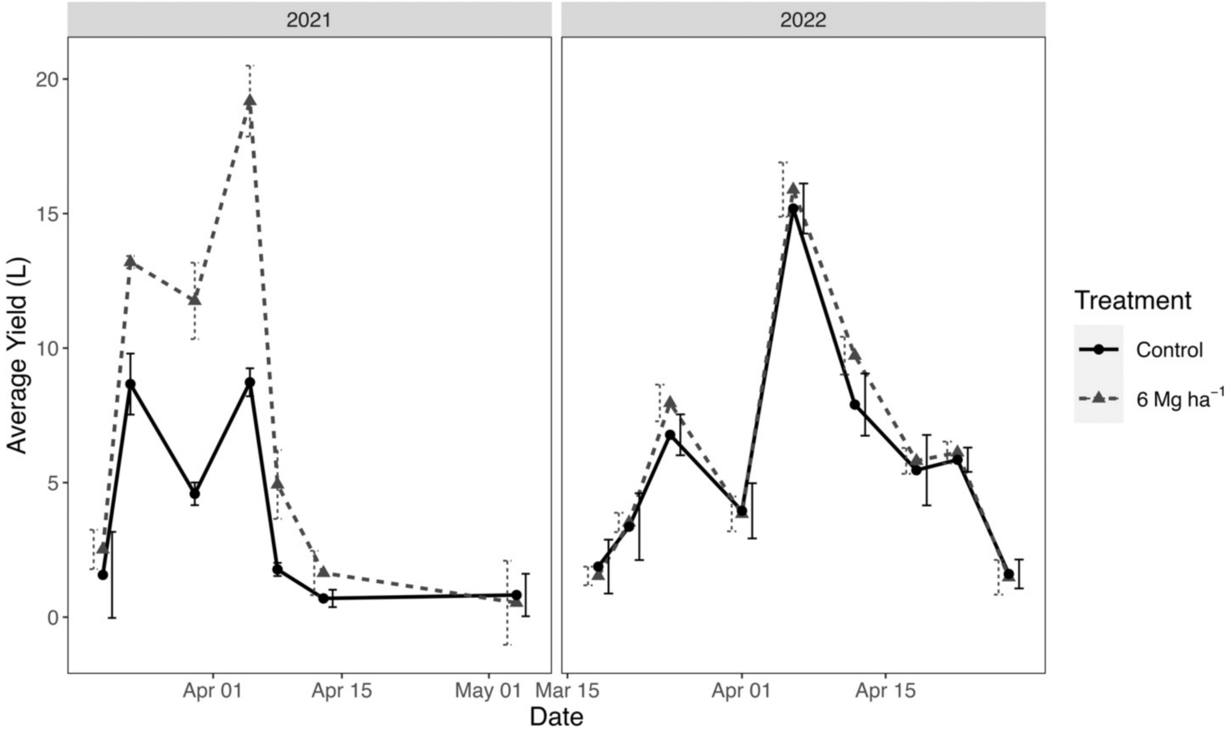


Table 2. Average (±SE) volume-weighted pH, °Brix, total yield, and elemental concentrations in sugar maple sap ($n = 24$) during the 2021 and 2022 sampling seasons in the control and NIWA-treated (6 Mg·ha^{-1}) plots.

	2021 Sap		2022 Sap		
	Control ($n = 12$)	Treatment ($n = 12$)	Control ($n = 12$)	Treatment ($n = 12$)	Range of concentrations in sugar maple sap[†]
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

Note: 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. As and B were below their detection limits and therefore removed.

[†] Review by Mohammed et al. 2022.

Table 3. Annual elemental flux (±SE) in sugar maple sap ($n = 24$) during the 2021 and 2022 sampling seasons.

	2021 Sap		2022 Sap	
Element	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)*

Note: Significant differences from control indicated by an asterisk (∗, $p < 0.05$; ∗∗, $p < 0.01$; ∗∗∗, $p < 0.001$) as determined by Wilcoxon rank-sum test. As and B were below their detection limits and therefore removed.

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 ($\text{pH} < 5.0$; Reid and 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. 2007; Reid and Watmough 2014; Deighton and Watmough 2020). The lack of a significant pH

Table 4. Average (±SE) foliar nutrient and metal concentrations in sugar maple trees ($n = 4$) collected 8 months after application of 6 Mg·ha^{-1} NIWA to experimental plots in Bracebridge, ON.

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)	

Note: As, B, and Pb were below their detection limits and therefore removed.
†Kolb and McCormick (1993).

Table 5. Average sugar maple foliar concentrations ($n = 4$) based on DRIS 8 months after application of 6 Mg·ha^{-1} NIWA to experimental plots in Bracebridge, ON with critical foliar concentrations and DRIS norms for reference.

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	

Note: Significant differences between treatment and control indicated by an asterisk (*, $p < 0.05$) as determined by a Wilcoxon rank-sum test.
†Kolb and McCormick (1993).
‡Lozano and Huynh (1989).

response in the mineral layer 10 months after ash application was also expected. A longer delay before increases are noticeable in the mineral layers is well documented (Lundström et al. 2003; Ozolinčius et al. 2007; 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 7 years after wood ash fertilization but did see an increase 16 years after application. However, pH increases may be observed as early as 5 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 1 year following ash application, but no effect was found on organic matter content 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 and 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 and Watmough 2014; Arseneau et al. 2021). While the increases observed here were the most significant in the litter (Ca) and FH (Ca and Mg) layer, Ca and Mg concentrations were also higher in the upper mineral horizon 1 year after application, similar to other studies (Saarsalmi et al. 2001, 2004; Ozolinčius et al. 2007; Augusto et al. 2008; Deighton and 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. 2007; Augusto et al. 2008) but have been observed to persist in deeper soil profiles over longer time periods (6–16 years; Bramryd and 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 and Watmough 2014). Other research, however, has found no significant differences between control and treated plots in the forest floor or upper mineral soil K 3 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 1 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.

4.3. Sap yield and chemistry

The optimal January–May mean temperature for peak sap volume is 1 °C (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 0 °C, whereas in 2022 it was −2 °C. Additionally, earlier sap collection corresponded with a higher March mean temperature (−1 °C in 2021 and −3 °C in 2022). Sap production is highly influenced by climate variability (Marvin and Erickson 1956; Kim and 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 in 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 2022). 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 CO_2 assimilation leading to better allocation of ATP necessary for phloem loading ultimately resulting in a higher osmotic pressure (Mengel and 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 and thus influence sap flow (Shabala and 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 and 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 and 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 14.5 °C in 2020 and 15.9 °C 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. 1995). In contrast, in a long-term study, liming was found to improve sap sweetness up to 20%, 18 years after application in Québec (Moore et al. 2020) and N additions have been shown to increase sap sweetness 2 years after application in New Hampshire (Wild and 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. 1995) 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, 18 years after liming, Moore et al. (2020) found significant increases in Ca (5 $Mg{\cdot}ha^{-1}$ treatment) and Mg (2 and 5 $Mg{\cdot}ha^{-1}$ 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 and 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.

4.4. Foliar chemistry

In general, sugar maple foliar base cation and metal concentrations were similar between the control and treated plots 1 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 and 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 and 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 and 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 to 10.4 $g{\cdot}kg^{-1}$ (Kolb and McCormick 1993) and while average concentrations in the control plots remained within this range at 7.6 $g{\cdot}kg^{-1}$, treated trees averaged higher at 14.0 $g{\cdot}kg^{-1}$. Reported DRIS indices in the control plots also show foliar nutritional balances within the optimal range for sugar maples (−20%–+20%; Lozano and 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 and 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 and Camberato 2019). In comparison, application of a similar dose of dolomitic lime (5 $Mg{\cdot}ha^{-1}$) showed improved DRIS indices in Ca and Mg slowly over time, but decreases in K and P in sugar maple 10 years after application (Moore and 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 3 years after application of 20 $Mg{\cdot}ha^{-1}$ 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.

5. Conclusions

This study evaluated the effect of applying 6 $Mg{\cdot}ha^{-1}$ 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. 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.

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Author information

Author ORCIDs

Shelby M. Conquer https://orcid.org/0009-0000-5801-8746

Author contributions

Conceptualization: SMC, NDY, SAW
Formal analysis: SMC
Funding acquisition: NDY, SAW
Investigation: SMC
Methodology: SMC
Resources: SAW
Supervision: SAW
Writing – riginal draft: SMC
Writing – review & editing: NDY, SAW

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References

[AAFC] Agriculture and Agri-Food Canada. 2022. Statistical overview of the Canadian maple industry, 2022. AAFC, Ottawa, ON. Available from https://agriculture.canada.ca/en/sector/horticulture/reports/statistical-overview-canadian-maple-industry-2022# [accessed 25 July 2023].

[AAFC] Soil Classification Working Group. 1998. The Canadian system of soil classification. 3rd ed. AAFC, Ottawa, ON. Publication 1646, 187.

[ECCC] Environment and Climate Change Canada. 2016. Canada–United States Air Quality Agreement: Progress Report 2016. International Joint Commission ECCC, Ottawa, ON. Available from https://www.canada.ca/content/dam/eccc/documents/pdf/canada-us-aqa-report/canada-us-aqa-report-2016-en.pdf [accessed 17 January 2023].

[ECCC] Environment and Climate Change Canada. 2023a. Canadian Climate Normals 1981–2010 Station Data. ECCC, Ottawa, Ontario, Canada. Accessed from https://climate.weather.gc.ca/climate_normals/results_1981_2010_e.html?searchType=stnName&txtStationName=muskoka&searchMethod=contains&txtCentralLatMin=0&txtCentralLatSec=0&txtCentralLongMin=0&txtCentralLongSec=0&stnID=4477&dispBack=1 [accessed 17 October 2020].

[ECCC] Environment and Climate Change Canada. 2023b. Daily Data Report for January 2021. ECCC, Ottawa, Ontario, Canada. Accessed from https://climate.weather.gc.ca/climate_data/daily_data_e.html?hlyRange=2009-08-27%7C2023-01-16&dlyRange=2009-07-06%7C2023-03-16&mlyRange=%7C&StationID=48368&Prov=ON&urlExtension=_e.html&searchType=stnName&optLimit=yearRange&StartYear=1840&EndYear=2023&selRowPerPage=25&Line=0&searchMethod=contains&Month=1&Day=16&txtStationName=muskoka&timeframe=2&Year=2021 [accessed 21 June 2022].

[FMW] Friends of the Muskoka Watershed. 2023. Available from https://fotmw.org/.

Arseneau, J., Bélanger, N., Ouimet, R., Royer-Tardif, S., Bilodeau-Gauthier, S., Gendreau-Berthiaume, B., and Rivest, D. 2021. Wood ash application in sugar maple stands rapidly improves nutritional status and growth at various developmental stages. For. Ecol. Manage. **489**: 119062. doi:10.1016/j.foreco.2021.119062.

Augusto, L., Bakker, M.R., and Meredieu, C. 2008. Wood ash applications to temperate forest ecosystems—potential benefits and drawbacks. Plant Soil, **306**(1–2): 181–198. doi:10.1007/s11104-008-9570-z.

Azan, S.S.E., Yan, N.D., Celis-Salgado, M.P., Arnott, S.E., Rusak, J.A., and Sutey, P. 2019. Could a residential wood ash recycling program be part of the solution to calcium decline in lakes and forests in Muskoka (Ontario, Canada)? Facets, **4**(1): 69–90. doi:10.1139/facets-2018-0026.

Bal, T.L., Storer, A.J., Jurgensen, M.F., Doskey, P.V., and Amacher, M.C. 2015. Nutrient stress predisposes and contributes to sugar maple dieback across its northern range: a review. For. Int. J. For. Res. **88**(1): 64–83. doi:10.1093/forestry/cpu051.

Ball, D.W. 2007. The chemical composition of maple syrup. J. Chem. Educ. **84**(10): 1647. doi:10.1021/ed084p1647.

Bramryd, T., and Fransman, B. 1995. Silvicultural use of wood ashes? Effects on the nutrient and heavy metal balance in a pine (*Pinus sylvestris*, L) forest soil. Water. Air. Soil Pollut. **85**(2): 1039–1044. doi:10.1007/BF00476967.

Casey, M.I. 2021. Paddling with the spirit: the story of Camp Big Canoe. Volumes Publishing, Waterloo, ON, CA. pp. 118–119.

Casson, N.J., Eimers, M.C., and Watmough, S.A. 2012. An assessment of the nutrient status of sugar maple in Ontario: indications of phosphorus limitation. Environ. Monit. Assess. **184**(10): 5917–5927. doi:10.1007/s10661-011-2390-2. PMID: 21994163.

Cirelli, D., Jagels, R., and Tyree, M.T. 2008. Toward an improved model of maple sap exudation: the location and role of osmotic barriers in sugar maple, butternut and white birch. Tree Physiol. **28**(8): 1145–1155. doi:10.1093/treephys/28.8.1145. PMID: 18519246.

Clément, A., Lagacé, L., and Panneton, B. 2010. Assessment of maple syrup physico-chemistry and typicity by means of fluorescence spectroscopy. J. Food Eng. **97**(1): 17–23. doi:10.1016/j.jfoodeng.2009.08.029.

Deighton, H.D., and Watmough, S.A. 2020. Effects of non-industrial wood ash (NIWA) applications on soil chemistry and sugar maple (*Acer saccharum*, Marsh.) seedling growth in an acidic sugar bush in central Ontario. Forests, **11**(6): 693. doi:10.3390/f11060693.

Deighton, H.D., Watmough, S.A., Basiliko, N., Hazlett, P.W., Reid, C.R., and Gorgolewski, A. 2021. Trace metal biogeochemical responses following wood ash addition in a northern hardwood forest. Can. J. For. Res. **51**(6): 817–833. doi:10.1139/cjfr-2020-0320.

Demeyer, A., Nkana, J.C.V., and Verloo, M.G. 2001. Characteristics of wood ash and influence on soil properties and nutrient uptake: an overview. Bioresour. Technol. **77**(3): 287–295. doi:10.1016/S0960-8524(00)00043-2. PMID: 11272014.

Driscoll, C.T., Lawrence, G.B., Bulger, A.J., Butler, T.J., Cronan, C.S., Eagar, C., et al. 2001. Acidic deposition in the northeastern United States: sources and inputs, ecosystem effects, and management strategies. BioScience, **51**(3): 180. doi:10.1641/0006-3568(2001)051[0180:ADITNU]2.0.CO;2.

Duchesne, L., Ouimet, R., and Houle, D. 2002. Basal area growth of sugar maple in relation to acid deposition, stand health, and soil nutrients. J. Environ. Qual. **31**(5): 1676–1683. doi:10.2134/jeq2002.1676. PMID: 12371186.

Elliott, A., Mahmood, T., and Kamal, A. 2022. Boiler ash utilization in the Canadian pulp and paper industry. J. Environ. Manage. **319**: 115728. doi:10.1016/j.jenvman.2022.115728. PMID: 35982553.

Fritze, H., Smolander, A., Levula, T., Kitunen, V., and Mälkönen, E. 1994. Wood-ash fertilization and fire treatments in a Scots pine forest stand: effects on the organic layer, microbial biomass, and microbial activity. Biol. Fertil. Soils, **17**(1): 57–63. doi:10.1007/BF00418673.

Fromard, L., Babin, V., Fleurat-Lessard, P., Fromont, J.C., Serrano, R., and Bonnemain, J.L. 1995. Control of vascular sap pH by the vessel-associated cells in woody species (physiological and immunological studies). Plant Physiol. **108**(3): 913–918. doi:10.1104/pp.108.3.913. PMID: 12228516.

Government of Ontario. 2002. O. Reg. 267/03, General under Nutrient and Management Act, 2002, S.O. 2002, c.4. Government of Ontario, Ottawa, ON. Available from https://www.ontario.ca/laws/regulation/030267 [accessed 17 October 2020].

Gregory, R.A., and Hawley, G.J. 1983. Sap extraction and measurement of soluble sap sugars in sugar maple. Can. J. For. Res. **13**(3): 400–404. doi:10.1139/x83-060.

Hannam, K.D., Deschamps, C., Kwiaton, M., Venier, L., and Hazlett, P.W. 2016. Regulations and guidelines for the use of wood ash as a soil amendment in Canadian forests. Natural Resources Canada, Canadian Forest Service, Great Lakes Forestry Centre, Sault Ste. Marie, ON. Information Report GLC-X-17.

Hannam, K.D., Venier, L., Allen, D., Deschamps, C., Hope, E., Jull, M., et al. 2018. Wood ash as a soil amendment in Canadian forests: What are the barriers to utilization? Can. J. For. Res. **48**(4): 442–450. doi:10.1139/cjfr-2017-0351.

Hansen, M., Kepfer-Rojas, S., Bjerager, P.E.R., Holm, P.E., Skov, S., and Ingerslev, M. 2018. Effects of ash application on nutrient and heavy metal fluxes in the soil and soil solution in a Norway spruce plantation in Denmark. For. Ecol. Manage. **424**: 494–504. doi:10.1016/j.foreco.2018.05.005.

Houle, D., Duchesne, L., Moore, J.D., Laflèche, M.R., and Ouimet, R. 2002. Soil and tree-ring chemistry response to liming in a sugar maple stand. J. Environ. Qual. **31**(6): 1993–2000. doi:10.2134/jeq2002.1993. PMID: 12469849.

Huggett, B.A., Schaberg, P.G., Hawley, G.J., and Eagar, C. 2007. Long-term calcium addition increases growth release, wound closure, and health of sugar maple (*Acer saccharum*) trees at the Hubbard Brook Experimental Forest. Can. J. For. Res. **37**(9): 1692–1700. doi:10.1139/X07-042.

Johnson, J., Graf Pannatier, E., Carnicelli, S., Cecchini, G., Clarke, N., Cools, N., et al. 2018. The response of soil solution chemistry in European forests to decreasing acid deposition. Glob. Change Biol. **24**(8): 3603–3619. doi:10.1111/gcb.14156.

Jones, A.R.C., and Alli, I. 1987. Sap yields, sugar content, and soluble carbohydrates of saps and syrups of some Canadian birch and maple species. Can. J. For. Res. **17**(3): 263–266. doi:10.1139/x87-044.

Juice, S.M., Fahey, T.J., Siccama, T.G., Driscoll, C.T., Denny, E.G., Eagar, C., et al. 2006. Response of sugar maple to calcium addition to northern hardwood forest. Ecology, **87**(5): 1267–1280. doi:10.1890/0012-9658(2006)87[1267:ROSMTC]2.0.CO;2. PMID: 16761605.

Kalra, Y.P., and Maynard, D.G. 1991. Methods manual for forest soil and plant analysis. Natural Resources Canada, Canadian Forest Service, Northern Forestry Centre, Edmonton, AB. Information Report NOR-X-319.

Kim, Y.T., and Leech, R.H. 1985. Effects of climatic conditions on sap flow in sugar maple. For. Chron. **61**(4): 303–307. doi:10.5558/tfc61303-4.

Kolb, T.E., and McCormick, L.H. 1993. Etiology of sugar maple decline in four Pennsylvania stands. Can. J. For. Res. **23**(11): 2395–2402. doi:10.1139/x93-296.

Lagacé, L., Leclerc, S., Charron, C., and Sadiki, M. 2015. Biochemical composition of maple sap and relationships among constituents. J. Food Compost. Anal. **41**: 129–136. doi:10.1016/j.jfca.2014.12.030.

Likens, G.E., Driscoll, C.T., and Buso, D.C. 1996. Long-term effects of acid rain: response and recovery of a forest ecosystem. Science, **272**(5259): 244–246. doi:10.1126/science.272.5259.244.

Long, R.P., Horsley, S.B., and Hall, T.J. 2011. Long-term impact of liming on growth and vigor of northern hardwoods. Can. J. For. Res. **41**(6): 1295–1307. doi:10.1139/x11-049.

Lozano, F.C., and Huynh, K.D. 1989. Foliar diagnosis of sugar maple decline by DRIS. Commun. Soil Sci. Plant Anal. **20**(17–18): 1895–1914. doi:10.1080/00103628909368191.

Lundström, U.S., Bain, D.C., and Taylor, A.F.S. 2003. Effects of acidification and its mitigation with lime and wood ash on forest soil processes: a review. Water Air Soil Pollut. **3**: 5–28. doi:10.1023/A:1024115111377.

Marvin, J.W., and Erickson, R.O. 1956. A statistical evaluation of some of the factors responsible for the flow of sap from the sugar maple. Plant Physiol. **31**(1): 57–61. doi:10.1104/pp.31.1.57. PMID: 16654837.

McLaughlin, S.B., and Wimmer, R. 1999. Tansley review no. 104: calcium physiology and terrestrial ecosystem processes. New Phytol. **142**(3): 373–417. doi:10.1046/j.1469-8137.1999.00420.x.

Mengel, K., and Haeder, H.E. 1977. Effect of potassium supply on the rate of phloem sap exudation and the composition of phloem sap of *Ricinus communis*. Plant Physiol. **59**(2): 282–284. doi:10.1104/pp.59.2.282. PMID: 16659834.

Moatar, F., and Meybeck, M. 2005. Compared performances of different algorithms for estimating annual nutrient loads discharged by the eutrophic River Loire. Hydrol. Process. **19**(2): 429–444. doi:10.1002/hyp.5541.

Mohammed, F., Sibley, P., Guillaume, D., and Abdulwali, N. 2022. Chemical composition and mineralogical residence of maple syrup: a comprehensive review. Food Chem. **374**: 131817. doi:10.1016/j.foodchem.2021.131817. PMID: 34906808.

Moore, J.D., and Ouimet, R. 2006. Ten-year effect of dolomitic lime on the nutrition, crown vigor, and growth of sugar maple. Can. J. For. Res. **36**(7): 1834–1841. doi:10.1139/x06-081.

Moore, J.D., and Ouimet, R. 2010. Effects of two Ca fertilizer types on sugar maple vitality. Can. J. For. Res. **40**(10): 1985–1992. doi:10.1139/X10-136.

Moore, J.D., and Ouimet, R. 2021. Liming still positively influences sugar maple nutrition, vigor and growth, 20 years after a single application. For. Ecol. Manage. **490**: 119103. doi:10.1016/j.foreco.2021.119103.

Moore, J.D., Duchesne, L., Ouimet, R., and Deschênes, M.L. 2020. Liming improves sap characteristics of sugar maple over the long term. For. Ecol. Manage. **464**: 118044. doi:10.1016/j.foreco.2020.118044.

Moore, J.D., Ouimet, R., and Duchesne, L. 2012. Soil and sugar maple response 15 years after dolomitic lime application. For. Ecol. Manage. **281**: 130–139. doi:10.1016/j.foreco.2012.06.026.

Moore, J.D., Ouimet, R., Long, R.P., and Bukaveckas, P.A. 2015. Ecological benefits and risks arising from liming sugar maple dominated forests in northeastern North America. Environ. Rev. **23**(1): 66–77. doi:10.1139/er-2014-0048.

Muhr, J., Messier, C., Delagrange, S., Trumbore, S., Xu, X., and Hartmann, H. 2016. How fresh is maple syrup? Sugar maple trees mobilize carbon stored several years previously during early springtime sap-ascent. New Phytol. **209**(4): 1410–1416. doi:10.1111/nph.13782. PMID: 26639654.

Noland, T.L., McVey, G., and Chapeskie, D. 2006. Ice storm and fertilization effects on root starch, sap productivity and sweetness, diameter growth, and tap hole closure in sugar maple stands of eastern Ontario. Ministry of Natural Resources, Ontario Forest Research Institute. Forest Research Note No. 68. Available from https://www.researchgate.net/profile/Thomas-Noland/publication/253186119_Ice_storm_and_fertilization_effects_on_root_starch_sap_productivity_and_sweetness_diameter_growth_and_tap_hole_closure_in_sugar_maple_stands_of_eastern_Ontario/links/0deec52a73aa226216000000/Ice-storm-and-fertilization-effects-on-root-starch-sap

-productivity-and-sweetness-diameter-growth-and-tap-hole-closure-in-sugar-maple-stands-of-eastern-Ontario.pdf [accessed 27 January 2023].

Ohno, T. 1992. Neutralization of soil acidity and release of phosphorus and potassium by wood ash. J. Environ. Qual. **21**(3): 433–438. doi:10.2134/jeq1992.00472425002100030022x.

Ozolinčius, R., Varnagirytė-Kabašinskienė, I., Armolaitis, K., Gaitnieks, T., Buožytė, R., Raguotis, A., et al. 2007. Short term effects of compensatory wood ash fertilization on soil, ground vegetation and tree foliage in Scots pine stands. Balt. For. **13**(2): 158–168.

Penn, C., and Camberato, J. 2019. A critical review on soil chemical processes that control how soil pH affects phosphorus availability to plants. Agriculture, **9**(6): 120. doi:10.3390/agriculture9060120.

Perkins, T.D., Heiligmann, R.B., Koelling, M.R., and van den Berg, A.K. 2022. North American Maple Syrup Producers Manual. 3rd ed. The University of Vermont, North American Maple Syrup Council, Proctor Maple Research Center. Available from http://157.245.92.171/wp-content/uploads/NAMSPM3sm.pdf [accessed 25 July 2023].

Pitman, R.M. 2006. Wood ash use in forestry—a review of the environmental impacts. Forestry, **79**(5): 563–588. doi:10.1093/forestry/cpl041.

Pramsohler, M., Lichtenberger, E., and Neuner, G. 2022. Seasonal xylem sap acidification is governed by tree phenology, temperature and elevation of growing site. Plants, **11**(15): 2058. doi:10.3390/plants11152058. PMID: 35956536.

R Core Team. 2022. R: a language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. Available from https://www.R-project.org/ [accessed 15 September 2020].

Rapp, J.M., Lutz, D.A., Huish, R.D., Dufour, B., Ahmed, S., Morelli, T.L., et al. 2019. Finding the sweet spot: shifting optimal climate for maple syrup production in North America. For. Ecol. Manage. **448**: 187–197. doi:10.1016/j.foreco.2019.05.045.

Reid, C., and Watmough, S.A. 2014. Evaluating the effects of liming and wood-ash treatment on forest ecosystems through systematic meta-analysis. Can. J. For. Res. **44**(8): 867–885. doi:10.1139/cjfr-2013-0488.

Reid, C., and Watmough, S.A. 2016. Spatial patterns, trends, and the potential long-term impacts of tree harvesting on lake calcium levels in the Muskoka River Watershed, Ontario, Canada. Can. J. Fish. Aquat. Sci. **73**(3): 382–393. doi:10.1139/cjfas-2015-0231.

Saarsalmi, A., Mälkönen, E., and Kukkola, M. 2004. Effect of wood ash fertilization on soil chemical properties and stand nutrient status and growth of some coniferous stands in Finland. Scand. J. For. Res. **19**(3): 217–233. doi:10.1080/02827580410024124.

Saarsalmi, A., Mälkönen, E., and Piirainen, S. 2001. Effects of wood ash fertilization on forest soil chemical properties. Silva Fenn. **35**(3): 355–368. doi:10.14214/sf.590.

Schaberg, P.G., Tilley, J.W., Hawley, G.J., DeHayes, D.H., and Bailey, S.W. 2006. Associations of calcium and aluminum with the growth and health of sugar maple trees in Vermont. For. Ecol. Manage. **223**(1–3): 159–169. doi:10.1016/j.foreco.2005.10.067.

Schell, J. 1997. Interdependence of pH, malate concentration, and calcium and magnesium concentrations in the xylem sap of beech roots. Tree Physiol. **17**(7): 479–483. doi:10.1093/treephys/17.7.479. PMID: 14759841.

Schill, V., Hartung, W., Orthen, B., and Weisenseel, M.H. 1996. The xylem sap of maple (*Acer platanoides*) trees—Sap obtained by a novel method shows changes with season and height. J. Exp. Bot. **47**(1): 123–133. doi:10.1093/jxb/47.1.123.

Shabala, S., and Shabala, L. 2011. Ion transport and osmotic adjustment in plants and bacteria. Biomol. Concepts, **2**(5):407–419. doi:10.1515/BMC.2011.032.

Siddique, R. 2012. Utilization of wood ash in concrete manufacturing. Resour. Conserv. Recycl. **67**: 27–33. doi:10.1016/j.resconrec.2012.07.004.

St. Clair, S.B., Sharpe, W.E., and Lynch, J.P. 2008. Key interactions between nutrient limitation and climatic factors in temperate forests: a synthesis of the sugar maple literature. Can. J. For. Res. **38**(3): 401–414. doi:10.1139/X07-161.

Sullivan, T.J., Lawrence, G.B., Bailey, S.W., McDonnell, T.C., Beier, C.M., Weathers, K.C., et al. 2013. Effects of acidic deposition and soil acidification on sugar maple trees in the Adirondack Mountains, New York. Environ. Sci. Technol. **47**(22): 12687–12694. doi:10.1021/es401864w. PMID: 24102084.

Syeda, B.S., Watmough, S.A., and Yan, N.D. In press. Non-industrial wood ash chemistry. ON, Canada.

Tominaga, K., Watmough, S.A., and Aherne, J. 2008. Predicting tree survival in Ontario sugar maple (*Acer saccharum*) forests based on crown condition. Can. J. For. Res. **38**(7): 1730–1741. doi:10.1139/X08-021.

Tyree, M.T. 1983. Maple sap uptake, exudation, and pressure changes correlated with freezing exotherms and thawing endotherms. Plant Physiol. **73**(2): 277–285. doi:10.1104/pp.73.2.277. PMID: 16663208.

Violante, A., Cozzolino, V., Perelomov, L., Caporale, A.G., and Pigna, M. 2010. Mobility and bioavailability of heavy metals and metalloids in soil environments. J. Soil Sci. Plant Nutr. **10**(3): 268–292. doi:10.4067/S0718-95162010000100005.

Watmough, S.A., Eimers, C., and Baker, S. 2016. Impediments to recovery from acid deposition. Atmos. Environ. **146**: 15–27. doi:10.1016/j.atmosenv.2016.03.021.

Wild, A.D., and Yanai, R.D. 2015. Soil nutrients affect sweetness of sugar maple sap. For. Ecol. Manage. **341**: 30–36. doi:10.1016/j.foreco.2014.12.022.

Wilmot, T.R., Brett, P.W., and Tyree, M.T. 1995. Vigor and nutrition vs. sap sugar concentration in sugar maples. North. J. Appl. For. **12**(4): 156–162. doi:10.1093/njaf/12.4.156.

Wilmot, T.R., Ellsworth, D.S., and Tyree, M.T. 1996. Base cation fertilization and liming effects on nutrition and growth of Vermont sugar maple stands. For. Ecol. Manage. **84**(1–3): 123–134. doi:10.1016/0378-1127(96)03743-7.

Wong, B.L., Baggett, K.L., and Rye, A.H. 2003. Seasonal patterns of reserve and soluble carbohydrates in mature sugar maple (*Acer saccharum*). Can. J. Bot. **81**(8): 780–788. doi:10.1139/b03-079.

Xie, K., Cakmak, I., Wang, S., Zhang, F., and Guo, S. 2021. Synergistic and antagonistic interactions between potassium and magnesium in higher plants. Crop J. **9**(2): 249–256. doi:10.1016/j.cj.2020.10.005.

Yuan, T., Li, L., Zhang, Y., and Seeram, N.P. 2013. Pasteurized and sterilized maple sap as functional beverages: chemical composition and antioxidant activities. J. Funct. Foods, **5**(4): 1582–1590. doi:10.1016/j.jff.2013.06.009.

Appendix A

Table A1. Maximum allowable soil metal concentrations for NASM application.

Metals	Maximum concentration ($mg\cdot kg^{-1}$ soil, dw)[†]
As	14
Cd	1.6
Cu	100
Ni	32
Pb	60
Zn	220

Note: dw, dry weight by mass.
[†] Government of Ontario (2002)