REPORT

EFFECTS OF NON-INDUSTRIAL WOOD ASH ON SUGAR MAPLE SAP AND SOIL CHEMISTRY IN MUSKOKA, ONTARIO

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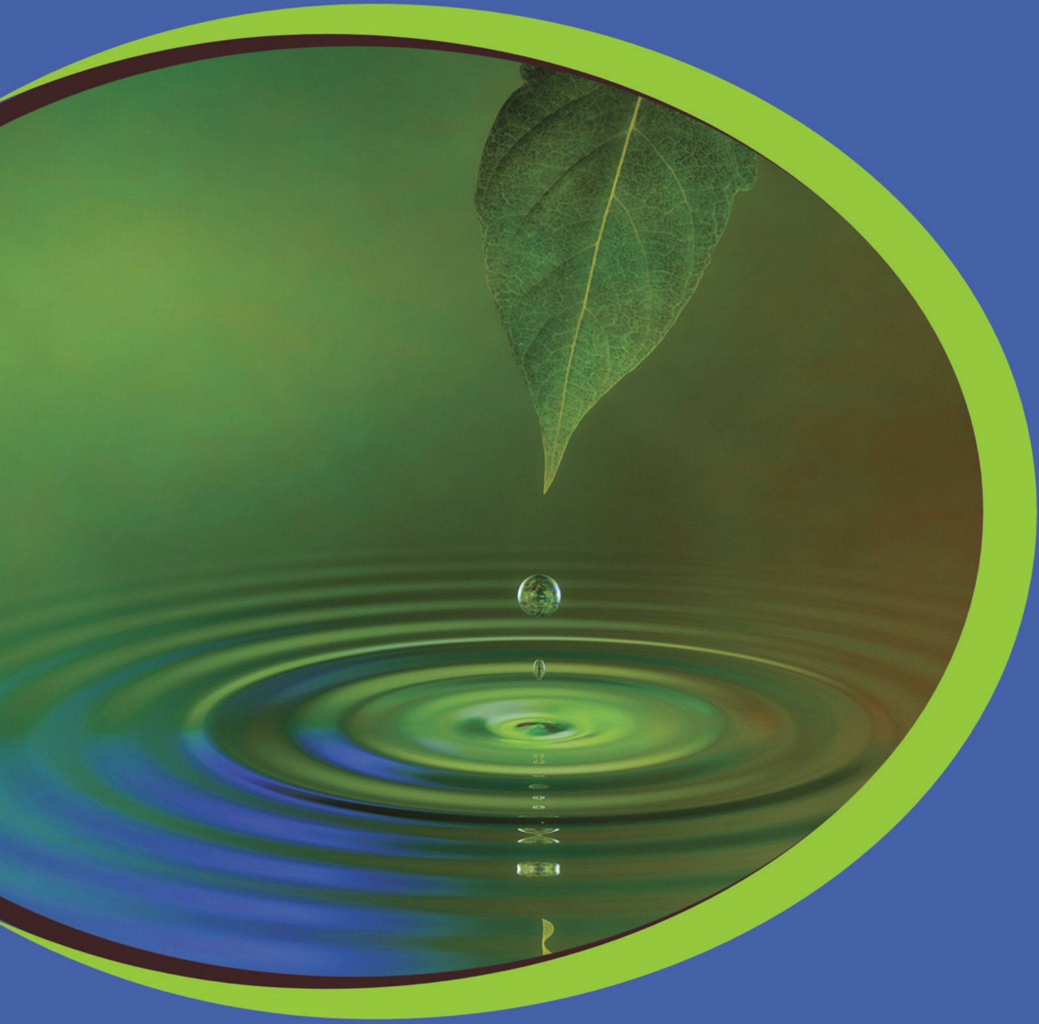

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Friends of the Muskoka Watershed

Effects of non-industrial wood ash on sugar maple sap and soil chemistry in Muskoka, Ontario

A report to the Friends of the Muskoka Watershed

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Abstract

Non-industrial wood ash (NIWA) was collected and applied to experimental plots in a sugar maple (*Acer saccharum* Marsh.) stand in Muskoka, Ontario at a rate of 6 Mg ha^{-1} to mitigate the legacy effects of acid deposition and understand its influence on sugar maple sap over two tapping seasons. The NIWA's main constituent was calcium (Ca) a approximately 30% by weight. Metal concentrations of the ash were well below provincial regulatory limits. One year following NIWA application soil pH and base cations (Ca, K, Mg) increased in the surface soil horizons. Metal concentrations increased significantly in the litter layer but only Cd and Zn were elevated in the FH horizon and no significant changes were observed in the mineral layer. Sap yield were almost twice as high in the treatment plots compared with non-ashed controls in the first year following application, but no significant differences were found the following year and sap sugar content remained similar in both years. Changes in sap nutrient and metal concentrations were generally small and inconsistent between years. Foliar chemistry also remained largely unchanged when measured one year following NIWA application except for K that almost doubled in the treated plots. Although NIWA application may influence sap yield in the short-term, NIWA is unlikely to significantly alter the chemistry of sugar maple sap indicating it as a viable soil amendment that may be used to enhance soil fertility in sugar bushes.

Introduction

Decades of atmospheric sulfur (S) and nitrogen (N) emissions have threatened the health of forest ecosystems in eastern North America and Europe (DeHayes et al., 1999; Driscoll et al., 2001). Anthropogenic increases in sulfate (SO_4) and nitrate (NO_3) deposition have led to leaching of base cations, particularly calcium (Ca, Johnson et al., 1985; Likens et al., 1996) and changes in soil chemistry that increase the availability of toxic metals such as aluminum (Al) (Al, Cronan & Schofield, 1990; de Vries et al., 1995). Despite reductions in acid deposition since the Canada-United States Air Quality Agreement in 1991 (Driscoll et al., 2001) (Environment and Climate Change Canada 2016) subsequent depletion of exchangeable base cation pools and slow weathering rates may cause a long-term lag before substantial natural chemical recovery is seen (Hedin et al., 1994; J. Johnson et al., 2018; Likens et al., 1996; Watmough et al., 2016; Watmough & Dillon, 2003). Sugar maples (*Acer saccharum* Marshall) are particularly sensitive to low concentrations of soil Ca (Schaberg et al., 2006; St.Clair et al., 2008) and have exhibited decreased vigor, canopy condition, growth rates, and recruitment on these nutrient depleted soils (Driscoll et al., 2001; Duchesne et al., 2002; McLaughlin & Wimmer, 1999; Sullivan et al., 2013).

Soil amendment studies have shown that the addition of lime, wood ash, or wollastonite can have beneficial effects on sugar maple. Increases in soil pH and concentrations of essential nutrients 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 (Long et al., 2011; Moore et al., 2012; Moore & Ouimet, 2021). Amendment studies have reported increased vigor, crown health, growth rate, recruitment of seedlings, base cation concentrations, and wound closure on sugar maple trees (Arseneau et al., 2021; Deighton & Watmough, 2020; Houle et al., 2002; Huggett et al., 2007; Juice et al., 2006; Long et al., 2011; Moore & Ouimet, 2010; Wilmot et al., 1996).

The impact of soil amendments on sugar maple sap production have not been widely studied despite its substantial economic value in Canada and the northeastern United States. Canada is the largest exporter of maple products in the world with exports in 2021 valued at $591 million (Agriculture and Agri-Food Canada 2022). However, various combinations of fertilization additions have produced mixed results on sap yield and sweetness. Wilmot et al. (1995) 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 been shown to increase 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).

Wood ash has been used widely in Europe but has been used sparingly in Canada due to regulation limitations. In Ontario, wood ash is classified as a non-agricultural source material (NASM). Non-agricultural source materials are regulated based on metal concentrations to ensure soils do not exceed critical levels. Trace metal concentrations are split into two categories: unrestricted (CM1) and restricted (CM2) (Government of Ontario 2002). As metal concentrations increase the restrictions on NASM application (e.g. proximity to any water source) also increase; if any metal concentrations are above CM2 guidelines, the material cannot be applied as a NASM (Hannam et al., 2016).

In response to the acidification of central Ontario a community-based effort has been launched to understand the potential benefits of wood ash application. Non-industrial wood ash

(NIWA) – ash produced from wood stoves – is donated by volunteers of the Muskoka region for use in a locally sourced recycling program; each year Ontario alone produces approximately 18,000 tonnes of NIWA that could be diverted from landfills (Azan et al., 2019). The chemical properties of wood ash are variable and depend on the soil conditions of the harvesting environment, tree species and tissue types, and combustion and storage conditions (Pitman, 2006). 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 amendment 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). Research conducted on the composition of mixed-hardwood NIWA averaged 30% Ca and all metal concentrations below CM1, except copper (Cu) and Zn which were marginally above CM1 but well below CM2 (Azan et al., 2019). Additionally, NIWA samples produced from sugar maple, white pine (*Pinus strobus* L.), and yellow birch (*Betula alleghaniensis* Britt.) each contained metal concentrations below CM1 guidelines, except for Cd, Cu, Zn, and selenium in yellow birch which were above CM1 but below CM2 (Deighton & Watmough, 2020).

The objective of this work is to quantify the effect of NIWA on the soils, foliage and sap yield and sugar content of sugar maple trees over the short-term (1-2 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 corresponding increase in the sap. It was predicted that there would be a limited increase, if any, in metal concentrations in the soils, foliage, and sap. Lastly, it was predicted that there would be no immediate effect on sap yield or sweetness.

Methodologies

Study Area

The study site is located on a mixed-wood Great Lakes-St. Lawrence ecozone east of the town of Bracebridge, Ontario (45°03'45.27" N, 79°08'43.62" W). The soil is acidic (approximately 4.1 pH) with an elevation approximately 282 m above sea level (Government of Canada, 2023). The average annual temperature is 5.2°C and the average annual precipitation is 1105 mm measured over a 30-year period (Government of Canada, 2023). The soils are shallow and typically poorly developed podzols and brunisols overlaying Precambrian gneiss and other metamorphic rock (Soil Classification Working Group, 1998). The forest is uneven-aged and dominated by sugar maple trees. It is located on a 240-acre plot owned by Camp Big Canoe that has been preserved since 1968. This location is ideal for ash application based on these characteristics, as well as its distance from major roads and urban areas that eliminates potential road salt or other contamination effects.

Experimental Design and Sampling

Plot Setup

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 site (Figure 1) 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 NIWA addition was applied to four plots (1, 3, 4, and 7) and the remaining four plots were left as controls (2, 5, 6, 8; Figure 1).

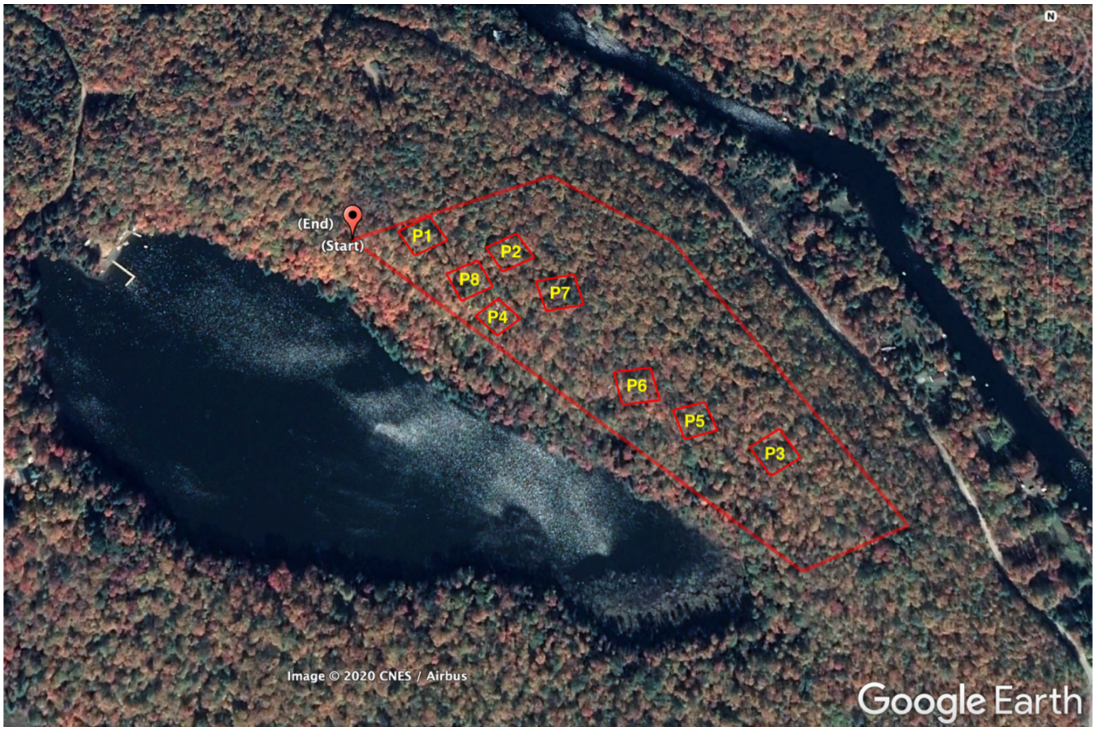


Figure 1. The 10 ha site perimeter with eight 40×40 m plots at Camp Big Canoe in Bracebridge, Ontario. Plots 1, 3, 4, and 7 received 6 t ha^{-1} NIWA treatment and plots 2, 5, 6, and 8 serve as controls. Copyright Google Earth.

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 = 72). Soil 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 horizon.

Non-industrial wood ash samples were generated by volunteer Muskoka residents and collected by the Friends of the Muskoka Watershed. At the time of collection ash was sieved (<2 mm) to remove charcoal and large debris. The ash was stored in a cool, dark environment in large polyethylene containers prior to application in November 2020. Ash was weighed and transported for each treatment plot in 10 kg buckets and then applied by hand to ensure even distribution. Ash was applied at a rate of 6 Mg ha^{-1} to the four treatment plots. During application six ash sub samples were taken randomly from each plot and kept separately until analysis (*n* = 24).

In February 2021 three sugar maple trees in each plot were tapped for sap collection (*n* = 24). Trees selected for tapping were greater than 25cm DBH and had no obvious wounds. Trees in the control plots averaged 40.7 cm DBH and trees in the treatment plots averaged 49.8 cm DBH. Holes were drilled at waist height on the south side of the tree using a 19/64 drill bit. A 5/16 plastic spile was then inserted and 5/16 tubing was attached feeding into a 10kg bucket with a lid. The buckets were then fastened around the tree using metal wiring and S-hooks. Plastic bags were placed into the buckets and were replaced with each sampling in order to avoid chemical contamination between sap samples (Figure 2). Sap sampling seasons were from March 20-May 4 in 2021 and March 18-April 27 in 2022. Sap yield was collected at least once per week during the sampling season but was occasionally collected more frequently when yield was high to avoid the buckets overflowing. Yield data was collected by measuring all of the sap into a large graduated cylinder. Once collected the sap was removed and a new bag was placed in the bucket. On days when sap samples were collected for analysis these samples were taken first in 50mL Falcon tubes and stored in a freezer 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 mature sugar maple tree where the soil samples were collected using extendable pole pruners from branches receiving direct sunlight and placed in separate plastic bags.

Figure 2. Experimental set-up of sap collection. Plastic tubing led from a spile into a 10 kg bucket where sap was collected in a bag to ensure independent analysis of samples. Bags were changed between collections.

Laboratory Sampling Analyses

Soil and Ash Analyses

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

Soil and ash pH was measured using 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 using

LOI. Two grams from the LFH layers and five grams from the mineral layer and the ash were weighed into crucibles and dried in an oven at 110°C to remove excess moisture. Once dried the crucibles were weighed and then transferred into a muffle furnace at 450°C for 8 hours. The equation ((wet mass – dry mass) / dry mass) x 100 was used to calculate percent moisture content. Ash samples were also analyzed for percent C and N content using a CNS combustion analyzer and NIST 1515-SRM apple leaf standards.

Soil exchangeable cations were analyzed using inductively coupled plasma optical emission spectroscopy Optima 7000DV (ICP-OES; PerkinElmer, Waltham, MA, US). One gram of the LFH horizonss and five grams of the mineral soil were weighed into 50mL Falcon Tubes before adding 25mL ammonium chloride (NH_4Cl), 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 25mL NH_4Cl was used. Samples were then diluted and refrigerated prior to analysis with an ICP-OES. Total metal concentrations for the soils and nutrient and metal concentrations for the 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 for 8 hours with 2% HNO_3 before digesting at room temperature for another 8 hours. The samples were then filtered with P8 Fast Flow Filter Paper, diluted to 25mL with B-pure water and then refrigerator prior to further dilution for analysis. Soil standards (EnviroMat SS-1) and blanks were used periodically to ensure accuracy and a standard curve was created for each analysis with the ICP-OES with standards from SCP Science (SCP Science, Quebec, CA).

Foliage Analyses

Foliar samples were collected from each of the mature sugar maple trees where soil samples were taken and combined per plot (n = 8). Each sample was dried in an oven for 24 hours at 110°C and then ground using a coffee grinder. Foliage samples were measured for CN content and nutrients and metals (Ca, Mg, K, P, Na, Al, As, B, Cd, Cu, Fe, Mn, Ni, Pb, Zn).

Carbon and N content were determined using a CN combustion analyzer with NIST 1515-SRM apple leaf standards throughout (SCP Science, Quebec, CA). Total nutrient and metal concentrations were determined using an acid digest. Samples were weighed to 0.2 grams into digiTUBEs (SCP Science, Quebec, CA) and digested on a hot plate for 8 hours with 2.5mL HNO_3 followed by digestion at room temperature for an additional 8 hours. Samples were filtered using P8 Fast Flow Filter Paper, diluted to 25mL with B-pure water, and refrigerated prior to analysis. NIST 1515-SRM apple leaf standards were used periodically to test for precision and a standard curve was created during ICP-OES analysis with standards from SCP Science (SCP Science, Quebec, CA).

Sap analyses

Sap collected for laboratory analysis was stored frozen in 50mL Falcon Tubes. Each sample was filtered through a 0.45µm nylon filter. Pure maple sap was analyzed for pH, total soluble solids (°Brix), and nutrient and metal content. Sap pH was measured using an OAKTON pH 510 series multimeter (Oakton Instruments, Vernon Hills, Il) and for °Brix using a brix refractometer. Nutrient and metal concentrations were determined using an ICP-OES. After filtration the sap was acidified to 2% HNO_3. Blanks were inserted periodically, and a standard curve was created during ICP-OES analysis using standards from SCP science (SCP Science, Quebec, CA).

Statistical Analyses

Statistical analyses were conducted using RStudio Version 1.3.1093 (RStudio Team 2020). Soil, sap, and foliage control and treatment data were compared using a Wilcoxon rank-sum test. A one-way ANCOVA was used to test for differences in average yield while accounting for tree DBH. Significance was determined at $p < 0.05$.

Results

Non-Industrial Wood Ash Chemistry

Non-industrial wood ash averaged 27% Ca and 9% K by weight. 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 1). Mean concentrations of most metals were well below the unrestricted guidelines (CM1) for land application of non-agricultural source materials (NASM; Nutrient and Management Act, 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).

Table 1. Average pH_{CaCl2}, organic matter, and nutrient and metal concentrations of non-industrial wood ash (means ± SE) collected from 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	NASM Limits†	
		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)	267 (3.0)		
K (g/kg)	94.4 (2.9)		
Mg (g/kg)	19.4 (0.3)		
Mn (g/kg)	8.8 (0.3)		
P (g/kg)	7.5 (0.1)		
Al (g/kg)	3.8 (0.3)		
Fe (g/kg)	2.2 (0.2)		
Zn (mg/kg)	503 (18.5)	500	4200
Cu (mg/kg)	164 (9.4)	100	1700
Cd (mg/kg)	2.9 (0.2)	3	34
As (mg/kg)	9.9 (2.2)	13	170
Ni (mg/kg)	9.6 (0.6)	62	420
Pb (mg/kg)	48.2 (16.1)	150	1100
B (mg/kg)	265 (5.3)		

†Nutrient and Management Act, 2002

Sulfur concentrations were undetectable and therefore removed.

Soil Chemistry

Prior to ash application pH, organic matter, and average concentrations of nutrients were all similar between the control and treatment plots (Table 2). One year following ash application significant increases were observed in the pH of the LFH horizons in the ash treated plots, while a significant decrease in percent organic matter was only observed in the litter horizon of the treatment plots (Table 2). Significant increases were observed in Ca in the LFH and upper mineral horizons where concentrations in the treatment plots were almost double that of the controls (Table 2). Potassium and Mg exhibited similar behaviour, except that significant increases were only observed in the FH and upper mineral horizons (Table 2).

Average metal concentrations were also similar in the organic and mineral horizons of both the control and treatment plots prior to application (Table 3). Following the application of NIWA the average concentrations of almost all metals increased in the organic (L and to a lesser extent, FH) horizons, with Al, Mn, Cu, Pb, and Zn showing the largest increases (Table 3). Further, concentrations of metals in the upper mineral soils were unaffected by ash application, and all regulated metals remained lower than the maximum allowable concentrations in soils receiving NASM (Table 3; Appendix A-1).

Table 2. Average pH_{CaCl2}, percent organic matter, and exchangeable cations (± SE) in LFH and mineral soil sampled beneath mature sugar maple trees ($n = 24$) 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 (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

Element	**Treatment**	**Litter**		**FH**		**Min I (Ah)**	
		Control	*6t ha-1*	*Control*	*6t ha-1*	*Control*	*6t ha-1*
pH	*Pre-Ash*	4.7 (0.1)	4.9 (0.1)	4.0 (0.1)	3.8 (0.2)	4.1 (0.0)	4.0 (0.1)
	Post-Ash	4.7 (0.1)	6.3 (0.2)***	4.2 (0.1)	4.9 (0.3)***	4.3 (0.1)	4.2 (0.2)
LOI (%OM)	*Pre-Ash*	89.9 (0.4)	88.9 (0.5)	59.9 (3.3)	53.5 (5.2)	11.2 (1.0)	12.0 (0.8)
	Post-Ash	88.4 (1.1)	60.4 (5.3)***	52.2 (4.5)	57.3 (5.3)	10.5 (0.7)	10.1 (0.7)
Ca (mg/kg)	*Pre-Ash*	9755 (449)	9602 (498)	5074 (595)	4603 (618)	279 (42.5)	330 (67.3)
	Post-Ash	11,321 (409)	15,322 (451)***	5151 (486)	9631 (1262)**	223 (20.3)	520 (180)*
K (mg/kg)	*Pre-Ash*	1265 (117)	1040 (56.2)	477 (41.3)	346 (36.9)*	44.7 (7.0)	51.5 (6.6)
	Post-Ash	1449 (78.1)	846 (41.7)***	501 (53.1)	961 (125)*	46.3 (4.9)	396 (157)**
Mg (mg/kg)	*Pre-Ash*	1057 (51.5)	880 (36.1)**	409 (47.4)	293 (37.5)	27.7 (2.6)	26.7 (4.8)
	Post-Ash	1275 (53.8)	1404 (117)	460 (47.7)	1581 (324)**	20.9 (1.5)	67.4 (19.0)**

Table 3. Average elemental metal concentrations (± SE) of LFH and mineral soil sampled beneath mature sugar maple trees (*n* = 24) 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 (*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.001$).

Element	**Treatment**	**Litter**		***FH***		**Min I**	
		Control	*6t ha-1*	*Control*	*6t ha-1*	*Control*	*6t ha-1*
Al (g/kg)	*Pre-Ash*	0.3 (0.0)	0.4 (0.1)	2.1 (0.3)	3.6 (0.6)	8.1 (1.4)	10.0 (1.1)
	Post-Ash	0.4 (0.0)	1.3 (0.3)***	2.3 (0.4)	2.0 (0.2)	10.5 (1.0)	8.7 (1.1)
Fe (g/kg)	*Pre-Ash*	0.3 (0.04)	0.6 (0.1)	5.6 (0.8)	7.7 (1.1)	16.3 (0.9)	18.0 (1.2)
	Post-Ash	0.6 (0.1)	1.1 (0.2)**	5.4 (0.6)	4.2 (0.8)	15.5 (0.7)	14.1 (0.7)
Mn (g/kg)	*Pre-Ash*	1.8 (0.1)	2.2 (0.2)	1.3 (0.3)	2.0 (0.4)	0.3 (0.1)	0.8 (0.2)
	Post-Ash	1.5 (0.1)	4.9 (0.5)***	0.9 (0.1)	2.1 (0.4)	0.2 (0.0)	0.5 (0.1)
Cd (mg/kg)	*Pre-Ash*	0.4 (0.2)	1.1 (0.2)*	0.1 (0.1)	0.2 (0.1)	0.01 (0.01)	0.04 (0.02)
	Post-Ash	0.3 (0.2)	1.2 (0.2)**	0.7 (0.5)	1.0 (0.2)**	0.02 (0.01)	0.03 (0.01)
Cu (mg/kg)	*Pre-Ash*	12.6 (1.2)	9.4 (1.0)	13.7 (0.6)	13.2 (1.0)	2.5 (1.4)	2.5 (1.1)
	Post-Ash	16.1 (0.5)	68.7 (12.0)***	18.2 (1.3)	20.1 (4.2)	6.0 (0.6)	6.8 (0.8)
Ni (mg/kg)	*Pre-Ash*	0.9 (0.2)	0.9 (0.3)	3.6 (0.7)	5.3 (0.7)	4.5 (1.6)	3.0 (0.7)
	Post-Ash	4.4 (0.3)	5.4 (0.9)	4.9 (1.2)	4.9 (0.5)	3.6 (0.4)	3.8 (0.4)
Pb (mg/kg)	*Pre-Ash*	2.0 (0.5)	2.6 (0.5)	32.6 (6.5)	35.6 (3.8)	16.0 (2.4)	12.3 (2.5)
	Post-Ash	2.5 (0.5)	10.3 (1.7)***	18.1 (3.4)	19.3 (4.3)	8.5 (2.6)	6.5 (1.3)
Zn (mg/kg)	*Pre-Ash*	60.0 (4.6)	58.6 (4.6)	63.0 (3.3)	69.5 (11.8)	33.8 (3.2)	36.3 (2.9)
	Post-Ash	57.3 (4.5)	271.4 (38.9)***	55.6 (4.1)	92.9 (16.6)*	25.4 (2.0)	26.7 (2.7)

*As and B were undetectable and therefore removed.

Sap Yield and Chemistry

The optimal January-May mean temperature for peak sap volume is 1ºC (Rapp et al., 2019) and reported 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 (Figure 3). Additionally, earlier sap collection corresponded with increasing March mean temperatures (-1ºC in 2021 and -3ºC in 2022). On the other hand, sap sugar content exhibits a negative linear relationship with the May-October mean temperature of the previous growing season (Rapp et al., 2019) which 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.

In 2021 sap yield in the treated plots was approximately twice that in the untreated plots, whereas in 2022 sap yield in both the treated and untreated plots was almost identical (Figure 4). 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 3; Figure 4). No significant treatment effect was found on pH or sap 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 4). In contrast to sap flow there were no distinct seasonal patterns observed in sap nutrient and metal concentrations (Appendix A-2). Significant increases were observed in Ca, Mg, Mn, Zn, Cu, and Ni concentrations in the treated trees in 2021, while in 2022 concentrations of K, P, Cu, and Ni were significantly higher in the treated trees (Table 4). Further, in 2022 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 4). Largely due to a much higher sap flow in 2021, significant increases were observed in the mean seasonal elemental flux of most nutrients and metals in the treated trees, but a significantly higher flux was only observed in K and P in 2022 (Table 5). In 2021, seasonal fluxes of nutrients and metals in the ash treated trees increased by between 2% and 226% (Table 5). Both sap nutrient and metal concentrations fell within the reported ranges of sugar maple syrup elemental concentrations as gathered by Mohammed et al. (2022) and adjusted for an approximate 50-times concentration during distillation (Table 5).

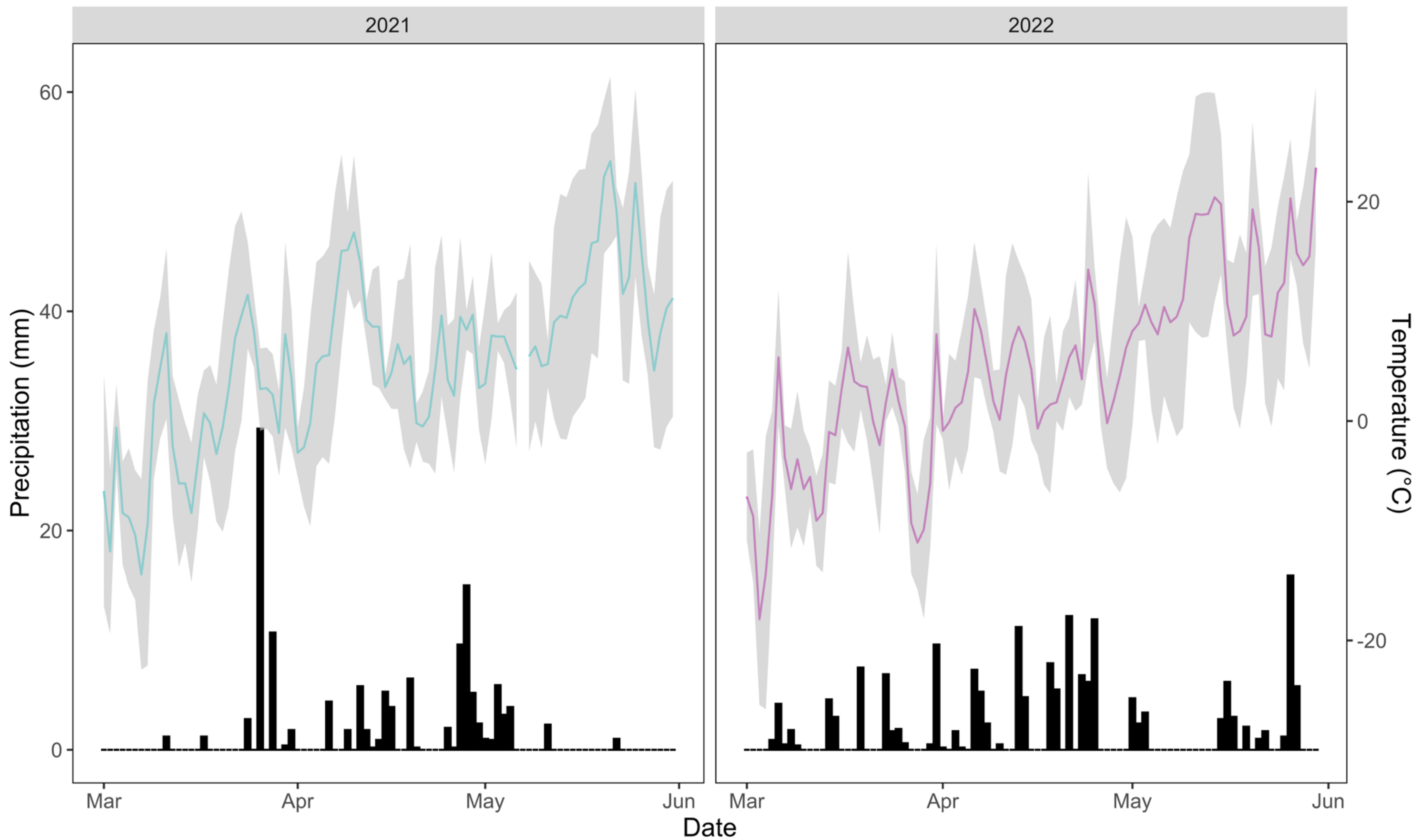


Figure 1. Climograph for Muskoka, ON during maple sap sampling seasons in 2021 (March – May) and 2022 (March – April). Bars indicate average daily precipitation (mm) and lines indicate average daily temperature (°C) with minimum and maximum daily temperatures in grey shading. Breaks in the lines indicate missing data (Environment and Climate Change Canada, 2022).

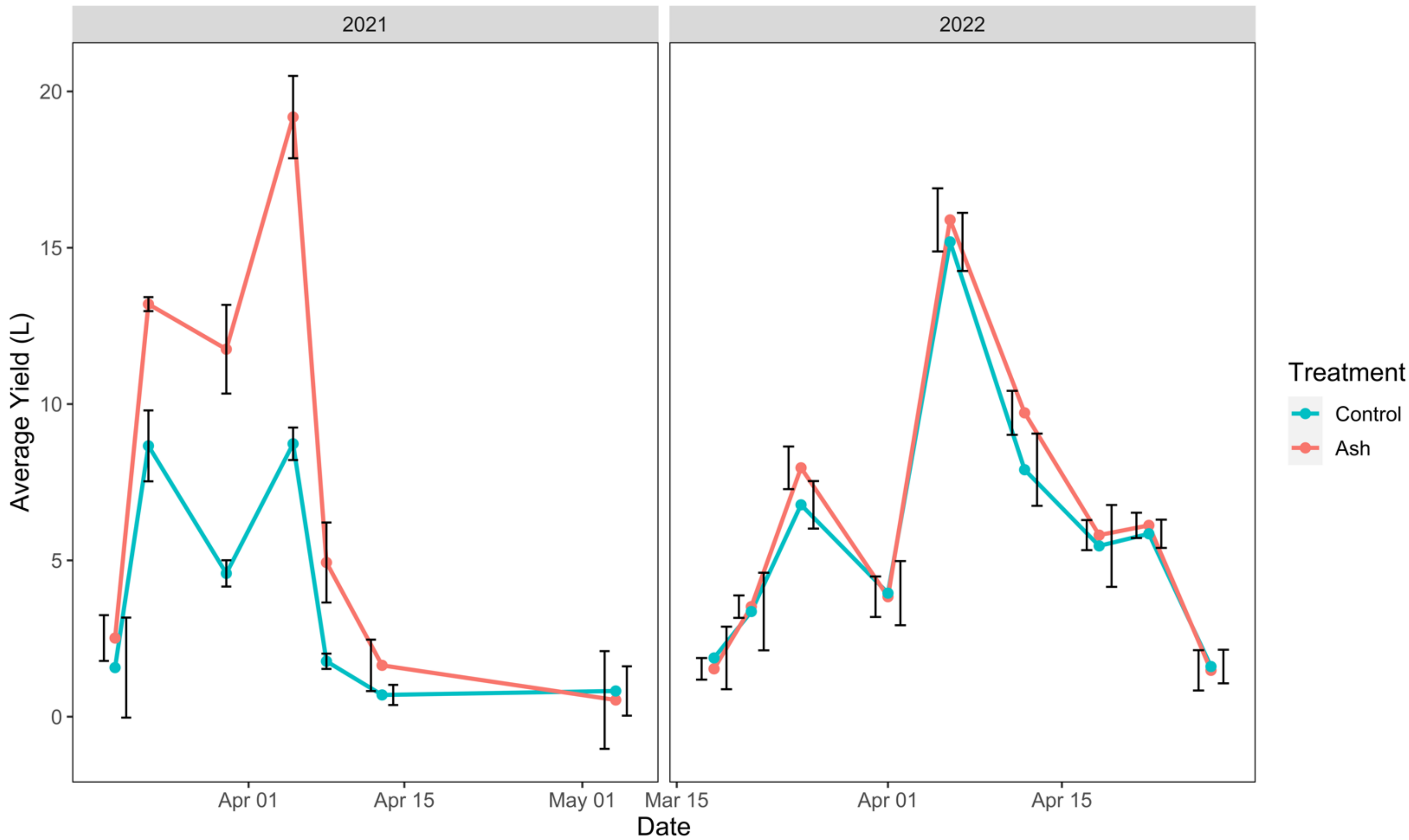


Figure 2. Average yield (L ± SE) produced by sugar maple trees (n = 24) in the control and ash treated plots. Sampling was conducted from March 20-May 4 in 2021 and March 18-April 27 in 2022.

Table 4. Average (± SE) volume-weighted pH, °Brix, total yield, and elemental concentrations in sugar maple sap (n = 24) 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 a Wilcoxon rank-sum test except for differences in average yield that were determined by a one-way ANCOVA to account for DBH. Also included are the ranges of elemental concentrations in sugar maple sap adjusted 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)	53.8 (5.5)**	52.0 (5.7)	55.9 (5.5)	
pH	6.40 (0.21)	6.41 (0.25)	7.00 (0.04)	7.04 (0.03)	
°Bx	1.35 (0.07)	1.44 (0.07)	1.37 (0.06)	1.39 (0.05)	
	$mg \cdot L^{-1}$				$mg \cdot L^{-1}$
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
Mn	5.1 (0.4)	6.0 (0.3)**	4.0 (0.2)	1.2 (0.1)***	0 – 5.0
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
	$ug \cdot L^{-1}$				$ug \cdot L^{-1}$
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

†Mohammed et al. (2022)

*As and B were indetectable and therefore removed.

Table 5. Annual elemental flux (± SE) in sugar maple sap ($n = 24$) 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---			
Ca	1524 (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)
Mn	127 (29.3)	279 (32.4)**	207 (34.7)	60.9 (7.3)**
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)
	---ug/tree---			
Zn	5528 (1015)	13095 (1333)***	13297 (2027)	8437 (836)
Al	621 (111)	1092 (103)**	1265 (205)	1370 (154)
Fe	420 (251)	431 (64.7)*	949 (437)	1133 (314)
Cu	170 (57.1)	555 (95.1)**	364 (97.0)	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 indetectable and therefore removed.

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 g/kg in the treated foliage compared with 7.6 g/kg in the controls) (Figure 5). 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 (Figure 5; Table 6). Percent C and N were also similar across treatments (Table 6).

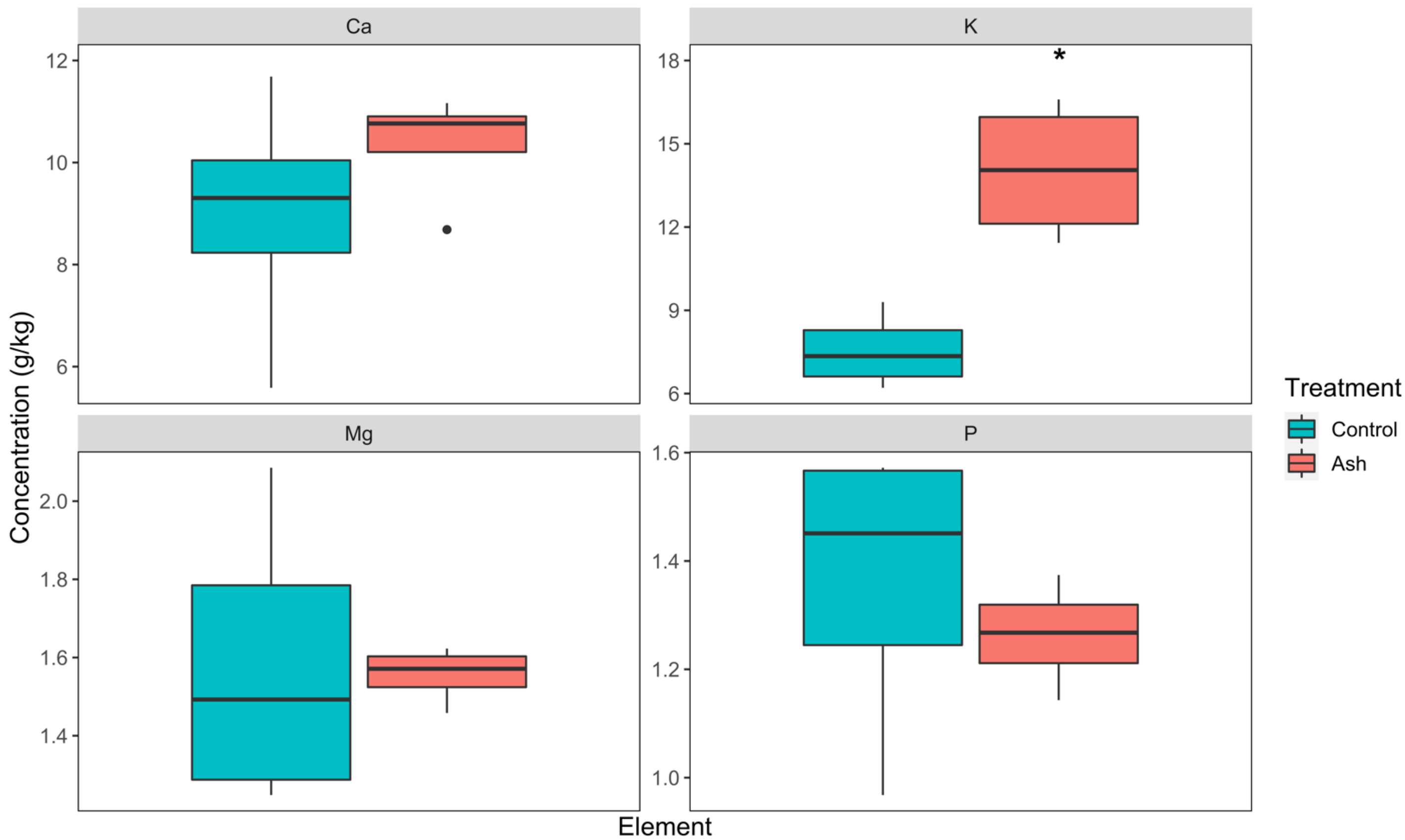


Figure 3. Average foliar nutrient concentrations of Ca, K, Mg, and P in mature sugar maple trees (*n* = 8) collected 8 months after application of 6t ha^{-1} non-industrial wood ash to experimental plots in Bracebridge, ON. Significant differences between treatment and control indicated by an asterisk (*, $p < 0.05$) as determined by a Wilcoxon rank-sum test.

Table 6. Average elemental concentrations (± SE) in mature sugar maple foliage (n = 8) during the 2021 sampling season. Significant differences between control and treatment indicated by an asterisk as determined by a Wilcoxon rank-sum test (*, $p < 0.05$).

Tissue Type	Treatment	---------------------------------------mg·kg^{-1}---------------------------------------							--ug·kg^{-1}--
		%C	%N	Mn	Fe	Zn	Al	Cu	Ni	Cd
Foliage	Control	46.1 (0.5)	2.3 (0.1)	885 (178)	49.9 (4.8)	26.8 (3.1)	14.3 (1.1)	2.9 (0.5)	0.3 (0.1)	0.4 (0.4)
	Treatment	45.3 (0.3)	2.2 (0.1)	1083 (150)	58.3 (8.1)	33.3 (2.6)	19.1 (2.5)	2.6 (0.4)	1.1 (0.7)	1.1 (0.9)

*As, B, Pb and Na were indetectable and therefore removed.

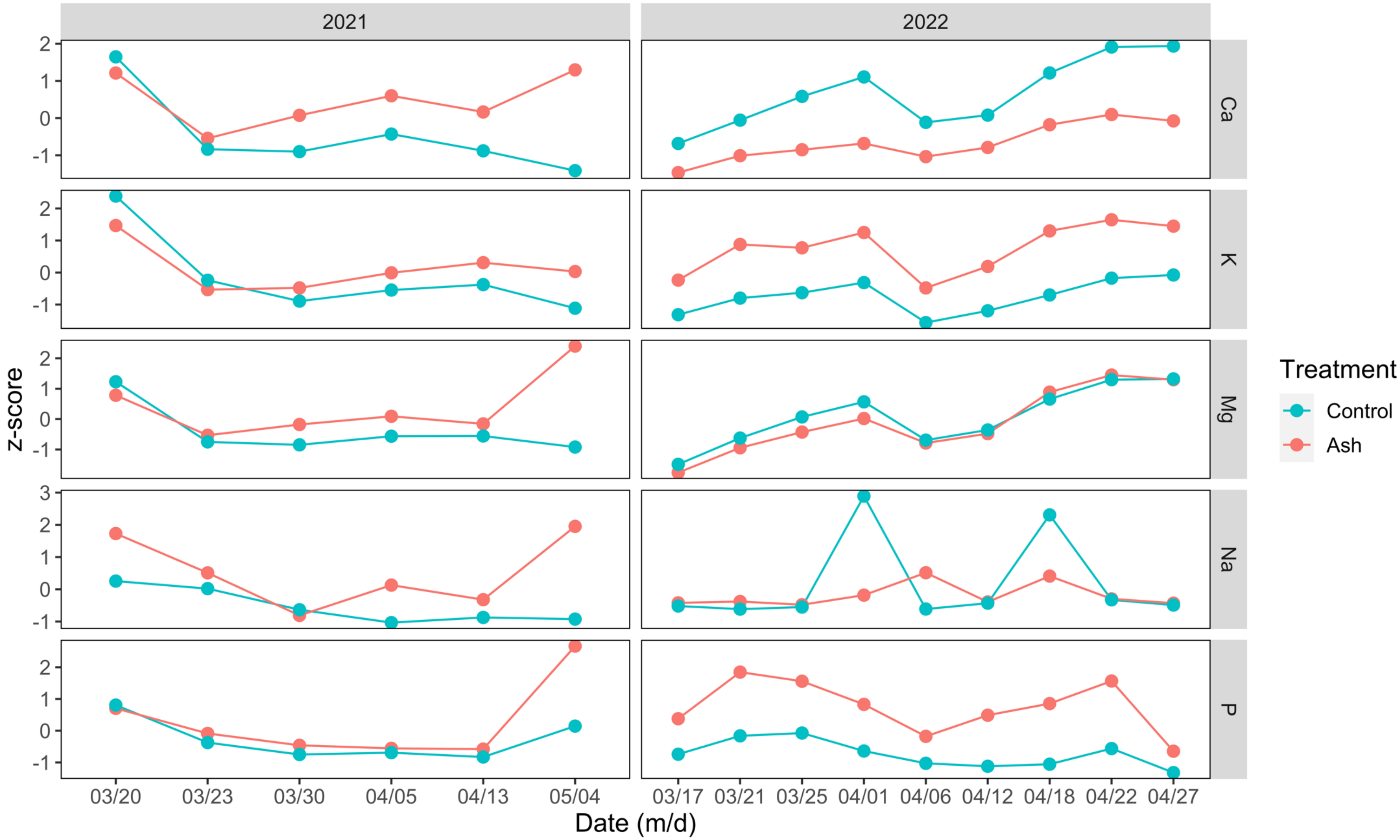
Appendices

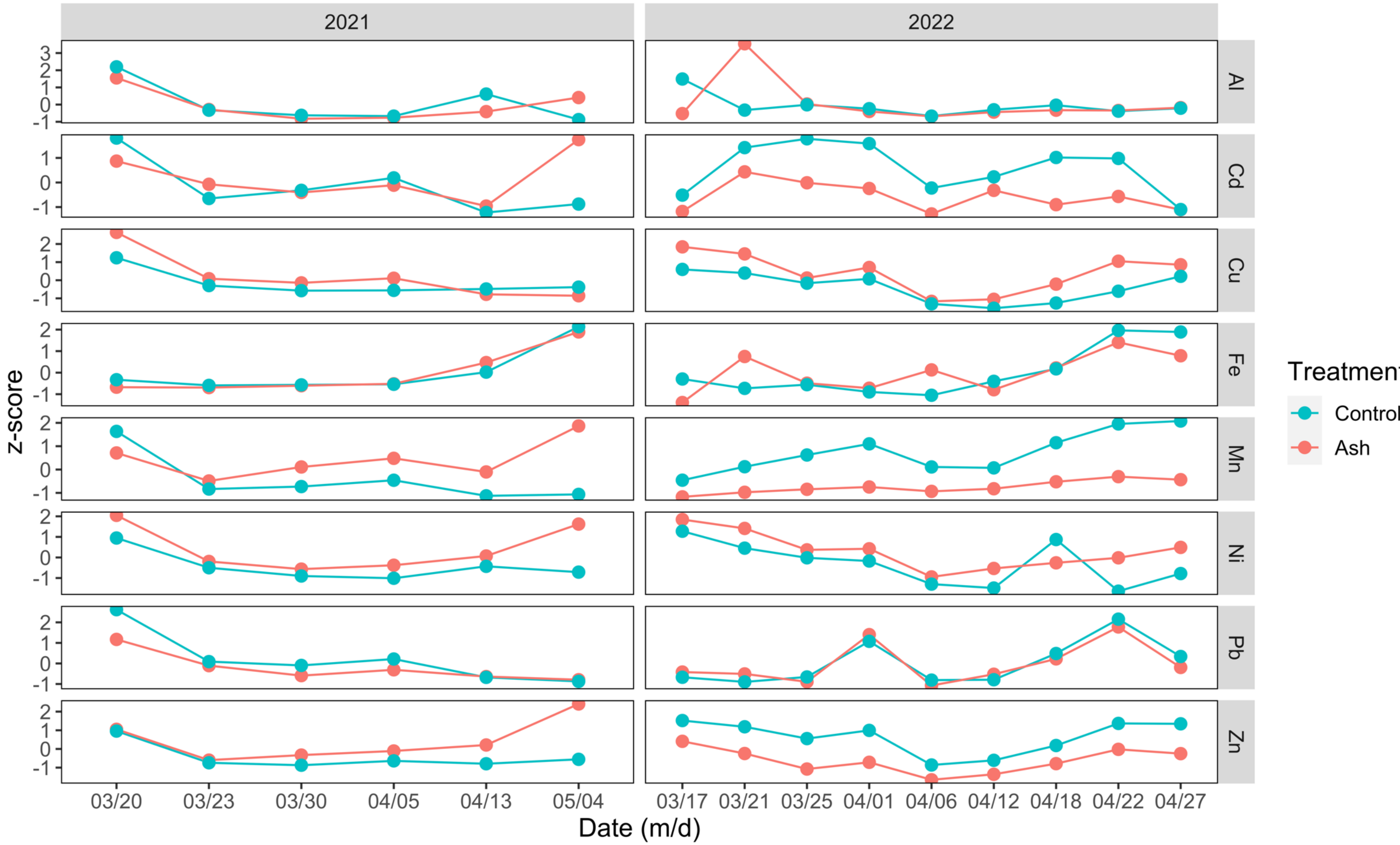
A-1. Maximum allowable soil metal concentrations for NASM application.

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


2021
2022
Ca
K
Mg
Na
P
z-score
Treatment
Control
Ash
03/20
03/23
03/30
04/05
04/13
05/04
03/17
03/21
03/25
04/01
04/06
04/12
04/18
04/22
04/27
Date (m/d)



A-2 a) b). Standardized elemental concentrations of a) Ca, K, Mg, Na, P, and b) Al, Cd, Cu, Fe, Mn, Ni, Pb, and Zn in sugar maple sap (*n* = 24) in both control and ash treated plots in 2021 and 2022.

Discussion

The purpose of this study was to evaluate the effects of NIWA on sugar maple soil, foliage, and sap yield and sweetness. Non-industrial wood ash collected from volunteer residents of Muskoka contained a variety of important nutrients (Ca, K, Mg, P) for plant growth and low metal concentrations that were within regulatory guidelines, limiting any potential toxicity effects. Ash application increased organic soil pH and extractable base cation concentrations. Metal concentrations increased in the litter layer in the treatment plots but remained similar in the FH and mineral layers of soil. While sap yield almost doubled in the treatment plots the first year following application, this effect was not sustained the following year where yield was almost identical between the control and treatment plots. Similarly, no effect was observed on sap sweetness in either year. Sap nutrient and metal concentrations were variable and no consistent pattern was observed between years and of the differences that were significant most were small. Foliar K also increased significantly in the treatment plots, but other base cation and metal concentrations were similar to controls.

Non-industrial wood ash collected from volunteer residents of Muskoka contained important nutrients for plant growth while metal concentrations remained low. The NIWA averaged a pH of 13, a value consistent with but at the higher range of those reported in the literature (Augusto et al., 2008; Deighton & Watmough, 2020; Demeyer et al., 2001; Pitman, 2006). Calcium was present in the greatest concentration at 27%, followed by K at 9% and Mg at 2% similar to values reported by Azan et al. (2019) at 30% Ca, 8% K and 2% Mg. These values are slightly higher than those reported by Deighton and Watmough (2020) who evaluated ash produced from sugar maple, white pine, and yellow birch and reported 16%, 25%, and 21% Ca, 3%, 6%, and 6% K, and 1%, 1%, and 1% Mg, respectively. These differences are likely due to the fact that the ash used in this study was mixed from various hardwood species unlike those in

Deighton and Watmough (2020) that were from single species only. 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. The concentrations reported here are 164 mg kg^{-1} Cu and 503 mg kg^{-1} Zn, similar to those reported in Azan et al. (2019) of 100.5 mg kg^{-1} Cu and 500.6 mg kg^{-1} Zn. Though they fall slightly above CM1, both concentrations are substantially lower than the CM2 limits and therefore not likely to pose a risk of toxicity to the soil or plants.

Prior to application no significant differences were observed between the treatment and control plots in soil pH or organic matter content. One year following treatment, soil pH increased significantly in the treated LFH layers, but no differences were observed in the mineral layers. 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 1.6 units and the FH layer by 0.7 units; such increases in pH tend to be found 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; Deighton & Watmough, 2020; Ozolinčius et al., 2007; Reid & Watmough, 2014). A longer period of time before increases are noticeable in the mineral layers is also well documented (Lundström et al., 2003; Moore et al., 2012; Ozolinčius et al., 2007) as the neutralization effects take longer to migrate to deeper layers of the soil as slow decomposition rates lead to base cations being largely retained in the organic horizons (Reid & Watmough, 2014). For example, Saarsalmi et al. (2001) found no effect on mineral soil pH 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). Percent organic matter in the litter layer decreased significantly in the treatment plots one 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 found (Deighton & Watmough, 2020; Saarsalmi et al., 2001) and those found here are most likely a result of the ash remaining on the uppermost layer of soil during sampling the following year. However, the application of wood ash has been observed to increase organic matter mineralization through an increase in abundance of ammonifying and nitrifying organisms (Augusto et al., 2008; Saarsalmi et al., 2004).

Significant increases in almost all nutrient concentrations were seen in the organic and upper mineral soil horizons while metal concentrations remained low. Short-term and sustained increases in extractable Ca and Mg in the organic horizons are commonly found in the literature (Arseneau et al., 2021; Augusto et al., 2008; Reid & Watmough, 2014; Saarsalmi et al., 2001, 2004). While the increases observed here were the most significant in the litter (Ca) and FH (Ca and Mg) layer, Ca and Mg did increase significantly in the upper mineral horizons one year after application as well, similar to other studies (Arseneau et al., 2021; Augusto et al., 2008; Deighton & Watmough, 2020; Ozolinčius et al., 2007; Saarsalmi et al., 2001, 2004). On the other hand, extractable K was significantly lower in the litter layer while increasing in the FH and upper mineral soil. The decrease in K in the litter layer is likely due to the high solubility of K and it's displacement off soil exchange sites by other cations such as Ca and Mg (Ohno, 1992; Reid & Watmough, 2014). It is possible that K increases occur more rapidly as Arseneau et al. (2021) found no significant differences between control and treated plots in the forest floor or upper mineral soil three years after 20 Mg ha^{-1} wood ash application. Increases in K concentrations in the organic horizons after application are most commonly observed in the short-term (1 – 5 years) (Augusto et al., 2008; Ozolinčius et al., 2007) but have been observed to persist in deeper soil profiles in the long-term (6 – 16 years, Augusto et al., 2008; Bramryd & Fransman, 1995; Saarsalmi et al., 2004). Significant increases in all metals except for Ni were noted in the litter layer of the treatment plots one year following wood ash application but were only observed in Cd and Zn in the FH layer, and none were observed in the upper mineral horizon. These increases in metal concentrations in the litter are to be expected due to the slow decomposition rate of the ash observed in this study and are consistent with other findings (Deighton et al., 2021; Hansen et al., 2018). Furthermore, metal mobility will likely be restricted with the decrease in acidity and high levels of organic matter (Augusto et al., 2008; Violante et al., 2010).

Sap yield increased significantly the first year following wood ash application but was not observed in the second year. While the observations here could suggest an effect of increased yield shortly following application, research on the effect of wood ash on sap yield is limited and the effects of fertilizer treatment on yield are quite variable. For example, a study conducted by Moore et al. (2020) in Québec found that liming indirectly improved sap yield in a base-poor stand eighteen years after application suggesting that the positive effect of liming on tree growth ultimately leads to increased sap yield in the long-term. 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). Additionally, sap production is highly influenced by climate variability (Kim & Leech, 1985; Marvin & Erickson, 1956). 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 (Cirelli et al., 2008; Tyree, 1983) and therefore sap production and total yield depend largely on temperatures during the production season and the preceding months (Rapp et al., 2019). In this study, sap yield peaked earlier in the year in 2021 than 2022 which corresponds with the earlier warming observed the first year following application. This does not however explain the differences observed between the control and treatment plots in 2021 and therefore it is possible that the increase in yield is a result of the wood ash application.

No effect was observed on sap sugar concentration in either year following ash application. This result is similar to one 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) but these results contrast with other fertilization research. 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 also limited. Sugar content in sap 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 nearly doubled the first year following application no dilution effect was observed in sap sweetness between the control and treated plots. Ultimately this study provides evidence that the application of wood ash would not negatively impact sweetness.

Changes in nutrient and metal concentrations in the sap post-application were not consistent between years and most differences were small. In both control and treated plots Ca, Mg, and K were present in the greatest concentrations in both years, as found previously (Lagacé et al., 2015; Mohammed et al., 2022; Yuan et al., 2013). The first year following application the most significant increases were seen in Ca (225%) and Mg (15%). In the second year, significant decreases were noted in Ca (52%) and Mg (5%) and increases 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}) and Mg (2 and 5 Mg ha^{-1}) but not K in the sap. These results provide evidence that surges in K concentrations are likely to occur in the short-term and increases in Ca and Mg availability may extend over longer periods of time (Moore & Ouimet, 2021). An antagonistic relationship between K and Mg uptake by plants is also well recognized in the literature and thus it's possible that high concentrations of Mg in the first year prevented 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 increases were noted in the metals but these were also inconsistent between years and all metal concentrations remained within the average range in maple syrup as gathered in a review by Mohammed et al. (2022). It is also common for metal concentrations to fluctuate across years in maple syrup (Mohammed et al., 2022). These results highlight the natural variability of sap composition and suggest that wood ash application may increase essential nutrient concentrations without risking toxicity from metals.

In general, foliar base cation and metal concentrations were similar between control and treated plots one year after application in mature sugar maple trees. No significant changes 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 (Bal et al., 2015; Kolb and McCormick, 1993). 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). Only foliar K concentrations exhibited a significant increase 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 because deficiencies have been linked with reduced photosynthetic capacity (Xie et al., 2021) which may ultimately impact sap sweetness. Foliar concentrations of K for a healthy sugar maple range from 5.5-10.4 g kg^{-1} 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} (Kolb and McCormick, 1993). A study conducted by Arseneau et al. (2021) found no significant differences in N, P, K, or Ca concentrations in mature 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 concentrations would not pose a risk. There were also no significant changes in foliar metal concentrations, and all values remained within healthy ranges for sugar maple trees except for Al that was below the healthy range (32-60 g kg^{-1}, Kolb and McCormick, 1993) but this was consistent between treatments.

Conclusion

This study evaluated the effect of applying 6 t ha^{-1} NIWA in a mixed-wood forest in Central Ontario. Wood ash application is of concern because of the metals that may pose a risk of toxicity to plants and soil, but the results here reveal that all metals are well-within regulatory guidelines for application to soils. One year following application significant increases were observed in the organic horizon pH of the treatment plots. Similarly, increases were observed in Ca, Mg and K concentrations in both the organic and mineral layers of the soil. Metal concentrations increased significantly in the litter layer – consistent with research previously mentioned – and aside from Cd and Zn in the FH layer these increases were not observed in either the FH or mineral layers. Despite these increases after application all metal concentrations in the soil remained below regulatory guidelines for soils prior to NASM application.

The first year following wood ash application sap yield almost doubled in the treatment plots but this effect was not observed the following year. It is possible that this is a treatment effect, but further research could be conducted to see if this result can be replicated. Non-industrial wood ash application had no effect on sap sweetness but did increase nutrient concentrations while metals remained within common concentration ranges found in sugar maple syrup. No obvious patterns were observed in either nutrient or metal concentrations in the sap and therefore fluctuations are likely a result of natural variability between years. One year after application significant increases were observed in foliar K concentrations that may increase photosynthetic capacity, but otherwise no foliar response was found. As a result, wood ash does not have a negative effect on sugar maple sap and may supplement essential nutrients for its production, sweetness, and overall quality.

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