Macronutrients, metals, and metalloid concentrations in non-industrial wood ash in relation to provincial land application limits in Ontario, Canada

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ABSTRACT

Non-industrial wood ash (NIWA) provided by residents who heat with wood, is rich in base-cations and other nutrients and may be used as a forest soil amendment to return nutrients lost through acid deposition. However, due to concerns regarding high trace metal concentrations, most wood ash is landfilled in Canada. This study investigated the chemical variability of NIWA of individual samples and homogenized mixtures to determine if they met Ontario provincial trace metal restriction limits. One hundred and seven ash and 10 charcoal samples collected from residents of Muskoka, Ontario, and three 10-sample composites were analyzed. Chemical composition varied among individual samples, but nutrient levels were within or higher than reported ranges for industrial wood ash, while trace metal values were lower. Ninety-seven percent (104 of 107) of the samples were within Ontario Regulation 267/03 of the Nutrient Management Act, and after homogenization, all samples were below soil application restriction limits. This study indicates that NIWA can be safely used as a forest soil amendment but recommends routine testing of batch samples prior to application.

Keywords: non-industrial wood ash, wood ash chemistry, trace metals, base cations, biomass

RÉSUMÉ

Les cendres de bois non-industriel (CBNI) fournies par les résidents qui chauffent au bois, sont riches en cations basiques et en éléments nutritifs et peuvent être utilisées en tant qu'en amendement des sols forestiers pour remplacer les éléments nutritifs perdus par l'acidification des sols. Toutefois par suite d'inquiétudes portant sur les fortes concentrations en oligo-éléments, la majeure partie des cendres de bois est actuellement au Canada jetée au dépotoir. Cette étude a relevé la variabilité chimique des CBNI à partir d'échantillons individuels et de mélanges homogènes, afin de déterminés si les normes provinciales de l'Ontario étaient respectées en termes de limites admissibles des oligo-éléments. Cent sept échantillons de cendres résidentielles et dix échantillons de charbons de bois ont été recueillis chez des résidents de Muskoka en Ontario et trois échantillons de cendres provenant de composites de dix éléments ont été chimiquement analysés. La composition chimique des CBNI variaient d'un échantillon à l'autre mais les concentrations en éléments nutritifs étaient dans les limites ou encore supérieures aux amplitudes rapportées précédemment pour les cendres de bois industriels, tandis que les valeurs pour les oligo-éléments étaient inférieures. Les échantillons individuels étaient pour 97% d'entre eux (104 de 107) dans les limites de la norme 267/03 de la Loi sur la gestion des éléments nutritifs de l'Ontario, et après homogénéisation, tous les échantillons se sont retrouvés sous les limites de restriction d'utilisation sur des sols. Cette étude indique que les CBNI peuvent être utilisées sécuritairement en tant qu'amendements des sols forestiers mais recommande des tests de routine de lots d'échantillons avant leur utilisation.

Mots clés : cendres de bois non industriel, chimie des cendres de bois, oligo-éléments, cations basiques, biomasse

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Introduction

Atmospheric deposition of sulphur (S) and nitrogen (N) has greatly affected the biogeochemistry of forests in Europe and in eastern North America (Talhelm *et al.* 2012). The effects on forest soils have been compounded by climate change and forest harvesting, both of which have accelerated base cation losses, calcium (Ca) in particular (Driscoll *et al.* 2001; Fernandez *et al.* 2003; Akselsson *et al.* 2007; Cleavitt *et al.* 2018). Losses of soil nutrients have reduced tree health in North America (Drohan *et al.* 2002; Duchesne *et al.* 2002), and together with increases in soluble aluminum (Al), have been linked to the decline in the health of sugar maple, *Acer saccharum* Marsh (Horsley *et al.* 2000); Bal *et al.* (2014), red spruce, *Picea rubens* Sarg. (DeHayes *et al.* 1999), and potential changes in forest composition (Lawrence *et al.* 2018). Additionally, in eastern North America, low levels of foliar potassium (K), magnesium (Mg), phosphorous (P) and Ca have similarly been linked to sugar maple decline (Bernier and Brazeau 1988a, 1988b; Pare and Bernier 1989; Kolb and McCormick 1993), and low soil exchangeable Ca has been linked to hardwood forest canopy decline in southern Ontario (Miller and Watmough 2009).

Although acidic deposition has declined substantially, recovery of lost soil nutrients is predicted to take centuries (Ott and Watmough 2022). This has encouraged active replacement of the lost nutrients, and several studies have shown significant improvements in soil base status and tree health after the addition of lime (Juice *et al.* 2006; Huggett *et al.* 2007; Long *et al.* 2011) and wood ash (Ludwig *et al.* 2002; Pitman 2006; Saarsalmi *et al.* 2006). Recent shifts to biomass combustion from conventional fossil fuels has increased the production of wood ash (Demirbas *et al.* 2009). However, most of the wood ash, a substance rich in macronutrients, is landfilled in Canada (Hannam *et al.* 2018) due to concerns related to concentrations of trace metals (Pitman 2006) and variability in its chemical composition (Azan *et al.* 2019).

Wood ash consists of inorganic and organic residues generated from the combustion of wood and wood by-products (Siddique 2012) through industrial, commercial, and domestic use (Azan 2017; Hannam *et al.* 2018). Wood ash can be highly variable in its chemical composition and physical properties (Hannam *et al.* 2018). The differences in wood ash pH, organic matter, and nutrient and metal concentrations can be attributed to factors such as burn temperature and differences in feedstock material (Pitman 2006). For example, wide variations in chemical composition have been reported in wood ash from common Canadian tree species, with calcium (Ca) concentrations ranging between 156 to 250 g kg^{-1} (Deighton and Watmough 2020). In addition, combustion temperatures can also significantly affect ash yield and metal concentrations, with ash quantity tending to decrease and metal concentrations increase at higher combustion temperatures (Etitgni and Campbell 1991).

Wood ash can have industrial or non-industrial sources. Industrial wood ash (IWA) is produced through wood combustion in industries such as timber mills and pulp and paper (Hannam *et al.* 2018). Non-industrial wood ash (NIWA) is generated mainly through residential combustion for home heating but can also include commercial sources such as small businesses (i.e., wood-fired pizzerias) (Azan 2017). While mainly comprised of fines, the ash can include fragments of wood charcoal, and like wood ash, wood charcoal from different tree species can vary widely in its physical and chemical properties (Pluchon *et al.* 2014; Gezahegn *et al.* 2019).The chemical composition of wood charcoal, however, can differ from that of finer ash, with lower nutrient concentrations and lower pH values (Pluchon *et al.* 2014). While there are studies on the effects of naturally occurring wood charcoal (i.e., charcoal produced from wildfires), and wood-derived biochar on the biogeochemistry of soils (DeLuca *et al.* 2006; Pluchon *et al.* 2014; Yargicoglu *et al.* 2015), the chemical composition of wood charcoal associated with NIWA and its potential adverse effects, if any, are not well known. For NIWA to be used as a soil amendment, more research is needed to understand the chemical composition of wood charcoal and to determine if it should be removed from ash mixtures before soil application.

Commercial burning of wood can generate up to 1% of ash by weight, resulting in significant waste byproduct (Pitman 2006). Currently in Canada, about 1 million Mg of wood ash is produced per year, based on the total contribution from pulp and paper mills and forest biomass (Lamer *et al.* 2018). In Quebec, 150 000 Mg of wood ash was landfilled in 2005, while British Columbia deposited 96% of approximately 235 000 Mg in 2014 (Hannam *et al.* 2017). Non-industrial wood ash production is substantially less in Ontario; Azan (2017) estimated that approximately 18 000 Mg is generated annually from residences and usually destined for landfill. However, this practice is slowly changing as forest biomass use increases along with disposal costs, and alternative disposal methods are being considered.

Several European countries have established trace metal concentration limits on wood ash for approval as a forest soil amendment to ensure that ash application will not substantially increase trace metal concentrations within the ecosystem, and samples must be tested for compliance before application (Hannam *et al.* 2018). In Canada, there is no federal legislation related to the disposal of wood ash, and ash management policies vary among the provinces (Hannam 2016). Similar to other countries however, the ash must undergo a regulatory process before it can be approved for land application. Trace metal levels in ash samples must be determined and if levels exceed regulated limits, it cannot be used as a soil amendment (Hannam 2016).

In Ontario, wood ash falls under the Non-Aqueous, Non-Agricultural Source Materials (NASM) Regulation 267/03 of the Nutrient Management Act. There are two metal content limits in the regulation, CM1 and CM2. CM1 refers to the concentration of regulated metals that do not exceed the limits as defined by the regulation (column 5, Table 1). CM2 pertains to the levels of regulated metals that exceed the concentrations in CM1 however, do not exceed the concentrations in column 6 of Table 1. (O. Reg 267/03; Nutrient and Management Act 2002). Ash samples with concentrations below CM1 do not have restrictions for soil application; ash exceeding CM1 can be applied given that it does not exceed CM2 restrictions (Hannam 2016). Concerns over wood ash chemical variability and regulatory approval policies need to be addressed before wood ash can become a common soil amendment (Hannam *et al.* 2017).

This study had two objectives: 1) to evaluate the variability of the chemical composition of NIWA and associated wood

Table 1. Literature values for industrial bottom and fly ash generated in plants across Canada and previously reported literature values for NIWA from Muskoka, On., along with Ontario Regulation 267/03 of the Nutrient Management Act limits for unrestricted (CM1) and restricted (CM2) use of wood ash for land application as a non-agricultural non-aqueous source material (NASM) are also shown.

	NIWA (Lit. Values)	Industrial Bottom Ash	Industrial Fly Ash	NASM Limits**	
				CM Level 1	CM Level 2
pH	–	7.5 - 12.9*	8.6 - 13.8*	–	–
C (%)	–	0.5 - 51.8*	2.7 – 43*	–	–
N (%)	–	<0.01 - 0.3*	0.04 - 0.4*	–	–
S (%)	–	<0.01 - 2.5*	<0.01 - 4.7*	–	–
Ca (g.kg^{-1})	–	3.9 - 211 *	92.1 - 247.9*	–	–
Mg (g.kg^{-1})	–	0.6 - 33.1*	6.4 - 29.4*	–	–
K(g.kg^{-1})	–	0.8 - 50.8*	13.5 - 90.8*	–	–
P (g.kg^{-1})	–	0.1 - 11.9*	3.2 - 10.6*	–	–
Cd (mg.kg^{-1})	2.02 †	0.4 - 0.7 ††	6 – 40 ††	3	34
As (mg·kg^{-1})	0.61 †	0.2 – 3 ††	1 – 60 ††	13	170
Ni (mg·kg^{-1})	4.18 †	40 – 250 ††	20 – 100 ††	62	420
Pb (mg·kg^{-1})	3.05 †	15 – 60 ††	40 - 10^3 ††	150	1100
Cu (mg·kg^{-1})	100.49 †	15 – 300 ††	~200 ††	100	1700
Zn (mg·kg^{-1})	500.6 †	15 – 10^3 ††	40 – 700 ††	500	4200
Mn(mg·kg–^{1})		(2-5.5) – 10^3 ††	(6 - 9) – 10^3 ††	–	–

*AshNet 2018 – Ash Chemistry database *, Nutrient and Management Act 2002**, Azan (2019) †, Pitman (2006) ††*

Table 2. pH, LOI, CNS, nutrient and metal concentrations of unamalgamated non-industrial wood ash (n=107), and non-industrial wood charcoal (n= 10), samples collected from residents of Muskoka District during 2019.

	Unamalgamated								
	Wood Ash Chemistry				Non-industrial Wood Charcoal				
Chemistry	Mean	SD	Cv (%)	SE	Mean	SD	Cv (%)	SE	P value
pH	11	–	–	–	**10**	–	–	–	*<0.0015*
LOI	2.3	1.7	72.6	0.2	14.8	7.5	50.7	0.7	n.s
C (%)	8.8	1.3	14.7	0.1	18.6	6.8	36.6	0.7	n.s
N (%)	0.1	NA	NA	NA	0.2	0.1	50	0.0	n.s
S (%)	BDL	NA	NA	NA	BDL	NA	NA	NA	n.s
Ca (g.kg^{-1})	323	80	24.7	7.7	**232**	46	19.6	4.4	*<0.001*
Mg (g.kg^{-1})	26.1	8.4	32.1	0.8	**21**	7	33.3	0.7	*<0.001*
K(g.kg^{-1})	120	44.2	36.7	4.3	134	41.9	31	4.1	n.s
P (g.kg^{-1})	9.1	3.8	41.4	0.4	8	4	50	0.4	n.s
Cd (mg.kg^{-1})	3	2.4	80.1	0.2	1.8	0.7	38.9	0.1	n.s
As (mg·kg^{-1})	8.7	37.8	435	3.7	BDL	NA	NA	NA	n.s
Ni (mg·kg^{-1})	10.5	11.3	107	1.1	8	3.7	46.3	0.4	n.s
Pb (mg·kg^{-1})	45	196	437	18.9	7	3	42.9	0.3	n.s
Cu (mg·kg^{-1})	163	259	158	25	127	33	26	3.2	n.s
Zn (mg·kg^{-1})	502	355	70.9	34.3	408	124	30.6	12.0	n.s
Mn (mg·kg^{-1})	4853	1743	35.9	169	5366	1536	28.6	148.5	n.s
B (mg·kg^{-1})	239	71.9	30.1	7	216	72.7	33.7	7.0	n.s
Fe (mg·kg^{-1})	2057	2786	135	269	1412	456	32.4	44.1	n.s
Al (mg·kg^{-1})	4177	3238	77.5	313	2616	1101	42.1	106.4	n.s

Significant differences between unamalgamated ash and wood charcoal were determined by a Kruskal Wallis test. P value significant at 0.05 (Bolded within the table), Detection limit S – 181 Peak Area, As 0.003 mg/l, NA not applicable, n.s: not significant charcoal samples and compare to previously reported IWA chemistry; and 2) to re-evaluate the variability of NIWA samples after they are homogenized in bulk mixtures to determine whether they could be applied to forest soils and comply with Ontario's regulatory guidelines.

It was hypothesized that NIWA would contain high concentrations of base cations, P and metal concentrations comparable to reported IWA values, however some individual NIWA samples might exceed Ontario's NASM regulation 267/03 CM2 guidelines for some trace metals. It was also expected that, after homogenization, bulk mixtures of NIWA would be less variable in chemical composition and metal concentrations of all samples would be below the CM2 limits established in the NASM regulation.

Materials and methods

Study area

The District of Muskoka (6475 km^2) is in south-central Ontario at the southern edge of the Canadian Shield. The region had a permanent population of 60 599 in 2017 (Statistics Canada 2017) and sees approximately 83 000 seasonal residents due to its popularity as a cottage destination (Gallant 2017). Three percent of Muskoka's population heat their homes with wood biomass producing approximately 235 000 kg of ash annually (Azan *et al.* 2019). In a survey by Azan (2017), 57% of respondents reported using mostly hardwood for their heating needs and the most common species included maple (*Acer spp.*), beech (*Fagus grandifolia* Ehrh.), oak (*Quercus* spp.), birch (*Betula* spp.), ash (*Fraxinus* spp.), and cherry (*Prunus* spp.) (Azan 2017).

Study design

All NIWA for this study originated from various residences and small businesses within the Muskoka region. Contributors were informed that ash from the burning of construction waste, preserved, painted or pressure-treated wood would not be accepted. Wood ash was collected during seasonal ash drives at government approved waste collection stations (https://ashmuskoka.ca/locations/). Samples were screened and sieved to remove foreign objects, and a large magnet used to remove metallic contaminants such as nails and staples. Large charcoal pieces were also removed from all samples. Samples were stored in covered galvanized metal bins and further protected with a plastic tarp until homogenization and laboratory analyses. One hundred and seven samples were collected in October 2019 for laboratory analyses. Grab samples were collected and placed into labelled small plastic zip-lock bags. Ash from bins which showed signs of rust were not used due to the possibility of metal contamination. Ten wood charcoal samples were also collected randomly from within the donated ash for laboratory analyses. Grab samples were collected after ash was sieved and only the charcoal pieces remained. All samples were placed into labelled zip-lock bags for laboratory analyses.

The 107 ash samples could not be homogenized in a single step. Multiple, smaller portions of unamalgamated ash were placed into a cement mixer at random, while the mixer was on and rotating to create homogenized bulk mixtures. Once the mixer was full, it was emptied into a metal bin and the process was repeated until all 107 ash samples had been homogenized, placed into multiple bins, and divided into amalgamated ash mixtures A, B and C. Ten subsamples of each were collected at random for laboratory analyses (n = 30). Sample collection was conducted in the field while the amalgamated ash mixtures were land applied. Samples were collected and placed into zip-lock bags and marked with an identification code unique to that sample.

Laboratory analyses

Ash and wood charcoal analyses

Samples were oven dried for 24 hours at 110°C and then sieved (2 mm) again as a secondary precaution to remove any remaining foreign objects (e.g., nails, plastic parts, partially burnt paper) which may have been missed during the field sieving process. Samples were analyzed for pH_{CaCl2}, loss-on-ignition (LOI), carbon, nitrogen, and sulphur content (CNS), total metals (Zn, Pb, Ni, Mn, Fe, Cu, Cd, B, As and Al) and macronutrients (Ca, Mg, K, P). Wood charcoal samples were screened using a 2-mm sieve and any foreign objects removed and discarded. Samples were ground with a mortar and pestle and analyzed for pH_{CaCl2}, LOI, CNS, and total metals and macronutrients.

An OAKTON pH 510 series multimeter measured pH of the ash and wood charcoal samples in a 0.01M $CaCl_2$ slurry at 1:5 (ash: $CaCl_2$). All slurries were gently agitated for 45 mins, rested for 45 min before taking a pH reading. Organic matter was determined using the LOI method (Ball 1964). Five-gram samples were weighed into porcelain crucibles and oven dried for 8 hours at 110°C to derive the moisture content; samples were then placed into a muffle furnace at 450°C for 8 hours to determine percent organic matter. Sample CNS content was determined using a CNS combustion analyzer (Elementar vario MACRO cube CNS). Approximately 0.2 grams of ash and wood charcoal samples were weighed into 50 ml digiTUBEs, and 2.5 mL of 100% trace metal grade nitric acid was added using a precision repeater; each tube was appropriately labeled with the corresponding sample ID. A hot plate was used to digest the samples for 8 hours at 100°C, after which they were further digested at room temperature for an additional 8 hours until the entire sample dissolved. Sample tubes containing the digestates were individually rinsed with B-pure water three times and filtered into 25 mL volumetric flasks using a P8 fast flow filter paper. Solutions were further diluted to 25 mL with B-pure water and transferred into 50 mL Falcon tubes and refrigerated until analyzed. The method used was based on Havlin and Soltanpour (1980) and Ippolito and Barbarick (2000). Apple leaf standards (NIST – 1515 – SRM) and blanks were run at the beginning and end of every run of approximately 44 samples to test for accuracy and contamination (recoveries were 95 to 100 %). Ash samples which exceeded the CM2 limits and random selected samples were re-run to confirm concentrations and ensure that repeat values were within 5%.

Statistical analyses

To test the null hypothesis that amalgamated ash did not differ in chemical composition compared with unamalgamated ash, comparisons were made between unamalgamated samples (n = 107) and amalgamated samples (30) for pH, nutrient and metal concentrations using a Kruskal-Wallis rank sum test, since assumptions of normality, according to the Shapiro-Wilk's test, were not met. Significance level was set at

$P = 0.05$. A *post hoc* Dunn's test with a Bonferroni correction, was completed only on those variables where a significant difference was determined. Comparisons between unamalgamated ash samples and wood charcoal samples were also conducted using a Kruskal-Wallis rank sum test.

To further investigate the chemical variability of unamalgamated NIWA, Spearman's correlation matrices were calculated to identify correlations between unamalgamated sample metal and nutrient concentrations.

Results

Unamalgamated non-industrial wood ash chemistry

Unamalgamated non-industrial wood ash chemistry varied considerably among the 107 samples; however, elemental concentrations were generally consistent with previously reported industrial ash values, with some notable exceptions (Tables 1 and 2). For example, mean ash Ca and K concentrations were higher in our study compared with reported ranges for industrial ash, and mean Mg and P levels were comparable to the highest values reported for industrial wood ash (Tables 1 and 2). In addition, average concentrations of most metals fell below or were at the lower end of reported industrial ranges. Nevertheless, mean Cd (3.0 mg kg^{-1}), Zn (502.1 mg kg^{-1}) and As (8.7 mg kg^{-1}) concentrations were above the unrestricted (CM1) NASM guidelines but well below the restricted (CM2) guidelines (Tables 1 and 2). Ash S concentrations were below detection limits, while reported ranges for industrial ash were generally higher. Average pH was generally within the reported industrial ash values while C and N were at the lower end of reported values (Tables 1 and 2).

While mean values indicate that NIWA was generally rich in base cations and P concentrations, with low levels of trace metals compared with industrial ash, large variations in unamalgamated ash pH, nutrient and metal concentrations was observed. For example, approximately 50% of the samples had a pH > 13, but pH could be as low as 10. Similarly, Ca concentrations in approximately 35% of the samples were <299 g kg^{-1} yet individual samples could have Ca concentrations as high as 676 g kg^{-1} (Fig. 1). Similarly, trace metal con-

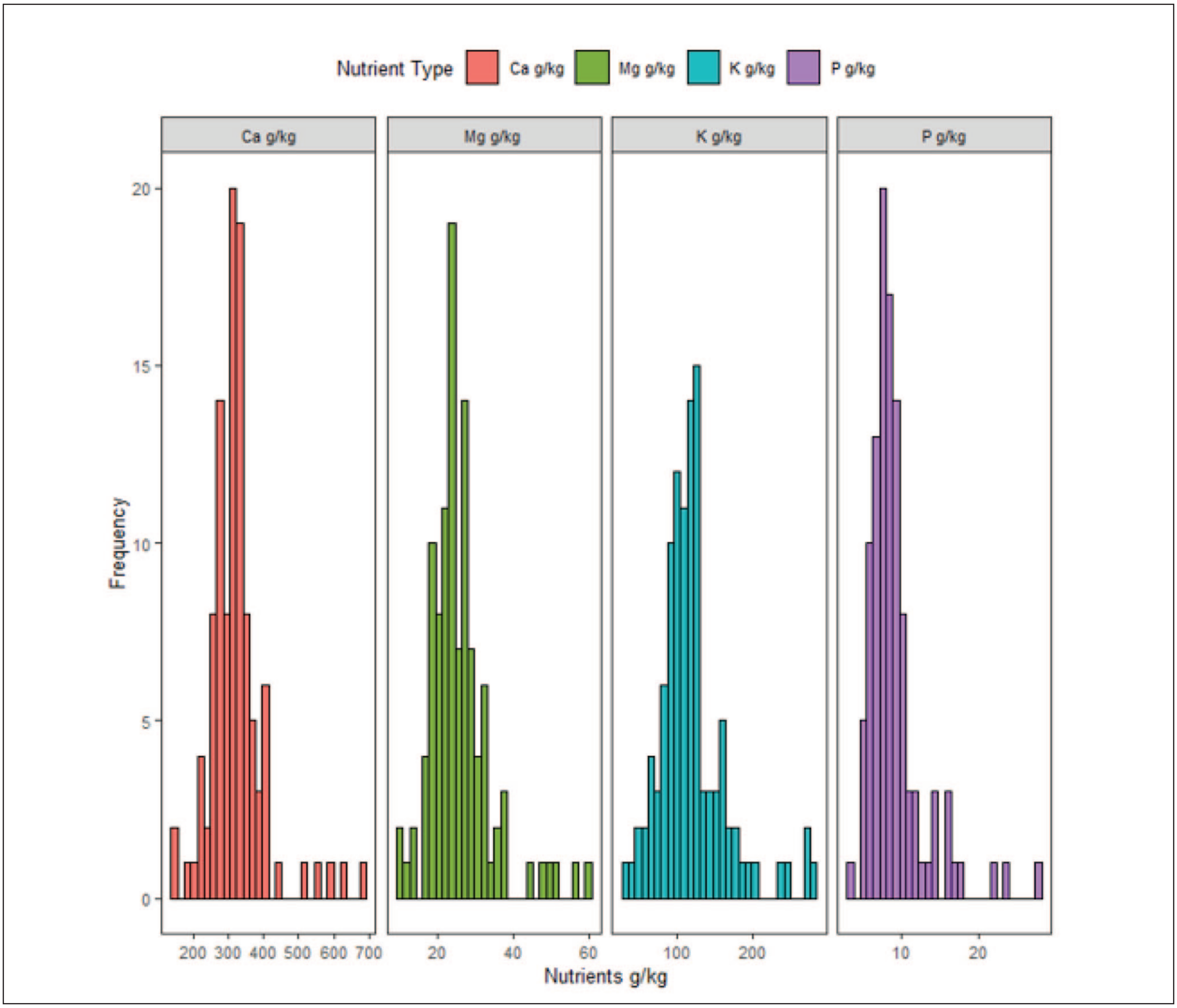

Fig. 1 Unamalgamated Wood Ash Chemistry – Nutrients; Distribution of Ca, Mg, K and P concentrations of unamalgamated non-industrial wood ash (n = 107)

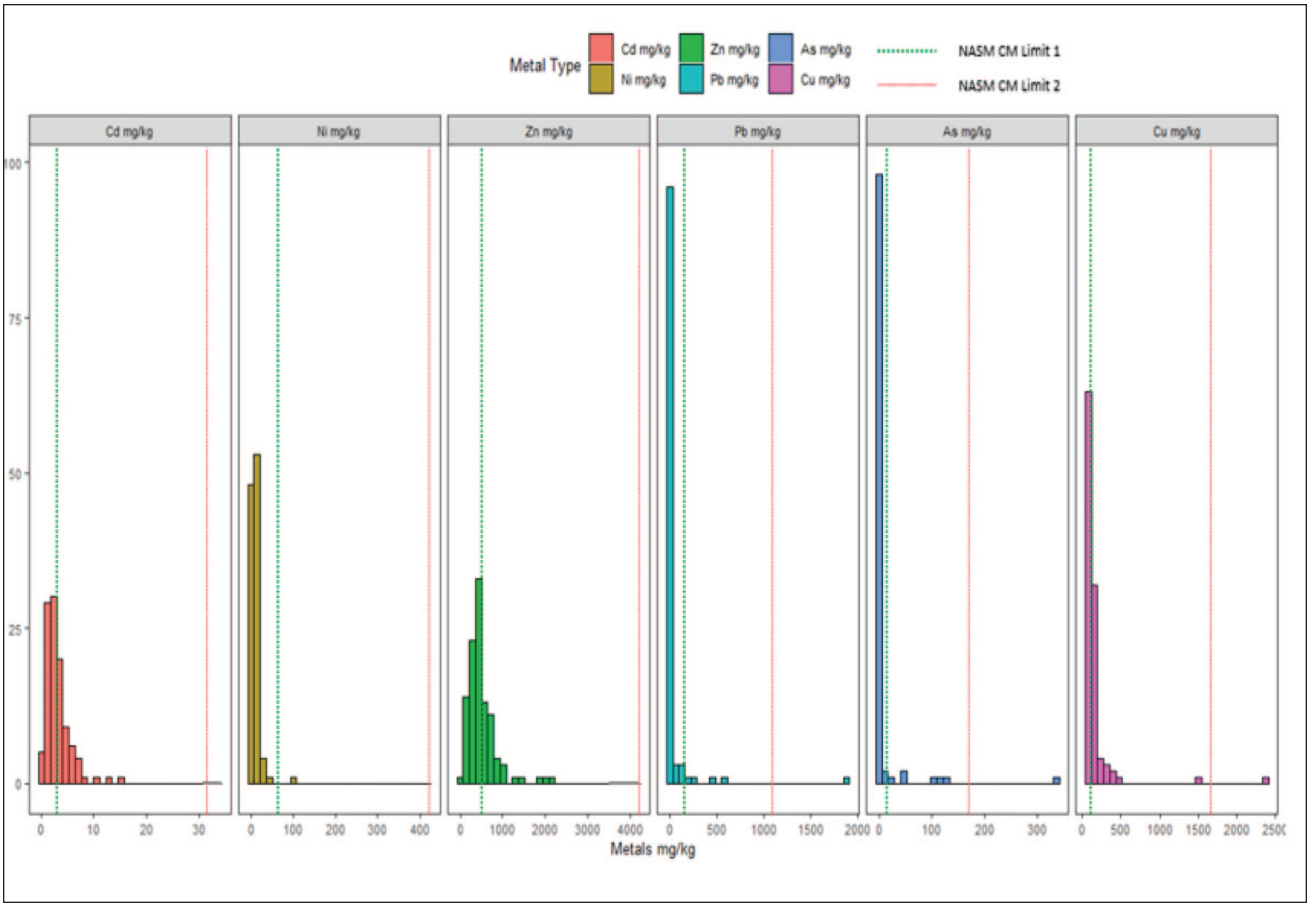

Fig. 2 Unamalgamated Wood Ash Chemistry – Metals; Distribution of Cd, Ni, Zn, Pb, As and Cu concentrations in unamalgamated non-industrial wood ash (n=107) samples, and Ontario Regulation 267/03 of the Nutrient Management Act limits for unrestricted (CM1) and restricted (CM2) use of wood ash for land application as a non-agricultural non-aqueous source material are indicated with dashed green and red lines, respectively.

centrations were also highly variable with coefficients of variation for most metals > 100% (Table 2). Importantly, concentrations of Pb, As and Cu were below the restricted (CM2) limits in every test sample except three (out of 107 that were re-run), whereas all 107 samples had Zn, Ni, and Cd concentrations under restricted (CM2) limits (Fig. 2). However, of the 107 samples analyzed, Zn (35%), Ni (38%), Cd (6%), Pb and As (7%) had concentrations above the unrestricted (CM1) limits. Of the trace metals, Cu most consistently exceeded guideline thresholds and in 63% of the samples exceeded the unrestricted (CM1; 100 mg kg^{-1}) limit (Table 1).

Spearman's correlation heat matrices indicated significant positive relationships among macronutrients (Ca, Mg, P, K), but metals were not significantly correlated with the exception of B, a critical plant micronutrient (Fig. 3). There were also some significant relationships among metals. Pb, Zn, Cd, Al, Fe, and Cu were positively correlated (Fig. 3). In contrast, As was negatively correlated with Al and Fe (Fig. 3).

Non-industrial amalgamated wood ash mixture chemistry

Amalgamated wood ash mixtures generally exhibited greater consistency in their chemical composition compared with unamalgamated ash, and typically had coefficients of variation well below 100%. All test samples fell below the CM2 regulated metals restriction limits established by NASM guidelines; however, some differences among the three mixtures were observed (Table 3). Ash pH and Cu concentrations were significantly lower for amalgamated ash mixture C compared with mixtures A and B. Amalgamated ash mixture B contained lower concentrations of Pb than mixtures A and C, and concentrations of Fe varied among all three sample sets. Average concentrations of K and Ca were higher for all amalgamated ash mixtures than reported ranges for industrial ash. Mean concentrations of Cd, Cu, and Ni were lower than the reported ranges for fly ash, while As and Zn levels were above the ranges for bottom ash but were within the ranges for fly ash (Table 3).

Concentrations of Cd, As, Ni, and Pb in all amalgamated ash mixtures were also under unrestricted (CM1) limits. In contrast, similar to the unamalgamated samples, a high proportion of samples from the three mixtures exceeded CM1 limits for Zn and Cu. For the amalgamated ash mixture A, 80% of the samples exceeded CM1 limits for Cu while 50% exceeded CM1 Zn limits. In comparison, 90% of amalgamated ash mixture B samples exceeded CM1 Cu limits and 50% exceeded CM1 Zn limits. For amalgamated ash mixture C, 60% exceeded CM1 Cu limits and one sample exceeded CM1 Zn limit. Additionally, mean metal concentrations for As, Cu, Pb, Ni, Zn for all three mixtures did not exceed restriction limits set out for five European countries for ash

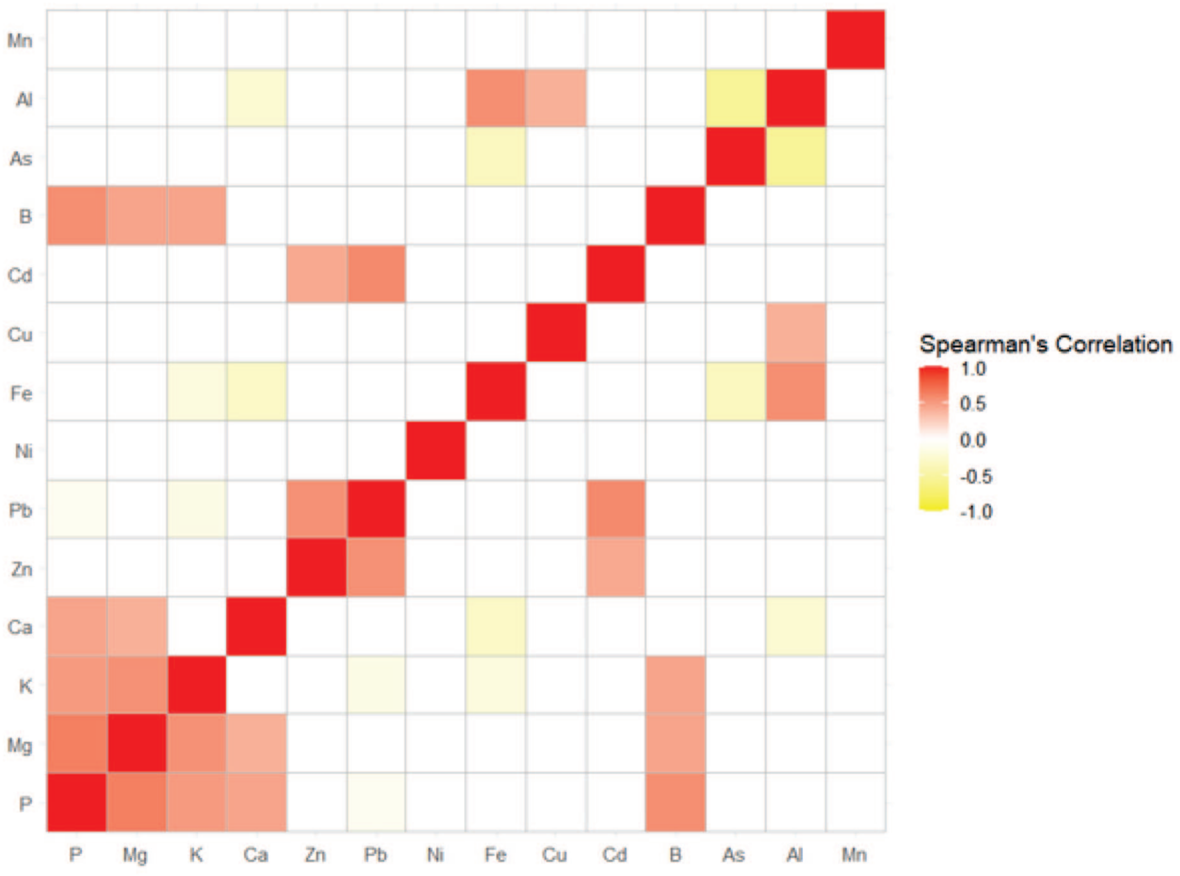

Fig. 3 Unamalgamated Wood Ash – Spearman's Correlation Heat Matrix; Spearman's correlation matrices of unamalgamated non-industrial wood ash samples collected from residents of Muskoka, Ontario (n = 107). White boxes or those not included were not significant at $p < 0.05$. Significance shown at 0.05*, 0.01**, and 0.001***

application to forest soils. The one exception was the average Cd concentration exceeded Germany's restriction values (Table 4). With respect to Canadian legislation, mean ash metal concentrations of all three samples were below the restriction limits for trace metal in Alberta, British Columbia, Nova Scotia, and Quebec (Table 4).

Non-industrial wood charcoal chemistry

Average charcoal pH and nutrient and metal concentrations tended to be lower than unamalgamated wood ash values, but this was significant only for pH, Ca, and Mg (Table 2). Average pH was 10.8 and mean concentrations for Ca, and Mg were 232 g kg^{-1} and 21 g kg^{-1}, respectively, compared with 11.0, 323.7 g kg^{-1}, and 26 g kg^{-1} for unamalgamated ash. Average levels for Cd, Ni, Pb, Cu, Zn, Fe, and Al were lower than for unamalgamated ash except for Mn; however, the differences were not statistically significant.

Compared to unamalgamated ash, metal concentrations in wood charcoal were lower than the NASM unrestricted (CM1) and restricted (CM2) limits. Concentrations for Cd, As, Ni, Pb, and Zn were below unrestricted (CM1) limits for all samples. Cu levels were the exception, as 80% of the samples were above the unrestricted (CM1) limit (100 mg kg^{-1}) but all samples were under the Cu restricted (CM2) limits (1700 mg kg^{-1}).

Muskoka residents wood ash survey results

A questionnaire was sent in January 2021 to residents of the Muskoka District who donated their wood ash to the Friends of the Muskoka Watershed. Forty-seven responses were received (Table 5). The residents were asked to provide information on species burnt, parts burnt (i.e., bark, trunk, branches) and furnace type. The respondents indicated they used multiple species and parts of the tree, rather than a single dominant species or tree part. The survey indicated that 70.2% burnt bark, 85.1% trunks and 74.5% branches. Most residents burnt mostly hardwoods such as maple (70.2%), birch (51.1%) or oak (27.7%); about 25% reported using softwood species such as pine, spruce and/or hemlock.

Discussion

This study evaluated the chemical composition of non-industrial wood ash (NIWA) generated from residential wood stoves and furnaces in the District of Muskoka, Ontario.

Table 3. pH, LOI, CNS, nutrient and metal concentrations of 3 amalgamated NIWA mixtures (n=10 each).

	Amalgamated Non-industrial Wood Ash Chemistry												
	Ash Mixture A				Ash Mixture B				Ash Mixture C				
	Mean	SD	Cv (%)	SE	Mean	SD	Cv (%)	SE	Mean	SD	Cv (%)	SE	P value
pH	**13.5** ^{a}	–	–	–	**13.3** ^{a}	–	–	–	**11.5** ^{b}	–	–	–	*<0.001*
LOI	3.5	0.8	22.9	0.3	5.8	1	17.7	0.3	5.6	1	17.8	0.3	n.s
C (%)	11.6	4	34	1.3	8.8	0.8	9	0.3	9.1	0.9	9.8	0.3	n.s
N (%)	0.2	0.2	100	0.1	0.1	NA	NA	NA	0.1	NA	NA	NA	n.s
S (%)	BDL	NA	NA	NA	BDL	NA	NA	NA	BDL	NA	NA	NA	n.s
Ca ($g.kg^{-1}$)	305	15.2	5	4.8	273	48.4	17.7	15.3	294	46.4	15.7	14.7	n.s
Mg ($g.kg^{-1}$)	24.2	2.5	10.7	0.8	22.1	3.5	16	1.1	22.5	3.8	16.9	1.2	n.s
K($g.kg^{-1}$)	109	13	12	4.1	112	21.7	19.3	6.9	104	20.3	19.5	6.4	n.s
P ($g.kg^{-1}$)	8.8	1.2	13.4	0.4	7.9	1.2	15.1	0.4	7.8	1.2	15	0.4	n.s
Cd ($mg.kg^{-1}$)	2.7	0.4	14.4	0.1	2.5	0.6	24.7	0.2	2.6	0.4	15.2	0.1	n.s
As ($mg \cdot kg^{-1}$)	3.9	6	153	1.9	3.1	7.4	237	2.3	3.7	5.7	153	1.8	n.s
Ni ($mg \cdot kg^{-1}$)	10.5	3	30.4	0.9	8.8	2	22.5	0.6	7.9	1.5	19.1	0.5	n.s
Pb ($mg \cdot kg^{-1}$)	**24.3** ^{a}	17	71.8	5.4	**12.7** ^{b}	3.8	30.3	1.2	**48.5** ^{a}	64.2	132	20.3	*0.015*
Cu ($mg \cdot kg^{-1}$)	**140** ^{a}	41.9	29.8	13.2	**154** ^{a}	92.1	59.8	29.1	**106** ^{b}	15.2	14.3	4.8	*0.026*
Zn ($mg \cdot kg^{-1}$)	523	109	20.9	34.5	516	151	29.3	47.8	439	61.5	14	19.4	n.s
Mn ($mg \cdot kg^{-1}$)	6306	683	10.8	216	6837	1023	15	324	6329	1215	19.2	384	n.s
Al ($mg \cdot kg^{-1}$)	4075.9	1031.8	25.3	326	3044.6	1019.2	33.5	322	3933.07	1261.5	32.1	399	n.s
B ($mg \cdot kg^{-1}$)	235.58	46	19.5	14.5	239.3	49.9	20.9	15.8	213.15	30.17	14.2	9.5	n.s
Fe ($mg \cdot kg^{-1}$)	**2793** ^{a}	1150	41	364	**1322** ^{b}	528	39.9	167	**1872** ^{ab}	634	33.9	200	*0.001*

Significant differences among the 3 amalgamated ash mixtures were determined by the Kruskal Wallis test with a post hoc Dunn's test indicated with different letters. P value significant at 0.05, (Bolded within the table), BDL below detection limit, S Detection limit – 181 Peak Area, NA not applicable, n.s. not significant

Table 4. Restriction values for trace metal concentrations of biomass ash for forest soil application in 5 European countries and 4 Canadian provinces (Hannam *et al.*, 2016 and 2018), compared with mean metal concentrations of three amalgamated ash mixtures tested in this study.

Ash Metals	AB Wood Ash	BC Soil Amendment	NS Biosolids		Qc Fertilizing Residual		Denmark	Finland	Germany	Lithuania	Sweden
			Class A	Class B	C1	C2					
As mg kg^{-1}	–	75	13	75	13	41	–	40	40	30	30
Cd mg kg^{-1}	46	20	3	20	3	10/15	20	25	1.5 $^{A/B/C}$	30	30
Cu mg kg^{-1}	–	2200	400	760	400	1000	–	700	–	400	400
Pb mg kg^{-1}	–	500	150	500	120	300	250	150	150	300	300
Ni mg kg^{-1}	–	180	62	180	62	180	60	150	80	70	70
Zn mg kg^{-1}	5500	1850	700	1850	700	1850	–	4500	–	700	7000

Amalgamated ash mixtures' mean metal concentrations that failed to meet requirements are highlight as A = Amalgamated ash mixture A, B = Amalgamated ash mixture B, and C = Amalgamated ash mixture C.

NIWA was rich in macronutrients (Ca, Mg, K and P), and metal levels were within NASM guidelines for soil application. There was considerable chemical variability amongst individual samples, however, once homogenized, the chemical composition of the bulk sample was relatively consistent and metal concentrations were under CM2 levels. Average metal concentrations for the homogenized ash mixture were also within the restriction limits established by four Canadian provinces and five European countries where wood ash is used as a soil amendment.

Unamalgamated non-industrial wood ash

Wood ash samples in this study contained concentrations of Ca, Mg, K, and P that were at the higher end of reported industrial ash levels. For example, Hannam *et al.* (2018) examined the chemical properties of fly ash and bottom ash generated in bioenergy plants across Canada and reported total Ca concentrations between 92.2 g kg^{-1} and 247.9 g kg^{-1} for fly ash and 3.9 g kg^{-1} to 211.0 g kg^{-1} for bottom ash, while this study range was 141.6 g kg^{-1} to 679.9 g kg^{-1}. The high levels of macronutrients found in NIWA can be attributed to the

Table 5. Survey responses to wood ash questionnaire, from the residents of Muskoka District, respondents indicated tree species commonly used along with parts of tree burned, (n = 47).

Tree species	Total occurrence per survey respondent	Percent %
Ash (*Fraxinus spp.*)	3	6.4
Basswood *(Tilia americana)*	1	2.1
Beech (*Fagus grandifolia*)	10	21.3
Birch (*Betula spp.*)	24	51.1
Cherry (*Prunus spp.)*	4	8.5
Hemlock (*Tsuga canadensis*)	3	6.4
Iron wood *(Ostrya virginiana)*	3	6.4
Maple (*Acer spp.*)	33	70.2
Oak (*Quercus spp.*)	13	27.7
Pine (*Pinus spp.*)	7	14.9
Poplar *(Populus spp.)*	3	6.4
Spruce (*Picea spp.)*	2	4.3
Hardwoods	**10**	**21.3**
Softwoods	**3**	**6.4**
Tree parts burned		
Bark	33	70.2
Trunk	40	85.1
Branches	35	74.5

chemical composition of the species, as well as the parts of the tree burnt (Pitman 2006; Deighton and Watmough 2020). Hardwoods generally contain higher levels of macronutrients than softwoods, while tree bark tends to be rich in Ca (Pitman 2006). The survey results of the Muskoka residents indicated that ash samples were dominated by hardwoods. Higher levels of K in NIWA may also be attributed to lower temperatures associated with home wood stoves, resulting in lower volatilization losses of K compounds as volatilization occurs around 1300°C (Naylor and Schmidt1986).

Metal concentrations in individual samples varied considerably. Such dissimilarities are consistent with findings on NIWA. Non-industrial wood ash originates from various sources and there is inconsistency in feedstock, burn temperatures, and tree parts used (Azan *et al.* 2019), factors which collectively contribute to ash chemical composition (Pitman 2006). Metal concentrations vary significantly among tree species in Muskoka (Landre *et al.* 2010). Deighton and Watmough (2020), for example, reported that yellow birch ash had higher concentrations of metals compared with sugar maple and white pine ash. In this study, Zn concentrations in yellow birch ash were 12 times higher than sugar maple and white pine ash, while As and Pb concentrations were nine and six times greater, respectively.

Nevertheless, NIWA metal concentrations were low compared with industrial wood ash (IWA) and were mostly within NASM guidelines. For example, Cd and Ni levels reported by Pitman (2006) ranged between 6–40 and 20–100 mg kg^{-1}, respectively, for IWA, while ash samples in our study had average Cd and Ni concentrations of 3.0 and 10.5 mg kg^{-1} respectively. Ash metal concentrations for most of the samples tested were under CM2 NASM guidelines for restricted use with a few (1 sample/analyte) notable exceptions for Pb, As, and Cu. Concentrations of Cd, Zn and Ni were under CM2 limits for all samples. Exceedance of CM1 limits was, as expected, more frequent and was observed in decreasing concentrations within Cd, Zn, As, Pb, and Ni. The trace metal with the highest number of samples exceeding the CM1 limit was Cu. These results are similar to the findings of a NIWA study conducted in Muskoka by Azan *et al.* (2019) who analyzed 10 samples and reported average Cu concentrations along with Zn to be slightly above CM1 limits. Elevated levels of Cu in NIWA could be a result of our samples containing a higher percentage of species rich in this trace metal such as yellow birch. Deighton and Watmough (2020) also reported yellow birch ash levels for Cu, Cd, and Zn to be above CM1 guidelines. Yellow birch ash contained two to three times more Cu than ash from sugar maple or white pine. Additionally, Cu volatizes at temperatures above 1000°C (Misra *et al.* 1993), while most home stoves commonly burn below 1200°C (Pitman 2006).

Some metals exhibited a significant relationship with each other. Pb, Zn and Cd, and Al, Fe, and Cu were positively correlated, while As was negatively correlated with Fe and Al. However, ash metals were not correlated with macronutrients, indicating that ash rich in nutrients does not have similarly high metal concentrations. Ash B concentrations showed a positive correlation to macro-nutrients such as Mg, K, and P, indicating that NIWA can be a source of these essential micronutrients. This is possibly due to the variation in the chemical composition of the tree species and parts of the tree used, along with feedstock growing conditions (Pitman 2006; Landre *et al.* 2010). For example, high levels of Mn, Ni and Zn have been found in foliage (Landre *et al.* 2010), while Ca concentrations are highest in bark (Elliott and Mahmood 2006). Therefore, ash samples which originate mainly from bark will be higher in Ca while ash from burnt foliage will have higher levels of metals.

Concentrations of Mg in NIWA in this study were generally consistent with previously reported industrial ash concentrations (Pitman 2006), however they were higher than average concentrations reported by Deighton and Watmough (2020). In addition, large variations in Zn among ash samples from sugar maple, white pine and yellow birch have been reported (Deighton and Watmough 2020). Similarly, Naylor and Schmidt (1986) reported large variations in Zn in ash from home stoves using only hardwoods. Metal bins used for storage of NIWA, foreign objects (e.g., metal nails) found in the donated samples, and tools for the homogenization of ash, may also contribute to trace metal levels through leaching. Leaching of metals such as Zn, Fe, Cr, and Pb can occur overtime through corrosion and/or erosion (Verissimo *et al.* 2006; Gonzalez *et al.* 2013; Ghada *et al.* 2015). Additionally, trace metal concentrations were generally much lower than reported by Smolka-Danielowska and Jablonska (2022). These inconsistencies may be attributed to different feedstock locations and or species used compared with this study. For example, only 25% of survey respondents indicated the use of beech (21.3%) or spruce (4.3%). Nevertheless, these differences highlight the importance of variation in feedstock quality.

Amalgamated non-industrial wood ash

The three amalgamated non-industrial wood ash mixtures were similar in chemical composition and only pH, Pb, Cu and Fe differed among subsets and these differences were relatively small. This suggests that homogenizing smaller batches of individual samples will not result in large differences in the chemical composition of ash applied to soils. Additionally, macro and micro element concentrations in all three amalgamated mixtures were below the CM2 NASM limits, but a few were above CM1 restriction limits for Pb, Cu and As. Moreover, when compared with trace metal restrictions set out in five European countries for ash application to forest soils, our samples mean metal concentrations were below limits, except for Cd that exceeded the limit in Germany which has stringent national guidelines. Additionally, mean ash metal concentrations in the three mixtures were below the restriction limits for trace metal concentrations in Alberta, British Columbia, Nova Scotia and Quebec, indicating that the NIWA used in this study meets trace metal guidelines for soil application in locations within Canada and internationally.

Municipalities considering implementing a NIWA recycling program can ensure bulk mixtures fall within regulatory guidelines by testing individual batches and excluding any which test above the regulatory limits. Additionally, municipalities may consider creating guidelines for residents who wish to donate ash on acceptable species to use and which to avoid. Use of species such as yellow birch that are known to have high concentrations of trace metals can be restricted, thus further limiting trace metal contamination.

Non-industrial wood charcoal

To our knowledge, this is the first study to evaluate the chemical composition of wood charcoal fractions of NIWA in Canada. Charcoal typically had lower metal concentrations and higher C content than wood ash but only pH, and Ca and Mg concentrations were significantly lower in charcoal. Concentrations of most macronutrients were within the range for reported industrial wood ash, except for K levels which were higher in our samples. Research has shown that metal concentrations in ash decrease as particle size increases (James *et al.* 2014; Smołka-Danielowska and Jabłońska 2022), and although mean metal concentrations were lower in wood charcoal compared with finer wood ash, a significant difference was only observed in As levels. Concentrations of all regulated metals were under CM1 limits, except for Cu, where most of the samples tested were above CM1 but still under CM2 limits. These results suggest that wood charcoal metal concentrations may not differ significantly from finer NIWA and may not need to be removed from ash mixtures before soil application.

Given the high C values in charcoal, it may qualify as a Class C biochar under existing classification systems (International Biochar Initiative 2015), and it was noted that metal and nutrient concentrations of charcoal were generally higher than published elemental concentrations of wood derived biochar (Yargicoglu *et al.*2015; Berek and Hue 2016). Wood-derived biochar Ca concentrations have been reported to have been reported to fall below 80 g.kg^{-1} while average Ca concentration in this study was 232 g.kg^{-1} (Yargicoglu *et al.* 2015). Similar to wood ash, the variation in biochar chemistry in the literature reflects the variations in feedstock (Vaughn *et al.* 2015; Gezahegn *et al.* 2019; Arous *et al.* 2021) which likely accounts for the differences observed. It should also be noted that recent field trials suggest that high-carbon wood ash biochar that meets existing standards for metal content can nevertheless have pronounced negative effects on tree regeneration due to metal toxicity (Bieser and Thomas 2019). Therefore, although charcoal in this study contained lower concentrations of wood ash, further studies are warranted.

Conclusions

Potentially high trace metal concentrations, large variability in feedstock chemistry, and poor understanding of the effects of homogenization on ash chemical composition are some of the barriers associated with the use of NIWA as a forest soil amendment. This study tested NIWA samples collected from residential wood stoves in Muskoka, Ontario and found that NIWA was rich in important macronutrients including Ca and K, and low in most trace metals. Furthermore, nutrient concentrations were not correlated with metal concentrations, indicating that high levels of nutrients may not always be a good indicator of ash metal concentrations. Although there was substantial variability in the chemistry among individual samples, concentrations of most elements were within a relatively narrow range. Ash mixtures, amalgamated in the field, were relatively homogenous in chemical composition and metal concentrations were generally below NASM regulation guidelines. Only Cu and Zn exceeded CM1 guidelines consistently, however these levels were always below restricted application limits (CM2). This study suggests that once homogenized, non-industrial wood ash chemical composition is within the regulatory guidelines set out by several European countries and Canadian provinces, and thus meets the requirements as a forest soil amendment and can be incorporated into wider forest management policies. However, it is recommended that large scale research be conducted to test for differences in ash chemical variability as it relates to differences in feedstocks and geographical locations. In addition, municipalities considering the implementation of a NIWA recycling program should routinely test composite samples to ensure they continue to meet NASM regulations.

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