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RESEARCH ARTICLE

Limited effect of wood ash application on soil quality as indicated by a multisite assessment of soil organic matter attributes

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Abstract

In Canada, the combustion of forest biomass for bioenergy production has been increasing with an associated increase in residual wood ash. Wood ash is typically landfilled as waste but there is growing interest in applying wood ash to the soils of commercial forests. Ideally, wood ash supplies nutrients that may have been removed through biomass harvesting, increases soil pH, which improves nutrient availability, and potentially improves site productivity, but there is also potential for detrimental effects, such as toxicity, that impair soil functions. The objective of this study was to investigate the effects of wood ash application on soil organic matter attributes at eight experimental sites across Canada that are examining the effects of wood ash application on site fertility, productivity, and soil biodiversity. Wood ash application had an effect on total carbon (TC) and total nitrogen, microbial biomass carbon (MBC), hot water extractable carbon (HWEC), mineralizable C, sand size C, and HWEC and MBC normalized to TC, but changes were typically restricted to single sites or differed in their direction, that is, positive or negative. Based on the limited and inconsistent effects of ash on established

indictors of soil quality measured in this study, there does not appear to be any advantageous or detrimental effects of adding wood ash to forest soil quality.

KEYWORDS

bioenergy, bottom ash, fly ash, forest soil, labile carbon, microbial biomass, soil carbon, soil quality

1 | INTRODUCTION

Although biomass bioenergy accounts for only 3% of renewable electricity production in Canada, the contribution of forest biomass to centralized energy production used outside of mills is increasing (Natural Resources Canada (NRCan), 2017). The forest industry has been producing bioenergy through the combustion of residual materials (lignin/black liquor and woody residuals) to support their operations for nearly one century (Hannam et al., 2017) and wood wastes from harvesting, milling and manufacturing are increasingly being used as biomass fuel for bioenergy production (NRCan, 2020). Biomass removal on an industrial scale can contribute to the degradation of the site productivity over successive rotations (Van Bich et al., 2018) and result in increased soil acidity, which may negatively impact soil biodiversity (Jacobson et al., 2014; Reid & Watmough, 2014). Generally, the impact on nutrient removal and ecosystem function is proportional to the intensity of harvesting and the degree of biomass utilization (McFee & Kelly, 1995; Paré et al., 2002). Therefore, increases in the intensity of biomass removal with increasing demand for forest bioenergy feedstock has been an area of concern due to the potential for increased export of nutrients, soil acidification, and declines in soil quality.

Bioenergy production through combustion of forest biomass creates wood ash as a by-product, and 63% of this wood ash is disposed into landfills in Canada (Hannam et al., 2018). Wood ash is alkaline with a pH ranging from 8 to 13 and contains micro and macronutrients essential for plant growth (Pitman, 2006). Applying wood ash to forest soils can replace nutrients (e.g., phosphorous [P], calcium [Ca], magnesium [Mg], potassium [K]) removed during timber harvesting and thus increase soil base saturation and alkalinity (e.g., Hannam et al., 2019; Reid & Watmough, 2014). In Europe, application of wood ash to the soil is encouraged, particularly on acidic and nutrient poor soils, to mitigate nutrient deficiencies created by biomass harvesting (e.g., Karltun et al., 2008; Titus et al., 2021). Application of wood ash to soils does come with concerns around trace element concentrations, impacts on ground vegetation, alterations in microbial processes, and changes in greenhouse gas emissions that are not fully understood (e.g., Huotari et al., 2015). Applying wood ash to forest soils in Canada does, however, present an opportunity to gain value from potentially increased forest productivity while enhancing the environmental sustainability of harvesting operations (Hannam et al., 2017). Furthermore, diverting wood ash from landfill for application to forest soils can reduce disposal costs and extend lifespans of landfills (Gaudreault et al., 2020; Hope et al., 2017).

Although our understanding of the effects of wood ash application on soil pH, base cation dynamics, tree growth and understory vegetation is increasing, comparatively less is known about the effects on soil organic matter (SOM) (Reid & Watmough, 2014). SOM is a key attribute in most definitions of "soil quality" because it influences the physical, chemical, and biological properties and processes in the soil (Doran & Parkin, 1994; Haynes, 2005; Larson & Pierce, 1991). Changes in soil quality, which is the capacity of a soil to function for specific land uses or within ecosystem boundaries, in response to management have been gaining attention because of concerns of soil degradation and sustainability (Haynes, 2005). Although soil quality (and soil health) is inherently a metaphor that cannot be fully measured directly (Janzen et al., 2021), attributes of soil quality can reveal how land management practices are impacting soil functions in time and space (Karlen et al., 1997).

Wood ash contains residual post-combustion organic matter where the carbon (C) and nitrogen (N) content is highly variable (Pitman, 2006). In addition to directly adding C to the soil, the application of wood ash may result in increased productivity of vegetation through increased soil pH, which typically increases the availability of nutrients and decreases the solubility of aluminum, and available nutrients (Reid & Watmough, 2014), thereby further increasing organic matter inputs via litterfall and roots. Conversely, the application of wood ash to soil can create conditions which enhance decomposition of native SOM pools and the wood ash itself (Hansen et al., 2016; Saarsalmi et al., 2004). Results from past studies examining the effects of wood ash application on SOM have been mixed. In a recent meta-analysis comparing wildfire and wood ash application on soil properties, Hannam et al. (2019) reported declines in C and N concentrations in both the forest floor and mineral soil following wood ash application. However, other studies have reported no effect on

soil C or N (e.g., Gomoryova et al., 2016; Ventura et al., 2019). Wood ash application directly adds C to soils and can enhance organic matter returns to the soil through increases in tree growth but to the best of our knowledge, no studies have reported significant increases in soil C or SOM content.

Detecting changes in SOM associated with land management practices is often challenging because the magnitude of the change is often small relative to the total soil SOM pool, and the intrinsic spatial variability within a site makes change detection difficult (Conant et al., 2003). Fractions (i.e., attributes) of SOM that represent small but dynamic C pools that are more sensitive to management may reveal changes not apparent from measurements of total C (TC) and N (TN) alone (e.g., Ghani et al., 2003; Gregorich et al., 2006). These fractions are also often robust indicators of critical soil functions, such as nutrient availability, structure and stability, and biological activity (Cardoso et al., 2013; Gregorich et al., 1994; Reeves, 1997), which are determinants of soil quality (e.g., Bünemann et al., 2018).

Identifying sensitive attributes or indicators to assess changes in soil quality with land management practices has been documented in the literature for some time (e.g., Gregorich et al., 1994; Larson & Pierce, 1991; Reeves, 1997). A minimum set of indicators that describes soil quality have included: TC and TN, C:N, mineralizable C, un-complexed organic C, microbial biomass C (MBC), and carbohydrate/hot water extractable C (Bünemann et al., 2018; Gregorich et al., 1994; Haynes, 2005). The ratio of C to N tends to reflect the capacity of the soil to store and recycle nutrients, with a narrowing of the C:N interpreted as being an indicator of microbial decomposition of SOM (Gregorich et al., 1994). Mineralizable C, estimated by the CO_2 released through the mineralization of SOM by the microbial community, is an indicator of the metabolic activity of the decomposer community, that, in turn influences SOM turnover and nutrient release (Gregorich et al., 1994). Un-complexed organic matter, such as the >53 um organic matter fraction (i.e., the sand size fraction), is a readily available pool of plant residues and soil biota in various stages of decomposition. This fraction, which represents the portion of the SOM pool that contains recognizable plant structures and soil biota, is a responsive indicator of change because it is sensitive to changes in the rates of input of plant residues and their persistence (Gregorich et al., 2006). Microbial biomass C is a gross estimate of the size of the microbial community, is an indicator of the soil's capacity to store and recycle nutrients, and responds quickly to changes in environmental conditions. Microbial biomass C is often expressed relative to TC or the amount of C mineralized to indicate microbial efficiency (e.g., Rice et al., 1997). Carbohydrate C, or C extracted by incubating SOM for 16h at 80°C, influences the formation and stabilization of soil structure (Ghani et al., 2003). Through concurrent measurements of these more sensitive soil C attributes, we seek to better describe the effects of wood ash application on forest soil quality, which is key to predicting site productivity and to ensure environmental sustainability. To date, we are not aware of any study that has taken this approach to the assessment of wood ash application effects (positive or negative) on soil quality. This work compliments ongoing work at each of the study sites evaluating the effects of wood ash application on site fertility, productivity, and soil biodiversity.

The study objectives were to determine the effects of wood ash application (e.g., different wood ash sources and quantities of application) on a suite of SOM attributes at eight sites across Canada and to identify sensitive attributes that can be used as robust indicators of change in forest soil quality in response to wood ash application.

2 | MATERIALS AND METHODS

Soil samples were collected from eight wood ash experiments across a broad geographic area in Canada (Table 1, Figure 1) in 2017 (June 7–July 25). Detailed site descriptions and study designs are described in Emilson et al. (2018). Wood ash characterization was carried out prior to the initiation of all field trials, and the wood ash chemistries varied among the sites due to different feedstock and combustion temperatures (Emilson et al., 2018). Although wood ash was often applied at similar rates across sites (Table 2), the wood ashes differed in their elemental application rates and liming potential (Emilson et al., 2018). Wood ash was applied by hand at all sites, except the Eastern Townships site where it was applied using a mechanical spreader. Within each plot, the forest floor (LFH) was carefully removed by hand from within a 15 cm × 15 cm square frame at one location. At the Eastern Townships and Island Lake sites, the forest floor was separated into the litter moss (LM) and forest floor (FH) layers. The FH and LFH layers are later referred to as the forest floor, whereas the individual litter-moss layer is referred to as the LM layer. Three mineral soil samples were collected within this square footprint using PVC pipes that were 5 cm in diameter and 20 cm in length and later composited. Samples were immediately frozen at −20°C prior to analysis, which is a recognized approach for archiving soil samples (e.g., Kuhnel et al., 2019).

2.1 | Laboratory analyses

A subsample of each frozen sample was air dried and sieved to 2 mm (mineral soil samples) or 4 mm (forest floor

TABLE 1 Location and site attributes of sites included in this study

Site	Province	Coordinates	Elevation (masl)	MAP (mm)	Jan. Min T (°C)	July Max T (°C)	Soil type	Soil texture
Aleza Lake N	BC	54.08° N; 122.09° W	660	714	−12.2	23.1	Gray Luvisol; Luvic Gleysol	Silty clay loam to clay
Aleza Lake S	BC	54.07° N; 122.08° W	670	719	−12.2	22.9	Gray Luvisol; Luvic Gleysol	Silty clay loam to clay
Mistik	SK	53.7° N; 108.2° W	691	431	−22.2	22.4	Orthic Gray Luvisol	Clay loam, with pockets of sandy loam
Pineland	MB	49.5° N; 96.1° W	320	635	−22.5	25	Brunisol	Sand
25th Sideroad	ON	48.4° N; 89.4° W	215	694	−19.2	23.8	Orthic Eutric Brunisol	Sandy loam
Island Lake	ON	47.7° N; 83.6° W	455	927	−20.6	23.1	Eluviated Dystric Brunisol	Sand to sandy loam
Haliburton	ON	45.3° N; 78.6° W	375	1074	−17	24.7	Orthic or Eluviated Dystric Brunisol	Sandy loam
Eastern Township Sugar Maple (*Acer saccharum* Marsh.)	QC	45.57° to 45.69° N; 71.25° to 71.86° W	270–400	1264	−16.6	23.7	Orthic Humo-Ferric; Ferro-Humic Podzol	Sand to loamy sand to sandy loam

Note: Soil classification according to the Canadian System of Soil Classification (Soil Classification Working Group, 1998).

[organic] samples) for optimal homogenization. Moisture contents were determined on the field moist and air-dried samples by drying to a constant weight at 105°C. All methods described herein are adopted from Carter and Gregorich (2008). A representative fraction of air-dried sample was ground in a SPEX 8000 M Mixer/Mill to pass through a 53-um sieve to further homogenize the sample. Concentrations of TC and TN in ground samples were determined using flash combustion (1100°C) and infrared (TC) and thermal conductivity (TN) detection in an elemental analyzer (vario EL cube, Elementar). Physical fractionation on the basis of particle size was carried out to determine TC and TN associated with the sand size fraction of the mineral soil samples. Approximately 25 g of air-dried and sieved mineral sample, 125 ml of distilled water and 30 borosilicate glass beads of 5 mm diameter were added to a 250-ml centrifuge bottle. Bottles were shaken on a mechanical shaker for 16.5 h to disperse soil aggregates. Samples were then wet-sieved using 250-μm sieve, which separated the sand from the silt/clay fraction. The sand and silt/clay fractions were oven dried at 60°C and then ground to powder (about 60 μm or smaller) in the SPEX 8000 M Mixer/Mill. TC and TN associated with the sand size fraction and silt/clay size fractions were determined using the Elementar vario EL cube.

Hot water extractable C (HWEC) was determined following Ghani et al. (2003). Approximately 20 g of mineral soil and 5 g of forest floor (sieved and air-dried samples) were combined with a 0.05 M $CaCl_2$ solution at a soil to solution ratio of 1:2 and 1:10, respectively. The mixture was heated in a water bath at 80°C for 16 h and was then extracted using a microfiltration unit that contained 0.45 μm Whatman membrane filter. Dissolved organic carbon (DOC) concentrations were determined using a SKALAR San++ Automated Wet Chemistry Analyzer, equipped with UV detector and DOC chemistry module (311-000).

Laboratory incubation under specific conditions measures the combined respiration rate of all active organisms present in a soil sample (Carter & Gregorich, 2008). Each soil layer was incubated separately. Approximately 30 g of mineral soil and 15 g of forest floor or LM layer (air-dried equivalent weight) were saturated with distilled water. Moisture content was then adjusted to 60% water holding capacity by applying vacuum at −60 kPa, as it is within the optimum range of moisture content for mineralization (Papendick & Campbell, 1981). Samples were pre-incubated for 5 days in an incubator maintained at 24°C to allow for equilibration after preparation. The samples were then transferred to a closed chamber incubation vessel (i.e., a 1-L glass jar with a septum in the lid). The CO2 concentration in the headspace of the jar was determined by extracting approximately 30 ml of gas from

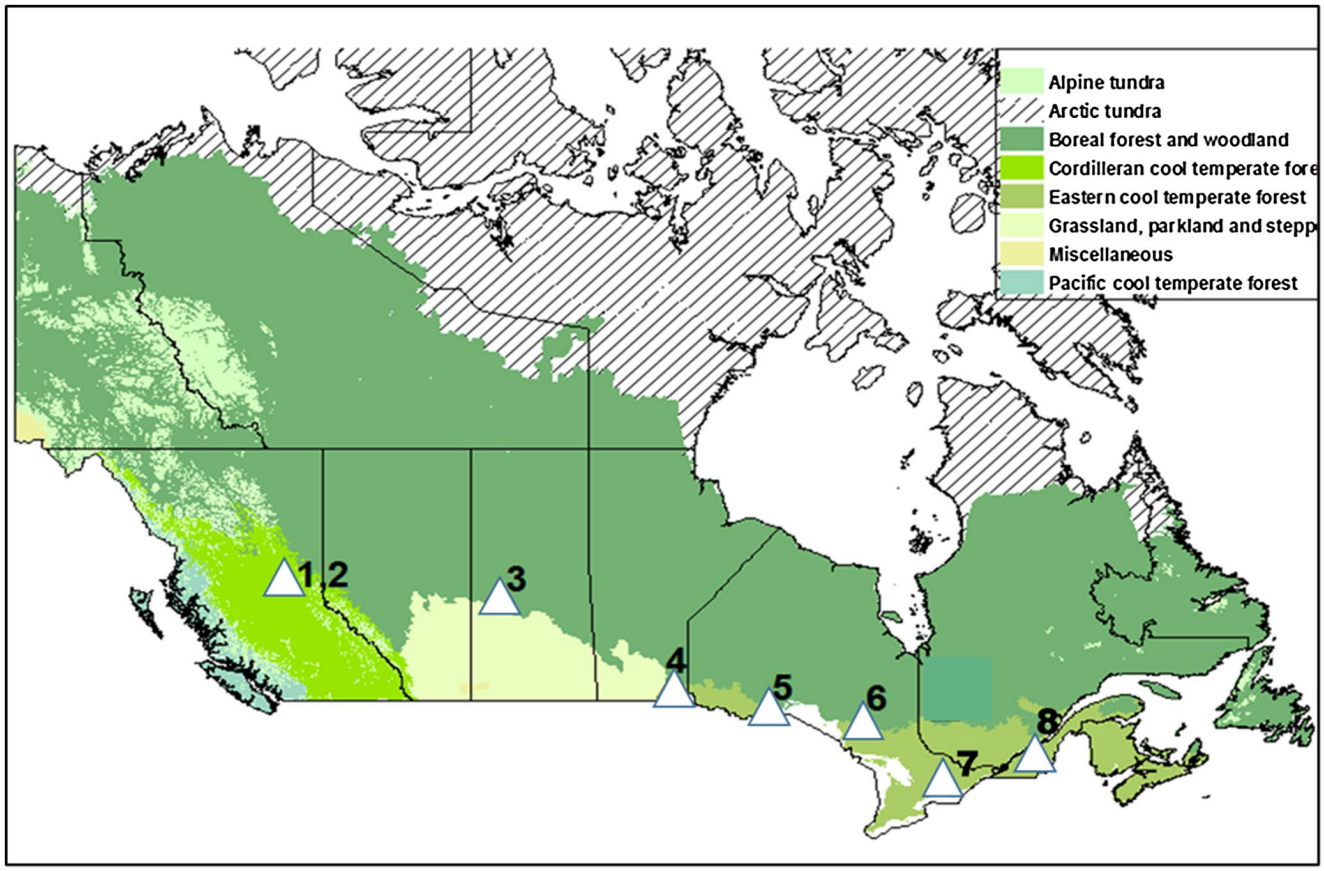

FIGURE 1 Location of the wood ash study sites across Canada. Site names from left to right include (1) Aleza Lake North (northern British Columbia), (2) Aleza Lake South (northern British Columbia), (3) Mistik (Saskatchewan), (4) Pineland (Manitoba), (5) 25th Sideroad (Northwestern Ontario), (6) Island Lake (Northeastern Ontario), (7) Haliburton (North Eastern Ontario), and (8) Eastern Townships (Quebec). The map is shaded by major forested Canadian National Vegetation Classification zones (Canadian National Vegetation Classification [online] 2018)

the headspace using a syringe and measured on an SRI 310 gas chromatograph. The samples in the sealed jars were incubated for 7 days, after which the measurement of CO2 concentration in the headspace was repeated. The difference in the quantity of C-CO2 evolved between day 0 and day 7, along with the dry mass of soil, volumes of water and funnel, and the incubation time were used to determine the rate of C mineralization.

The fumigation-extraction method was used for estimating MBC of the incubated samples. A 0.25 M K2SO4 solution was added to an unfumigated subsample at a soil to solution ratio of 1:3 to 1:5 for the mineral soil and forest floor, respectively. The mixture was shaken on the mechanical shaker for an hour. After shaking, the extract was filtered using Whatman grade 42 filter paper. The other sub-sample was fumigated with ethanol free chloroform. The samples were kept open in a thick-walled glass vacuum desiccator and exposed to a beaker containing 50 ml of CHCl3, then sealed under vacuum and kept in the fume hood in the dark for 24 h. After fumigation, chloroform vapors were evacuated and extraction was carried out using 0.25 M K2SO4 solution at the same ratios as previously discussed. The DOC concentration in the fumigated and non-fumigated extracts was determined using a SKALAR San++ Automated Wet Chemistry Analyzer, equipped with UV detector and DOC chemistry module (311-000). The difference in the DOC concentration between the fumigated and non-fumigated extracts and a k_{ec} factor of 0.45 were used to determine the MBC.

All C fractions were also normalized to TC to examine differences in C quality. The pH of the soil samples was measured in 0.01 M $CaCl_2$ at a soil to solution ratio of 1:2 for mineral soil samples and 1:4 for forest floor samples.

2.2 | Statistical analysis

The data considered here reflect specific combinations of soil type, dominant tree species, wood ash sources and application rates, and stand age at time of ash application.

TABLE 2 Description of installations at each of the eight sites included in this study

Site	Year of application	Wood ash application rates (Mg/ha)	*N*	Wood ash type	Stand age at application (years)	Dominant tree species	Forest floor thickness (cm)
Aleza Lake N	2015	0, 5	3	Bottom (2 types)[a]	18	Hybrid spruce (*Picea glauca* × *engelmannii* Parry × Engelm.), subalpine fir (*Abies lasiocarpa* (Hook) Nutt.)	~5.2
Aleza Lake S	2015	0, 5	3	Bottom (2 types)[a]	24	Hybrid spruce, subalpine fir	~5.2
Mistik	1995	0, 1, 5	3	Bottom	<1	White spruce (*Picea glauca* (Moench) Voss)	5–10
Pineland	2015	0, 1, 5	5	Mixed	<1	Jack pine (*Pinus banskiana* Lamb.)	2
25th Sideroad	2012	0, 1, 10	5	Fly	<1	White spruce	0
Island Lake	2011	1, 0.7, 1.4, 2.8, 5.6	4[b]	Bottom	<1	Jack pine	~10
Haliburton	2013	0, 1, 4, 8	4	Fly and bottom	Uneven	Sugar maple, American beech (*Fagus grandifolia* Ehrh.), eastern hemlock (*Tsuga canadensis* (L.) Carriere), yellow birch (*Betula allegheniensis* Britt.)	5–8
Eastern Township	2015	0, 20[a]	5	Bottom	~60–80	Sugar maple, American basswood (*Tilia americana* L.), American beech, white ash (*Fraxinus americana* L.), butternut (*Juglans cinerea* L.)	10–15

[a]Low carbon (7.3 g total C 100 g^{-1} dry ash) gasifier bottom ash and high-carbon (29.7 g total C 100 g^{-1} dry ash) boiler bottom ash were applied. Wet weight.

[b]Control had five replicates.

Although this represents a comprehensive soil C dataset, that includes multiple soil C indicators by depth, collected from 8 individual sites, we recognize that not all possible treatment combinations are represented at all study site locations. The resulting unbalanced design creates missing cells in the overall matrix of the full model, thereby requiring a staged analytical approach that uses subsets of the full dataset to examine specific questions regarding soil C response to the various factor combinations. This approach allowed us to highlight the dominant effects associated with wood ash application by developing a series of general linear models to address specific ecological questions customized to subsets of the full dataset, described below.

1. *What was the effect on soil C indicators when applying different ash types at comparable applications rates (i.e., low C vs. high C ash) to regenerating conifer stands approaching crown closure (approximately 20 years since stand establishment)?* (Aleza Lake North and South, British Columbia).
2. *Was there a differential response of wood ash application depending on soil texture (fine vs. coarse-textured soils) and the amount of wood ash applied to regenerating stands?* (Fine textured: Mistik, Saskatchewan and 25th Side Road, Ontario; Coarse textured: Pineland, Manitoba and Island Lake, Ontario).
3. *Was there an effect of wood ash application in mature, selection harvested, tolerant hardwood (sugar maple dominated) stands?* (Haliburton, Ontario and Eastern Townships, Quebec).

For these preliminary analyses that combined sites and similar ash application rates, we further considered any main factor effects and interaction with $p < 0.20$. Within each of these analyses, second- stage analysis was performed to better describe these differential (sometimes site specific) responses. Here, we highlighted significant effect using $p < 0.05$.

The normality of residuals was tested using the Anderson–Darling test and the homogeneity of variance was verified by Levene's test ($p > 0.05$). Tukey's honest significant difference was performed as post hoc following significance with an α of 0.05 to assess the difference between means of C concentrations with each treatment. If the distribution in the observations were non-normal, the data were log-transformed using log function in R to meet the assumptions of the ANOVA model. ANOVAs were performed on log-transformed values where the transformation satisfied the normality assumption of ANOVA. Nonparametric analyses using the Kruskal–Wallis test were conducted when transformation did not normalize the data. The results obtained from the Kruskal–Wallis test were similar to that of balanced ANOVA. Thus, parametric one-way ANOVA was preferred over the Kruskal–Wallis test to maintain the power. All analyses were conducted using R software, version 3.1.2 (R Core Team, 2020).

3 | RESULTS

Overall, adding wood ash to forest soils did not, generally, have a measurable or consistent result on the measured SOM attributes, and the greatest effects were observed between sites. Due to the differences in wood ash chemistry and amounts applied, differences in stand age at the time of application, and differences in soil textural classes and dominant tree species, we used a staged analysis to partition data to address three key questions.

Question 1: *What was the effect on soil C indicators when applying different ash types at comparable applications rates (i.e., low C vs. high C ash) to regenerating conifer stands approaching crown closure (approximately 20 years since stand establishment)?* (Aleza Lake North and South, British Columbia).

Although there were no clear significant effects ($p < 0.05$), there were subtle effects ($p < 0.20$) from wood ash application on the pH, C:N, Cmin:MBC, and HWEC of the forest floor layer (Table 3). Wood ash application increased soil pH and the C:N at both the Aleza Lake North (ALN) and Aleza Lake South (ALS) sites (Figure 2a and b, respectively). For Cmin:MBC, which is an indicator of the efficiency of the microbial community, was lower in soils receiving wood ash, and lowest when high carbon (29.7% total C), boiler ash was applied (Figure 2c). Application of wood ash decreased the concentrations of HWEC, with the greatest effect when the low carbon (7.3% total C) gasifier ash was applied (Figure 2d).

There were some notable ($p < 0.20$) interactions between site and treatment effects for TC, TN, Cmin, HWEC (%TC) in the forest floor at ALN and ALS (Table 3). TC and TN increased with ash addition in the forest floor at ALN but decreased with ash addition at ALS (Figure 3a and b, respectively). A similar trend was seen with Cmin at the ALN site. Here, the highest Cmin concentrations were observed in the soil receiving gasifier ash (Figure 3c). For HWEC (%TC), there was no effect of wood ash application at the ALS site, but the application of either ash at ALN resulted in lower HWEC (%TC) (Figure 3d).

For the mineral soil layer, there were measurable site differences for TN, HWEC, and HWEC (%TC) (Table 4), with higher values for the ALS site (Figure 4a–c, respectively). Only HWEC and TN showed significant effects from the ash application treatments in the mineral soil, with higher HWEC and TN concentrations for both the

TABLE 3 ANOVA table with F and *p*-values indicating the significant effect of treatment (ash application), site and treatment by site interaction on the measured soil parameters in the forest floor layer at Aleza Lake North and Aleza Lake South

	Treatment		Site		Treatment × site	
Variable	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**
Total C	0.120	0.893	0.228	0.680	1.882	**0.195**
Total N	0.143	0.875	0.027	0.885	3.226	**0.076**
C:N	6.282	**0.137**	2.779	0.237	0.234	0.795
HWEC	6.316	**0.137**	0.171	0.719	0.171	0.845
MBC	0.106	0.904	0.291	0.644	1.312	0.305
Cmin	0.088	0.920	0.018	0.905	2.917	**0.093**
pH	16.489	**0.057**	0.771	0.472	0.448	0.649
HWEC (%TC)	1.014	0.497	0.075	0.810	3.142	**0.080**
Cmin (%TC)	1.448	0.408	0.335	0.621	0.324	0.729
MBC (%TC)	0.464	0.683	0.155	0.732	1.618	0.239
Cmin:MBC	6.663	**0.130**	2.977	0.227	0.099	0.906

Note: Bold values indicate significance at $p < 0.20$.

high carbon boiler and low carbon gasifier ash (Table 4; Figure 5a and b).

There was a significant interaction between treatment and site for soil pH in the mineral soil (Table 4). In this case, there was no effect of wood ash addition at ALN, but the addition of the high carbon boiler ash increased soil pH at ALS (Figure 6).

Question 2: *Was there a differential response of wood ash application depending on soil texture (fine- vs. coarse-textured soils) and amount of wood ash applied to regenerating stands?* (Coarser textured: Island Lake, Ontario and Pineland, Manitoba; Finer textured: 25th Side Road, Ontario and Mistik, Saskatchewan).

Not unexpectedly, the coarse-textured sandy sites consistently had lower soil C indices in the forest floor and mineral soil compared with the finer-textured sites (Tables 5 and 6). Beyond the site/texture effects, the ash application did alter TC, TN, MBC, and Cmin:TC in the forest floor when compared to the control (Figure 7). More specifically, the application of ash at 1 Mg ha^{-1} resulted in lower concentrations of TC, TN, and MBC in the forest floor, but resulted in higher Cmin:TC.

There were notable interactions ($p < 0.20$) between site (texture) and ash treatment effects for Cmin and pH (Table 5). For soil pH, there was little change with ash addition on the fine-textured sites, but a pH increase on the coarse-textured sites, most notable with the 1 Mg ha^{-1} application rate (Figure 8a). For Cmin, there was no effect of ash application on the coarse textured sites, but on the fine textured sites the application of ash at rates greater than 1 Mg ha^{-1} did result in lower rates of respiration (Figure 8b).

For the mineral soil layer, the application of wood ash to the soils resulted in higher ($p < 0.20$) concentrations of HWEC, Cmin at 1 Mg ha^{-1} (Figure 9a and b) and sand fraction N across all rates of application (Figure 9c).

There were significant treatments by site (texture) interaction effects for Cmin, Cmin:MBC, sand fraction C:TC and sand fraction N:TN (Table 6, Figure 10). The application of ash resulted in higher C mineralization on the coarse-textured soils, but the higher rate of ash application resulted in significantly lower C mineralization in the fine textured soils. For the sites with coarse-textured soils, the application of ash resulted in significantly lower MinC:MBC at the highest rate of application, whereas, on the fine textured soils, ash application resulted in significantly higher MinC:MBC (Figure 10). The application of ash also increased the sand fraction C (%TC) and N (%TN) on the coarse-textured soils but had no effect in the fine textured soils (Figure 10).

Question 3: *Was there an effect of wood ash application in mature, selection harvested, tolerant hardwood stands?* (Haliburton, Ontario and Eastern Townships, Quebec).

There were differences in some C indices between the tolerant hardwood sites, with lower TC and HWEC but higher pH at Haliburton (HLB) compared with the Eastern Townships (ETM) in the forest floor layer (Table 7). For the mineral soil layer, HWEC, sand fraction C and N were also higher at HLB compared with ETM (Table 8).

Ash application resulted in slightly lower TC in the forest floor ($p = 0.072$) but higher HWEC (%TC) ($p = 0.128$) and MBC (%TC) ($p = 0.121$) in the mineral soil. There were, however, interaction effects ($p < 0.20$) between site and treatment for MBC and MBC (%TC) in the forest floor layer (Table 7) and for TN, MBC, and sand fraction N in the mineral soil (Table 8). Both MBC and MBC (%TC) increased with ash application at ETM and decreased with ash application at HLB (Figure 11). In the mineral soil, ash application at ETM had no effect on TN, MBC, and sand fraction N but resulted in significant declines in these indicators at HLB (Figure 12).

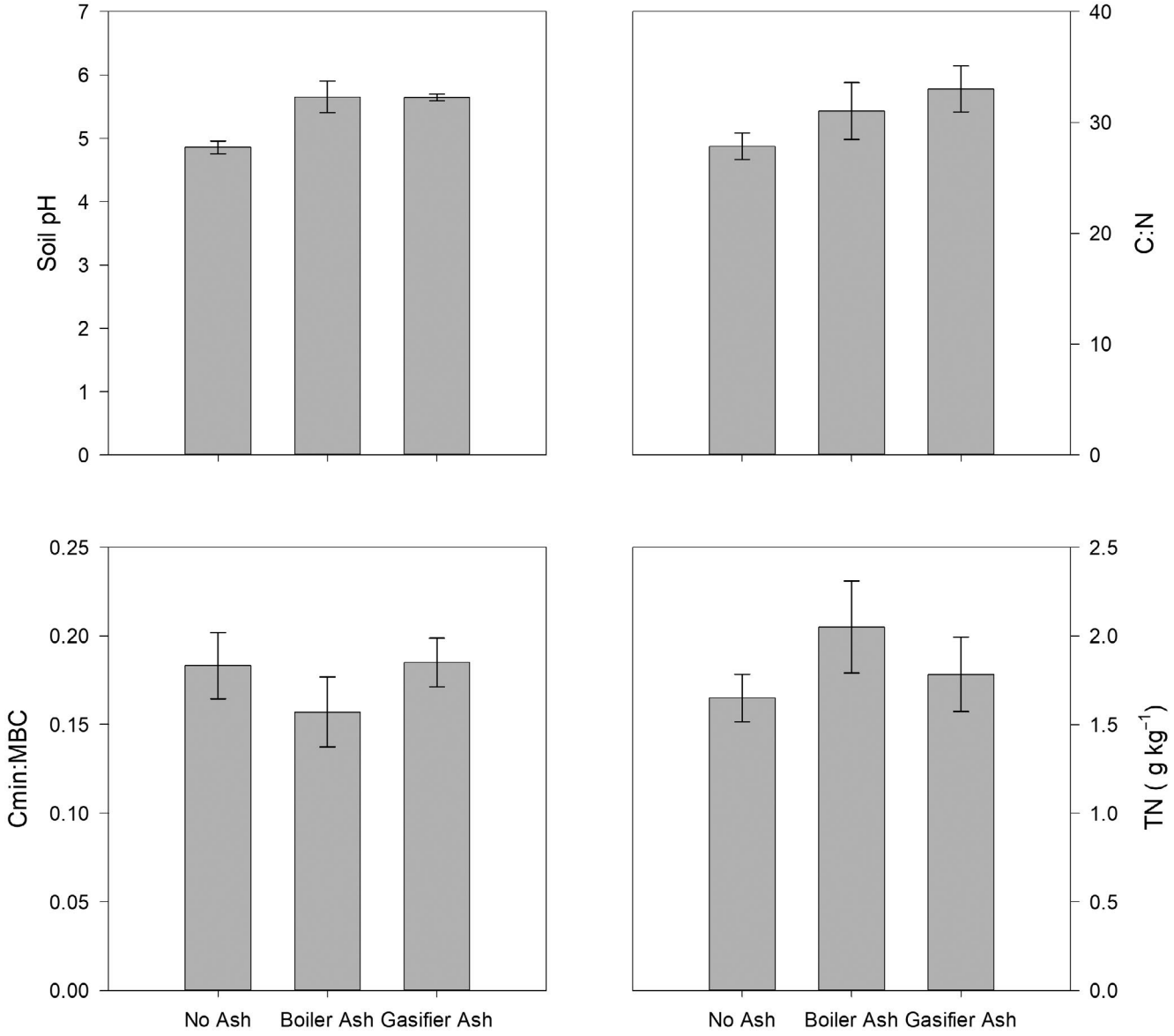

FIGURE 2 Difference between select indicators and ash application type (control, low carbon gasifier ash, high carbon boiler ash) on forest floor (a) pH, (b) C:N, (c) Cmin:MBC, and (d) hot water extractable C at Aleza Lake North and Aleza Lake South. Error bars standard error

Following the staged approach, we evaluated the effect of adding wood ash to the soil at each individual site. Like the staged approach, adding wood ash to the forest soils did not, generally, have significant ($p < 0.05$) or consistent effects on the measured SOM attributes (Supplementary Material). A significant effect of wood ash application on TC concentration was only observed at two of the AshNet sites, that is, Island Lake (northeast Ontario) and Pineland (eastern Manitoba). At Island Lake, the application of wood ash at a rate of 1.4 Mg ha^{-1} resulted in significantly lower TC concentrations in the litter-moss (LM) layer only (F = 7.20, $p = 0.00$). At the Pineland site (eastern Manitoba), application of wood ash at a rate of 1.5 Mg ha^{-1} resulted in a significantly higher TC concentration (10.8 g kg^{-1}) compared to the control (5.8 g kg^{-1}) in the mineral soil (F = 9.70, $p = 0.02$). Carbon concentrations in the sand size fraction were also significantly higher (4.4 g kg^{-1}) in the soils receiving wood ash compared to the control (1.5 g C kg^{-1}) (F = 6.47, $p = 0.03$) at the Pineland site.

The application of 20 Mg ha^{-1} of wood ash resulted in a significant increase (F = 7.44, $p = 0.03$) in N in the LM layer (16.6 g N kg^{-1}) compared to the control (14.6 g N kg^{-1}) at the Eastern Townships site. At the Pineland site, there was a significant increase (F = 13.09, $p = 0.01$) in N in the mineral soil layer (0.4 g N kg^{-1} in the control compared with 0.6 g N kg^{-1}) with the application of 1.5 Mg ha^{-1} of wood ash. At the Island Lake site, TN concentrations

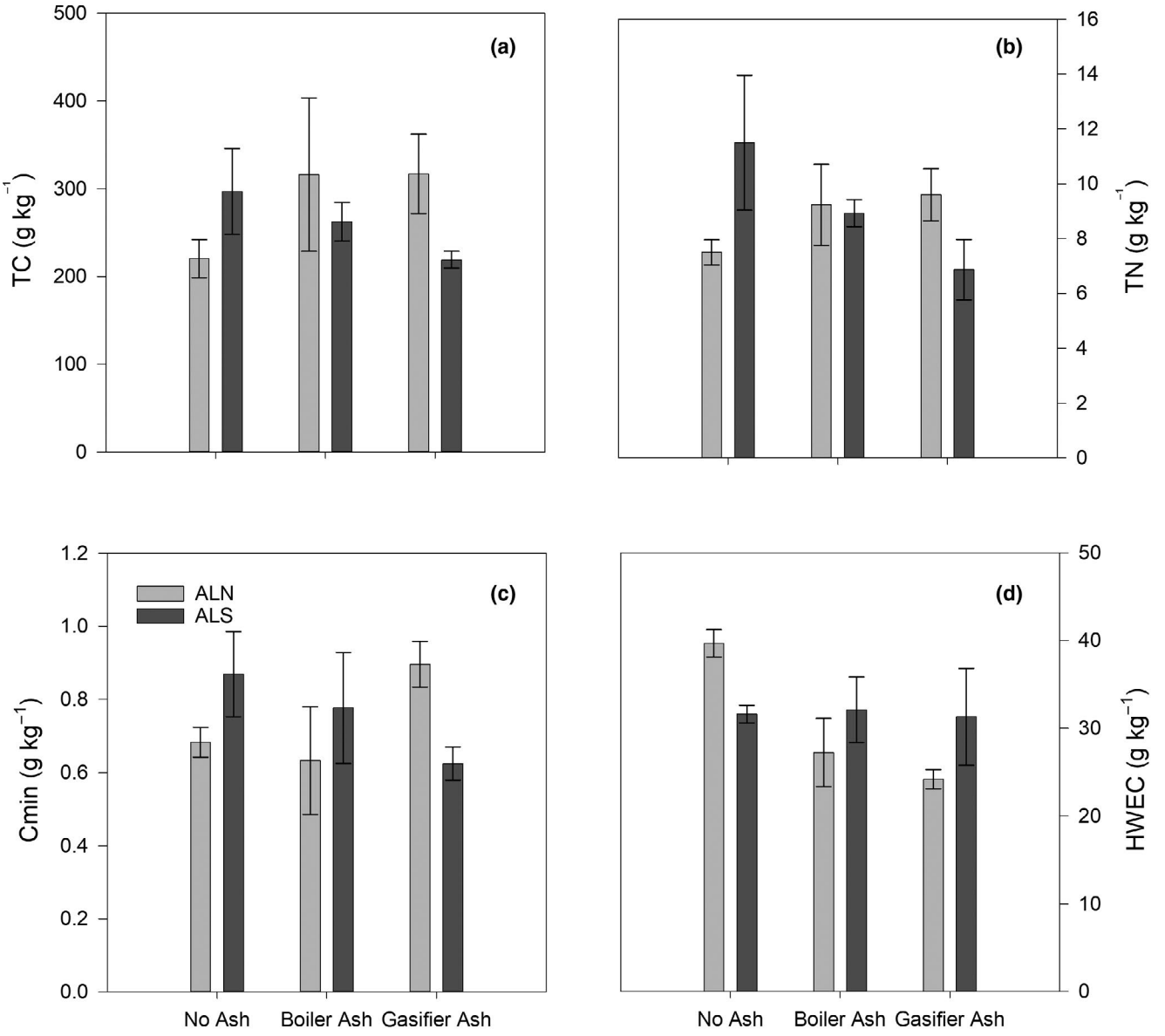

FIGURE 3 Interaction of treatment and site on concentrations of (a) total carbon, (b) total nitrogen, (c) carbon mineralized, and (d) hot water extractable carbon in the forest floor layer. Overall is the grand mean of the ash treatment. Error bars indicate standard error

were significantly lower than the control in the LM layer when wood ash was applied at rates of 1.4 and 2.8 Mg ha^{-1} (F = 6.20, p = 0.00; Figure 13). Differences in the mineral soil C:N with wood ash application were only observed at the Eastern Townships site (southern Quebec) (F = 5.70, p = 0.04), where the ratio was narrower when wood ash was applied at a rate of 20 Mg ha^{-1} (C:N = 26.2) compared with the control (C:N = 29.8). Hot water extractable C concentrations were higher in the wood ash treatment (0.4 g ka^{-1}) than the control (0.2 g kg^{-1}) in the mineral soil at the Pineland site (F = 10.81, p = 0.02) but lower than the control in the LM layer at the Eastern Townships site (23.6 and 18.2 g kg^{-1}, control and wood ash, respectively; F = 6.30, p = 0.04). Differences in the MBC fraction were only detected at the Eastern Townships site (F = 8.64, p = 0.02) where concentrations were higher in the LM layer with the application of 20 Mg ha^{-1} wood ash (6.08 g kg^{-1}) relative to the control (3.57 g kg^{-1}). Mineralizable C concentrations differed in soils receiving wood ash at the Pineland site (F = 7.70, p = 0.03) in the mineral soil (82 mg kg^{-1} relative to the control 34 mg kg^{-1}) (Supplementary Information).

Significant differences in carbon normalized concentrations of HWEC were observed in soils receiving wood ash at Aleza Lake North (northern British Columbia), Eastern Townships and Island Lake sites. At Aleza Lake North (F = 10.92, p = 0.01) and Eastern Townships (F = 6.90, p = 0.03) sites, values decreased with wood ash

TABLE 4 ANOVA table with F and *p*-values indicating the significant effect of treatment (ash application), site and treatment by site interaction on the measured soil parameters in the mineral soil

	Treatment		Site		Treatment × site	
Variable	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**
Total C	2.851	0.260	1.059	0.412	0.316	0.735
Total N	37.333	**0.026**	6.750	**0.122**	0.021	0.979
C:N	1.613	0.383	0.485	0.558	0.375	0.695
HWEC	28.251	**0.034**	85.222	**0.012**	0.023	0.977
MBC	3.487	0.223	0.523	0.545	0.453	0.646
Cmin	0.913	0.523	0.503	0.552	0.382	0.69
Sand fraction C	1.488	0.402	1.374	0.362	0.508	0.614
Sand fraction N	1.955	0.338	2.336	0.266	0.311	0.738
pH	0.579	0.633	0.163	0.725	2.664	**0.110**
HWEC (%TC)	1.907	0.344	3.998	**0.184**	0.341	0.718
Cmin (%TC)	0.062	0.941	0.768	0.473	0.365	0.702
MBC (%TC)	1.258	0.443	1.909	0.301	0.296	0.749
Cmin:MBC	0.155	0.866	2.782	0.237	0.544	0.594
Sand C (%TC)	0.865	0.536	1.164	0.393	0.602	0.563
Sand N (%TN)	0.755	0.570	1.804	0.311	0.652	0.538

Note: Bold values indicate significance at $p < 0.20$.

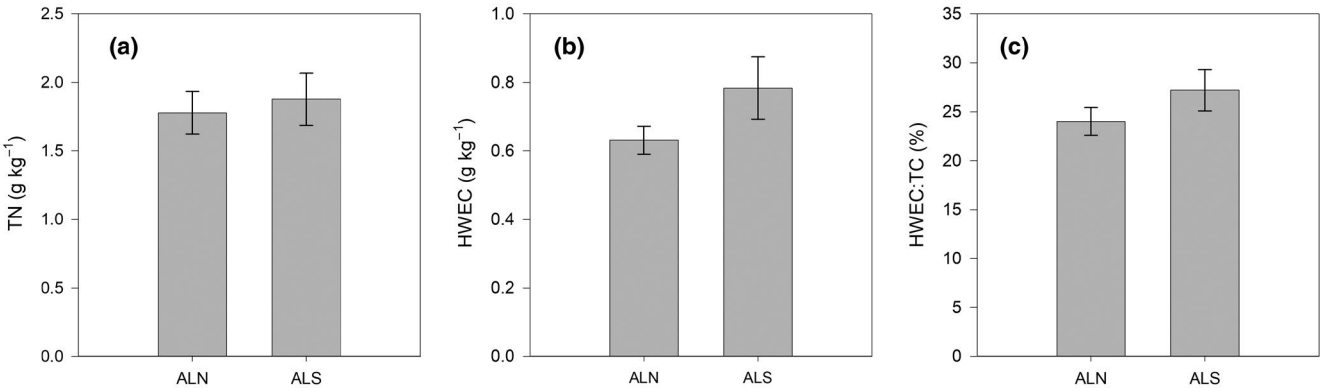

FIGURE 4 Site differences in (a) total nitrogen, (b) hot water extractable C, and (c) normalized hot water extractable carbon concentrations in the upper mineral soil at Aleza Lake North (ALN) and Aleza Lake South (ALS). Error bars indicate standard error

application in the LFH (5 Mg ha^{-1}; both wood ash types) and LM (20 Mg ha^{-1}) layers respectively. At Aleza Lake North, HWEC was 4% of TC in control soil whereas in the soil receiving wood ash, this fraction was 2.7 and 2.4% of TC for the boiler and gasifier wood ashes respectively. At the Eastern Townships site, HWEC accounted for 5.5% of TC in the control soil and 4.2% in the wood ash soil. At the Island Lake site, the normalized HWEC and mineralizable C fractions increased in the LM layer when wood ash was applied at a rate of 1.4 Mg ha^{-1} ($F = 3.19$, $p = 0.04$; Figure 13). A significant increase in the MBC/TC ratio was detected at the Eastern Townships site in the LM layer ($F = 9.33$, $p = 0.02$), where wood ash was applied at a rate of 20 Mg ha^{-1}. Microbial biomass C accounted for 0.8% of TC in the control and 1.4% in the wood ash treated soil. There were no significant differences in any normalized fraction at the Aleza Lake South, Mistik, Pineland, 25th Side Road, and Haliburton sites.

Although the application of wood ash resulted in an overall increase in soil pH, the increase was only significant at four of the sites. The pH in the LFH ($F = 9.73$, $p = 0.01$) and mineral layer ($F = 8.64$, $p = 0.02$) at the Aleza Lake South site (northern British Columbia) increased significantly at a wood ash application rate of 5 Mg ha^{-1}. The pH in the LFH was 4.9 in the control and 5.8 and 5.6 in the soils receiving the boiler and gasifier ash, respectively. In the mineral soil, the soil pH was 4.2 in the control and 4.6 and 4.3 in the soils receiving the boiler and gasifier ash respectively. At the Eastern Townships site, the application of wood ash at 20 Mg ha^{-1} significantly increased the pH in the LM layer ($F = 5.47$, $p = 0.05$) where the pH was 4.4 in the control and 4.9 in the soils receiving

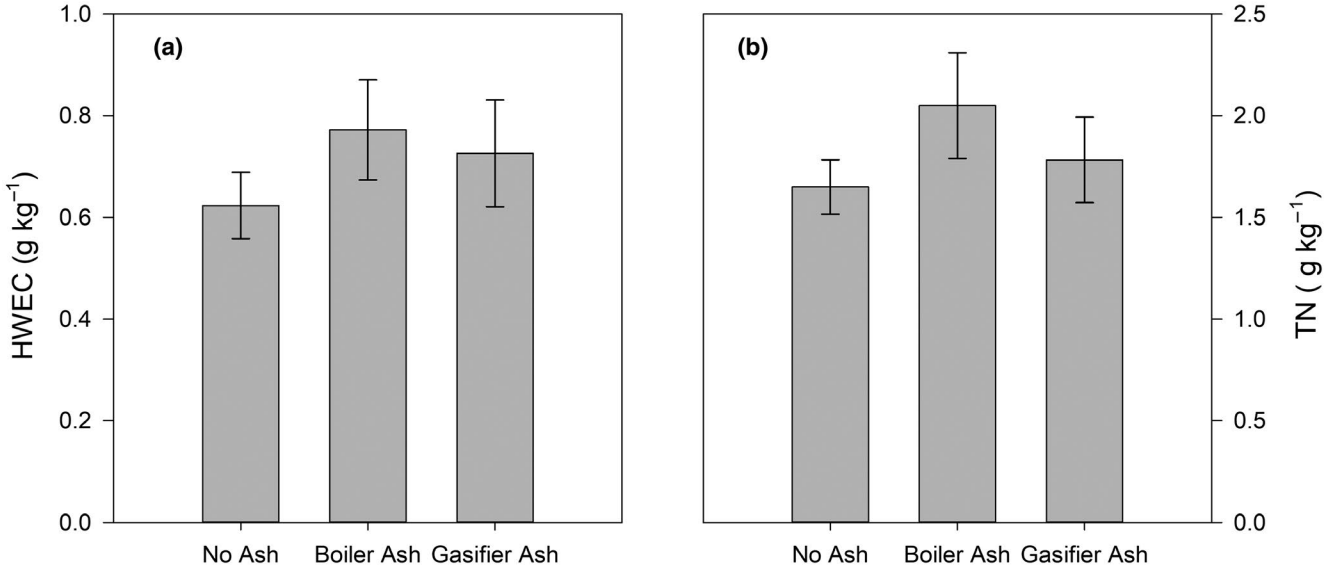

FIGURE 5 Response of (a) hot water extractable carbon and (b) total nitrogen concentrations to wood ash application in the mineral soil at Aleza Lake North and Aleza Lake South. Error bars indicate standard error

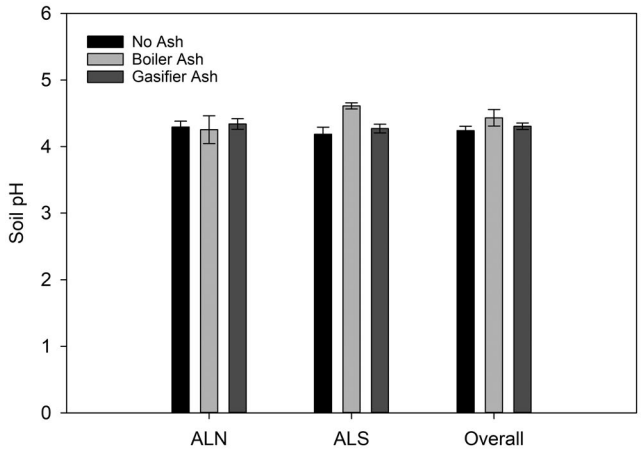

FIGURE 6 Interaction of site and ash application on soil pH in the mineral soil at Aleza Lake North (ALN) and Aleza Lake South (ALS). Overall is the grand mean of the ash application treatment. Error bars indicate standard error

ash. The application of fly and bottom wood ash mixture at the rate of 1.5 Mg ha^{-1} significantly increased the pH of the LFH layer at the Pineland site ($F = 5.95$, $p < 0.04$) from 5.0 to 60. Mineral soil pH also increased significantly at the 25th Side Road site (northwestern Ontario) from 5.2 to 6.2 when low C fly ash was applied at the rate of 10 Mg ha^{-1} ($F = 45.27$, $p < 0.00$).

4 | DISCUSSION

Little has been reported about the influence of wood ash application on SOM attributes in forest soils relative to pH and base cation concentrations (Reid & Watmough, 2014). This study represents the first to evaluate multiple SOM indicators across multiple sites that have received wood ash to the soil. The attributes measured in this study are known to be sensitive indicators of change in SOM and soil quality and have been shown to be responsive to changes in the rates of inputs and outputs of the actively cycled portion of the total soil C pool (e.g., Goh et al., 2000; Zagal et al., 2009). Surprisingly, adding wood ash to forest soils did not, generally, have measurable or consistent results on the measured SOM attributes and the greatest effects were observed between sites.

4.1 | Staged analyses

At the two sites in north-central British Columbia, there were few differences attributable to the chemistry of the ash applied, that is, boiler (high C) versus gasifer (low C) ash and only HWEC differed in both the forest floor and mineral soil layers. HWEC, an indicator of soil structure, declined in the forest floor, with the greatest decreases observed when gasifier ash was applied, while in the mineral soil, HWEC increased with greatest increases observed when boiler ash was applied. More notable is that interaction effects between ash application and site were more common, suggesting that site specific factors are important drivers of the response of soil attributes to ash addition.

In central Canada, soil texture and sites differences within textural class had the greatest influence on the measured attributes. The interaction effect of texture and treatment was significant, and only at the $p < 0.20$ level, for just two attributes in the FF and four attributes in

TABLE 5 ANOVA table with F and *p*-values indicating the significant effect of soil texture, site within texture class, treatment (ash application), and treatment by texture interaction on the measured soil parameters in the forest floor

	Texture		Site (texture)		Treatment		Texture × treatment	
Variable	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**
Total C	2.799	**0.099**	14.560	**0.000**	1.868	**0.162**	0.510	0.603
Total N	4.219	**0.044**	26.141	**0.000**	2.230	**0.115**	0.146	0.864
C:N	35.893	**0.000**	1.680	**0.194**	0.437	0.648	0.290	0.749
HWEC	31.773	**0.000**	2.171	**0.122**	0.053	0.949	0.254	0.776
MBC	2.489	**0.119**	4.981	**0.010**	2.075	**0.134**	0.266	0.767
Cmin	26.402	**0.000**	24.642	**0.000**	1.646	0.201	2.031	**0.139**
pH	123.694	**0.000**	63.241	**0.000**	3.536	**0.035**	2.287	**0.109**
HWEC (%TC)	2.246	**0.139**	1.513	0.228	0.958	0.389	0.195	0.823
Cmin (%TC)	17.419	**0.000**	8.219	**0.001**	1.733	**0.185**	0.251	0.779
MBC (%TC)	33.026	**0.000**	7.828	**0.001**	0.881	0.419	0.218	0.805
Cmin:MBC	3.536	**0.065**	7.097	**0.002**	0.667	0.516	0.228	0.797

Note: Bold values indicate significance at $p < 0.20$.

TABLE 6 ANOVA table with F and *p*-values indicating the significant effect of soil texture, site within texture class, treatment (ash application), and treatment by texture interaction on the measured soil parameters in the mineral soil

	Texture		Site (texture)		Treatment		Texture × treatment	
Variable	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**
Total C	0.149	0.701	7.512	**0.001**	1.273	0.289	0.714	0.495
Total N	6.051	**0.018**	5.934	**0.005**	1.645	0.204	1.482	0.238
C:N	26.045	**0.000**	16.201	**0.000**	0.589	0.559	0.139	0.871
HWEC	0.003	0.954	4.446	**0.017**	2.007	**0.146**	0.337	0.716
MBC	1.177	0.284	1.615	0.210	1.318	0.278	0.450	0.641
Cmin	2.362	**0.131**	1.457	0.244	2.087	**0.136**	1.706	**0.193**
Sand fraction C	0.367	0.547	4.092	**0.023**	1.447	0.246	0.605	0.550
Sand fraction N	5.305	**0.026**	1.572	0.218	1.701	**0.194**	1.080	0.348
pH	45.209	**0.000**	18.839	**0.000**	0.663	0.520	0.959	0.391
HWEC:TC	5.866	**0.019**	8.708	**0.001**	0.302	0.741	1.441	0.247
Cmin:TC	0.751	0.391	7.698	**0.001**	0.412	0.665	0.511	0.603
MBC:TC	3.218	**0.080**	4.672	**0.014**	0.464	0.632	0.376	0.689
Cmin:MBC	2.054	**0.159**	3.094	**0.056**	1.409	0.255	1.695	**0.196**
Sand C:total C	6.874	**0.012**	8.371	**<0.001**	1.709	**0.192**	2.640	**0.082**
Sand N:total N	4.368	**0.042**	2.369	**0.105**	0.935	0.400	2.365	**0.105**

Note: Bold values indicate significance at $p < 0.20$.

the mineral soil. The greatest changes were generally observed when wood ash was applied at a rate of 1 Mg ha^{-1} as opposed to higher rates of application.

Application of wood ash in mature, selection harvested, tolerant hardwood stands in Eastern Canada also had very few significant effects on the measured attributes, with the response more often differing between sites in both soil layers.

4.2 | Site-specific analyses

Of the 12 indicators measured at the eight sites in two to three soil layers per site in the site specific analysis, the application of wood ash to the soil only resulted in 16 significant differences between the control and ash applications. This detection rate of significance (8% for all tests) is low for this number of tests, and when the values

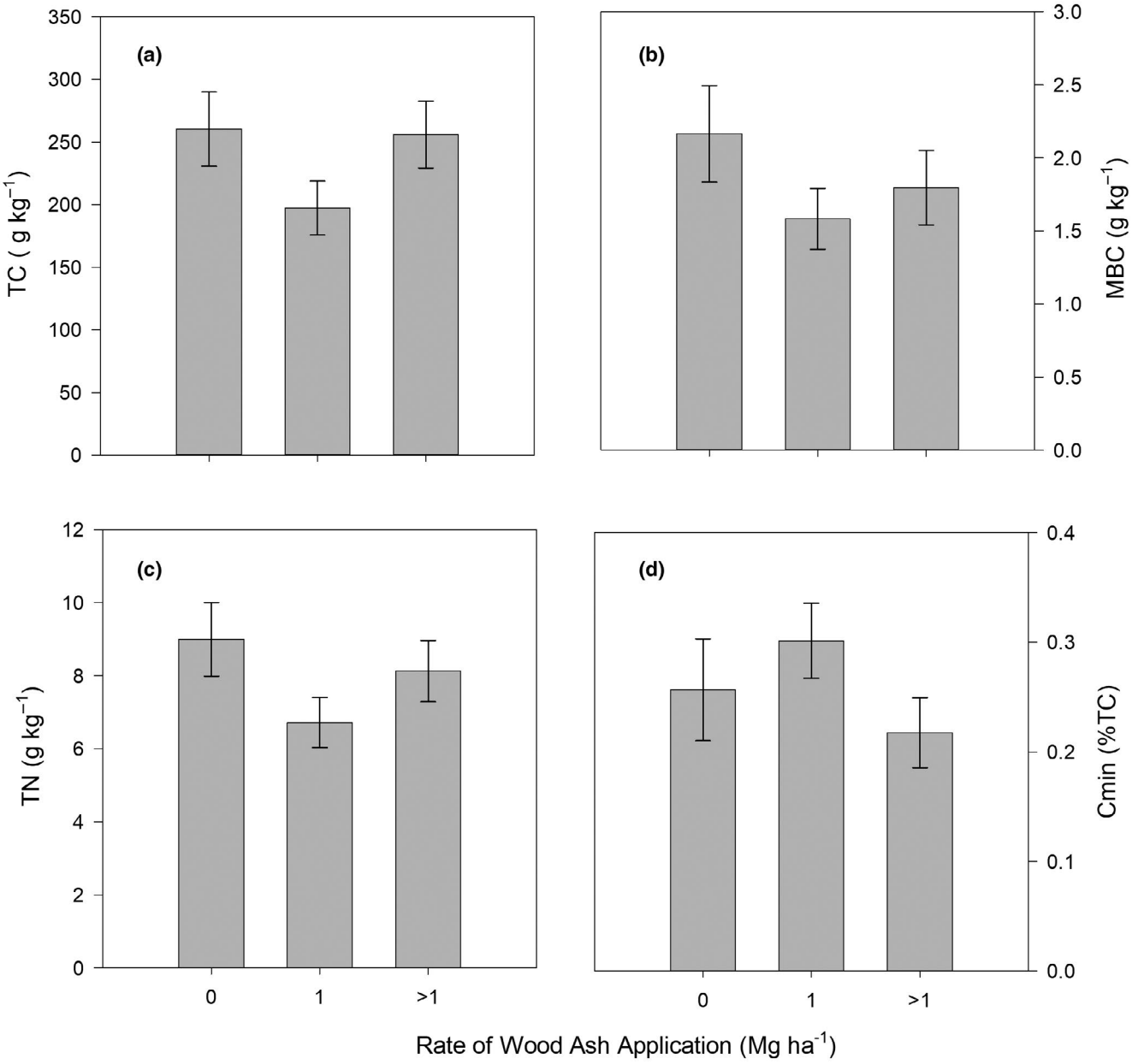

FIGURE 7 Concentration changes in response to ash application on the concentrations of (a) total carbon, (b) microbial biomass carbon, (c) total nitrogen, and (d) carbon mineralized normalized to total carbon in the forest floor. Error bars indicate standard error

are evaluated in the context of a Bonferonni corrected alpha (Armstrong, 2014) of 0.0028 (0.05/15), only TC and TN concentrations in the forest floor layer at the Island Lake site were significantly different from the other sites. Additionally, only the mineral soil pH increase observed at the 25th Side Road in the 10 Mg ha^{-1} treatment was significant.

Using the C content of each wood ash applied at each site, we estimated that the application of wood ash only added between 0.006 and 1.18 Mg C ha^{-1} to the soil. The density of wood ash is often low particularly for fly ash recovered from boiler exhaust (Demeyer et al., 2001), so even when wood ash is being applied at higher rates (i.e., large volumes) C mass additions remained relatively low. The rates of C applied across the treatments only represented <0.01 to 2.35% of the estimated C stocks in the forest floor and top 15 cm of mineral soil at these sites, respectively. It follows that unless wood ash is being applied at very high rates or the applied wood ash has a high C concentration, any differences in TC pools detected would likely be from the effects the wood ash had on C cycling through increased rates of decomposition that favored native SOM loss or increases in tree growth and site productivity that favored increased organic matter inputs.

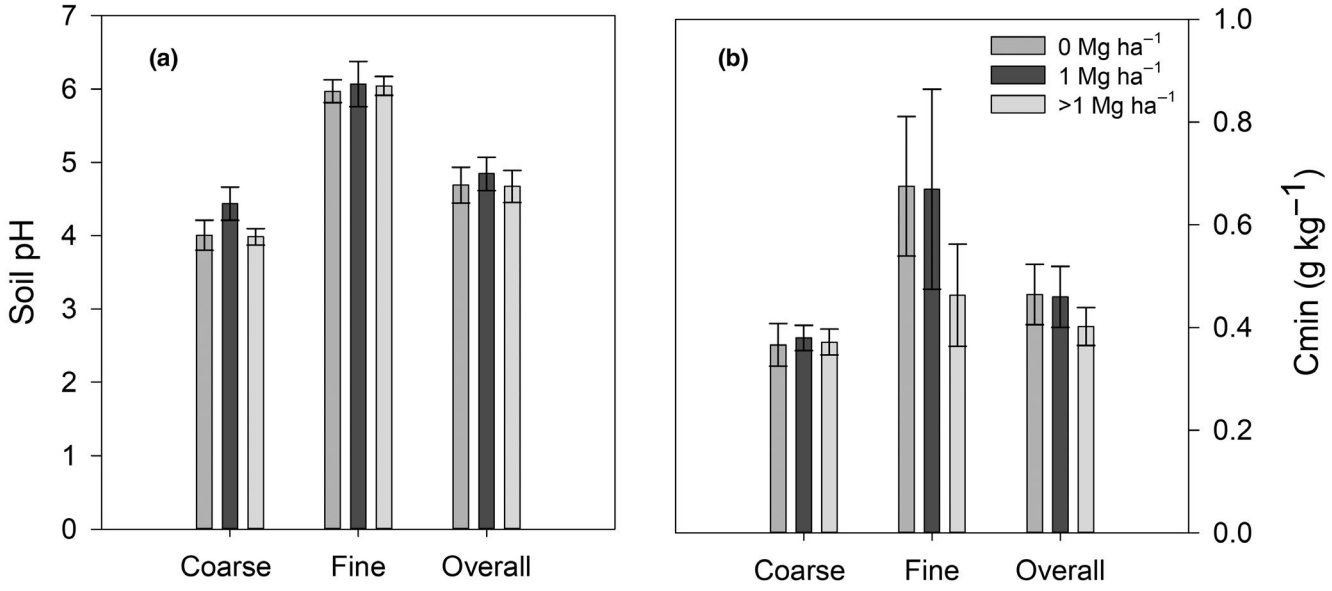

FIGURE 8 Response of (a) soil pH and (b) carbon mineralized in the forest floor layer to the interaction of soil texture and rate of ash application. Overall is the grand mean of the ash application treatment. Error bars indicate standard error

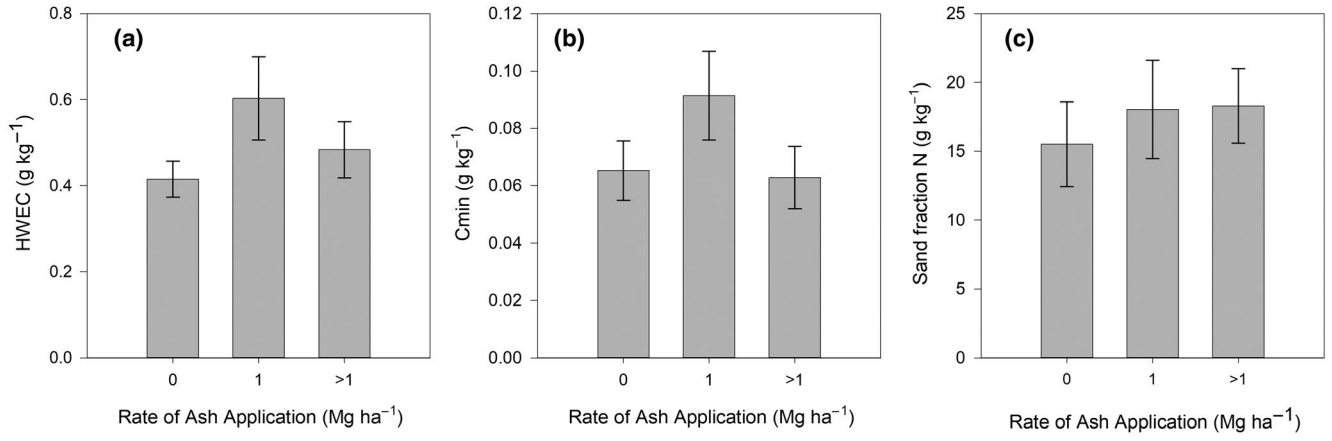

FIGURE 9 Response of concentrations of (a) hot water extractable carbon, (b) carbon mineralized, and (c) sand fraction N in the mineral soil to rate of ash application. Error bars indicate standard error

4.3 | Differences are most pronounced near the surface

When significant differences were detected, they were most often evident in the soil layer closest to the surface where the ash was applied, but the effects were inconsistent. At the Island Lake site, six years after wood ash application, TC concentrations in the LM layer were significantly lower when wood ash was applied at a rate of 1.4 Mg ha^{-1} than when wood ash was not applied (control) or applied at rates of 0.7 and 5.6 Mg ha^{-1}. The same trend, although not significant, was observed in the FH layer immediately below the LM. Brais et al. (2015) also reported a significant decline in TC concentrations in the forest floor in the first two years following wood ash application at 2 and 8 Mg ha^{-1} in a well-drained Dystric Eluviated Brunisol, but those concentrations remained significantly higher than the control 5 years after application. This was not the case at Island Lake. Gömöryová et al. (2016) reported that the decrease in C and N concentrations in the forest floor was accompanied by an increase in soil pH and base cation concentration which, in turn, appeared to favor increased rates of SOM decomposition. Organic matter decomposition results in the release of inorganic (root available) nutrients into the soil and when coupled with the increase in pH and nutrients from the wood ash itself, plant nutrient uptake and subsequent growth may be enhanced, thus resulting in increased rates of organic matter residue return to the soil. However, Brais et al. (2015) did report a

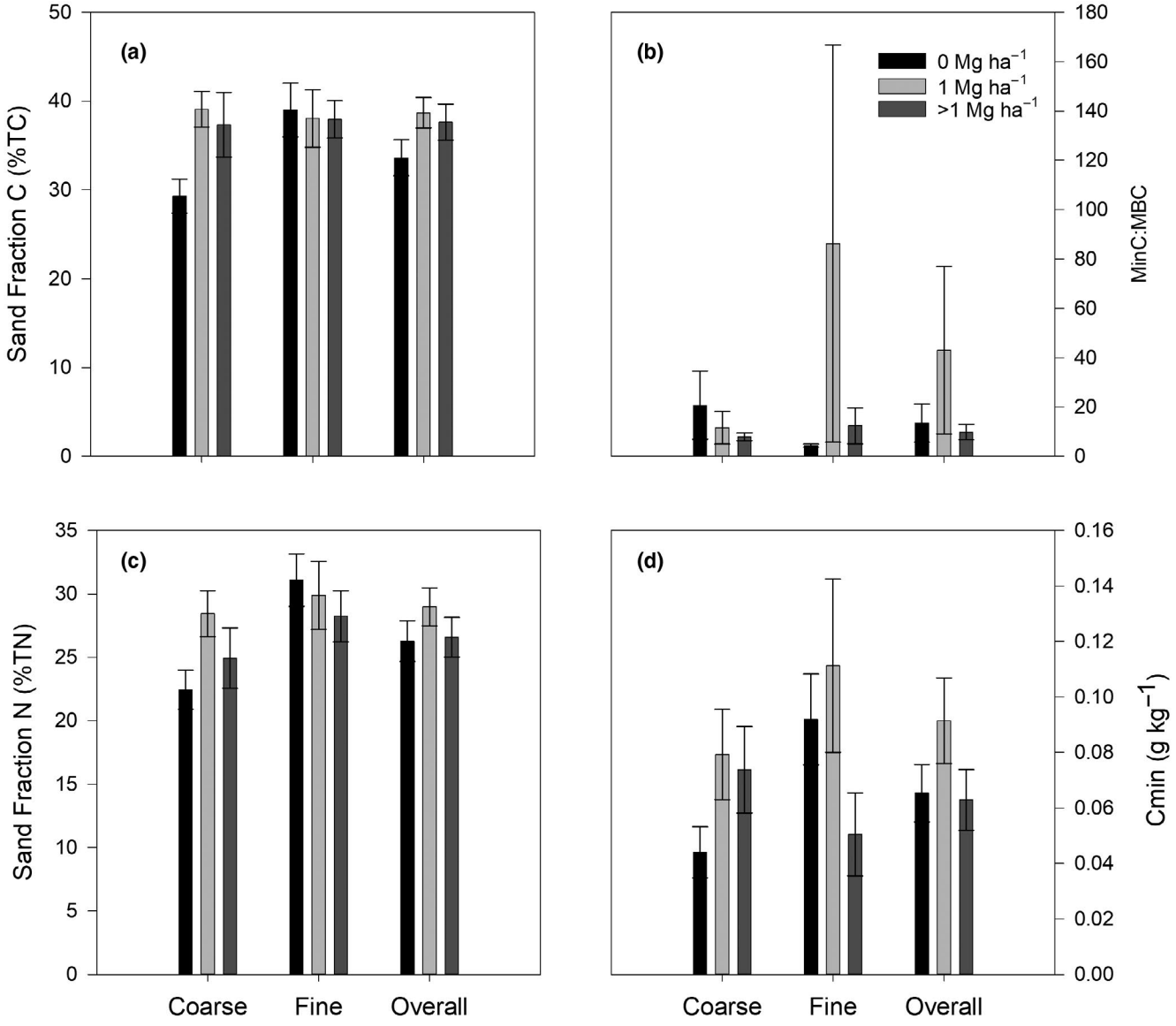

FIGURE 10 Response of (a) normalized sand fraction carbon, (b) MinC:MBC, (c) normalized sand fraction nitrogen, and (d) carbon mineralized in the mineral soil to the interaction effect of soil texture and ash application. Overall is the grand mean of the ash application treatment. Error bars indicate standard error

decrease in black spruce growth with increased wood ash loading rates, but this may have been an indirect effect where an increase in intraspecific competition from other plants that respond positively to wood ash application countered any potential black spruce growth response (e.g., Shepard, 1997). At Island Lake, the wood ash chemistry may have resulted in longer term changes in soil chemistry that, in turn, may have favored higher rates of SOM decomposition that exceeded litter input rates. It remains unclear why this result was only observed at an intermediate level of wood ash application. It may be that the nutrient limitations that appear to restrict SOM decomposition in the intermediate application rate are overcome when higher wood ash rates are applied. Although not significant, C concentrations in the forest floor did increase at Aleza Lake North but decreased at Aleza Lake South where the same wood ash rate was applied. In this case, wood ash was applied 2 years prior to sample collection to an 18-year-old planted stand (Aleza Lake North) and a 24-year-old planted stand that had been broadcast burned prior to planting (Aleza Lake South). These results suggest that the observed responses, when they do occur, are likely to be site specific and a function of site characteristics, wood ash chemistry and loading rate, and stand age at the time of application.

At Pineland, where there was a very thin forest floor and the lowest C concentration in the mineral soil compared to the other study sites, the concentration of C in the mineral soil doubled with the application of 1.5 Mg

TABLE 7 ANOVA table with F and *p*-values indicating the significant effect of treatment (ash application), site and treatment by site interaction on the measured soil parameters in the forest floor at HLB and ETM

	Treatment		Site		Treatment × site	
Variable	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**
Total C	77.889	**0.072**	801.665	**0.022**	0.007	0.936
Total N	0.536	0.598	9.192	0.203	0.417	0.524
C:N	0.318	0.673	3.172	0.326	1.031	0.319
HWEC	1.247	0.465	3.238	0.323	0.375	0.546
MBC	0.020	0.910	0.199	0.733	2.659	**0.115**
Cmin	1.347	0.453	9.391	0.201	0.128	0.723
pH	1.849	0.404	49.894	**0.090**	0.302	0.587
HWEC (%TC)	0.109	0.797	112.271	**0.060**	0.174	0.680
Cmin (%TC)	0.000	0.993	16.204	0.155	0.406	0.529
MBC (%TC)	0.147	0.767	0.035	0.882	6.796	**0.015**
Cmin:MBC	1.088	0.487	5.722	0.252	0.972	0.333

Note: Bolded values indicate significance at $p < 0.20$.

TABLE 8 ANOVA table with F and *p*-values indicating the significant effect of treatment (ash application), site and treatment by site interaction on the measured soil parameters in the mineral soil at Haliburton and Eastern Townships

	Treatment		Site		Treatment × site	
Variable	**F**	***p*-value**	**F**	***p*-value**	**F**	***p*-value**
Total C	1.172	0.475	9.019	0.205	0.932	0.347
Total N	1.178	0.474	4.807	0.272	1.800	**0.196**
C:N	0.005	0.957	20.365	**0.139**	0.363	0.554
HWEC	0.377	0.649	70.745	**0.075**	0.308	0.586
MBC	2.798	0.343	8.783	0.207	2.059	**0.168**
Cmin	3.387	0.317	8.257	0.213	1.066	0.315
Sand fraction C	1.064	0.490	6.731	0.234	1.627	0.218
Sand fraction N	1.111	0.483	5.885	0.249	2.126	**0.162**
pH	11.476	0.183	9.753	**0.197**	0.065	0.802
HWEC (%TC)	24.151	**0.128**	0.573	0.588	0.019	0.892
Cmin (%TC)	8.670	0.208	3.855	0.300	0.093	0.764
MBC (%TC)	26.819	**0.121**	0.964	0.506	0.019	0.892
Cmin:MBC	0.868	0.522	2.800	0.343	0.309	0.585
Sand C:total C	2.094	0.385	43.865	**0.095**	0.808	0.380
Sand N:total N	2.671	0.350	73.225	**0.074**	0.409	0.531

Note: Bolded values indicate significance at $p < 0.20$.

ha^{-1} wood ash. In contrast, there were no significant differences detected for any attribute at the 25th Side Road site, which has a very thin forest floor, and received both a similar and a higher rate of wood ash application. These contrasting results suggest that while the layers closest to the surface where wood ash was applied are likely to show the greatest change in response to wood ash application, there was no generalized response to wood ash application. These observations also suggest that soils with a low C concentration are the most likely to have a detectable increase in C with the application of wood ash that has a high C content.

4.4 | Labile C fractions are responsive but not consistent across sites

The labile C fractions accounted for the majority of the significant changes (10 of the 16 differences observed in the site-specific analysis in this study) and appear

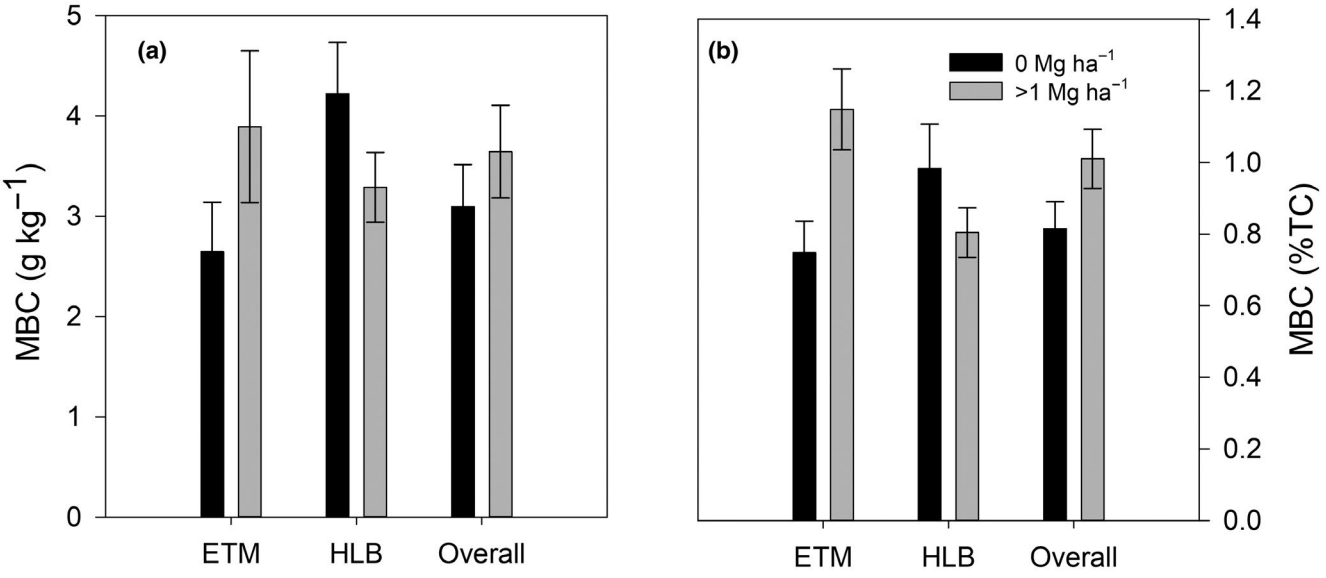

FIGURE 11 Response of ash application treatment and site on (a) microbial biomass carbon and (b) normalized microbial biomass carbon in the forest floor at Haliburton (HLB) and Eastern Townships (ETM) and to the interaction effect of ash application treatment site. Overall is the grand mean of the ash application treatment. Error bars indicate standard error

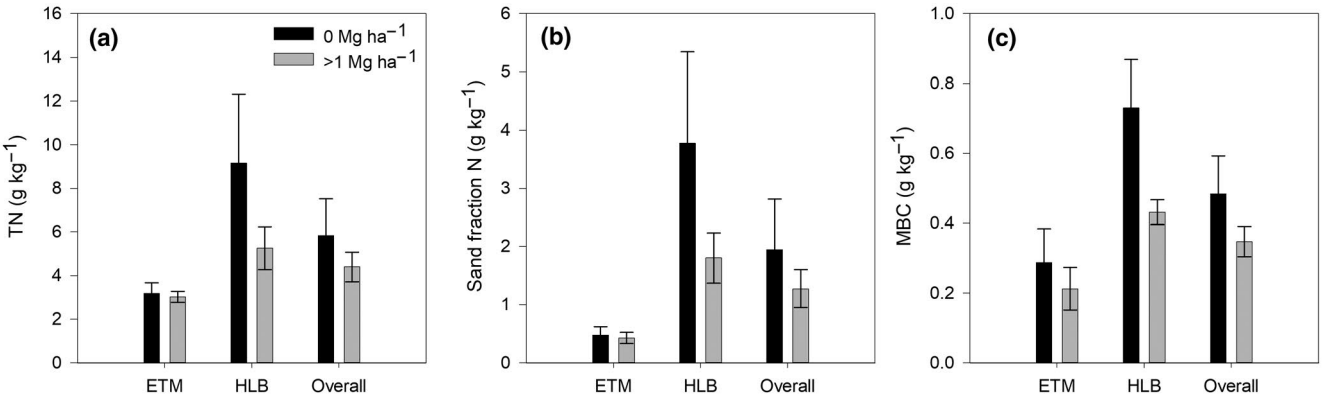

FIGURE 12 Response of ash application treatment and site on concentrations of (a) total nitrogen, (b) sand fraction nitrogen, and (c) microbial biomass carbon in the mineral soil to the interaction effect of ash application treatment and site. Overall is the grand mean of the ash application treatment. Error bars indicate standard error

to be more responsive than TC, TN and C:N to wood ash application. However, the sensitivity of these C fractions was also not consistent across the sites. The most responsive labile C fractions were HWEC and normalized HWEC (to TC). These were the only indicators that showed a significant response to wood ash application across multiple sites. Even then, the direction of the change was not consistent across sites. For example, HWEC concentrations decreased in the forest floor layer at the Eastern Townships site but increased in the mineral soil at the Pineland site. Normalized HWEC decreased in the forest floor at both the Aleza Lake North and Eastern Township sites but increased at Island Lake. HWEC and MBC fractions are commonly correlated with each other and with the rate of microbial respiration (Bera et al., 2019; Ghani et al., 2003; Weigel et al., 2011). Hot water extractable C is typically expected to be easily available for microbial decomposition (Ghani et al., 2003) and when HWEC increases, MBC and CO_2 from microbial decomposition typically increase. MBC increased at the Eastern Townships site with wood ash application but HWEC declined and there was no significant effect on microbial respiration (mineralizable C). The significant increase in the MBC and the proportion of soil C in MBC (MBC/TC) in the forest floor at the Eastern Townships site, combined with the significant decline in HWEC, could be attributed to an increase in in situ microbial activity (McFee & Kelly, 1995; Saarsalmi et al., 2012). This result could also be due to the significant, and persistent, increase in the soil pH at the site, thus resulting in enhanced microbial activity and a higher rate of C cycling (Bååth

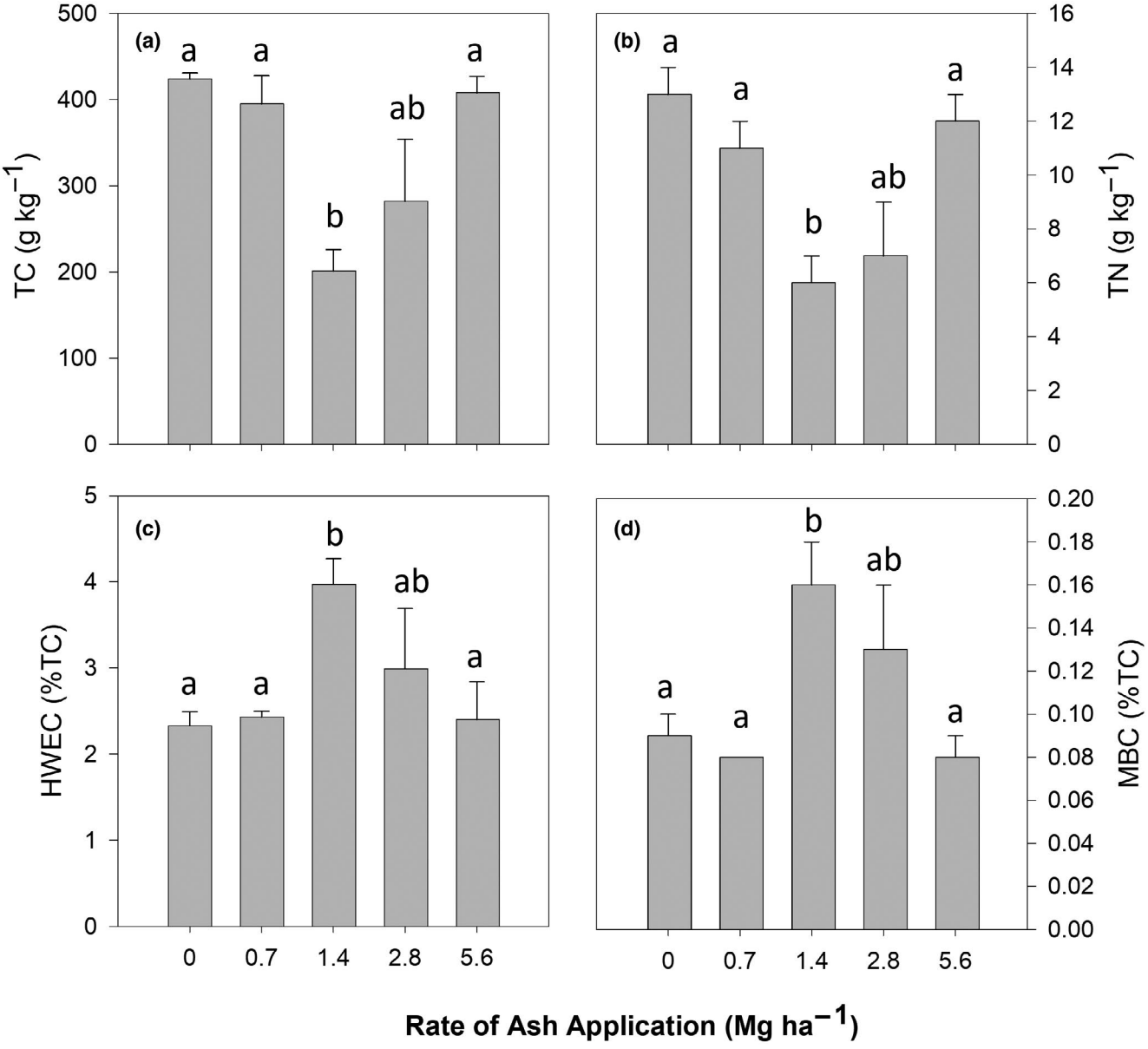

FIGURE 13 Response of select indicators to wood ash application at Island Lake on (a) total carbon concentrations, (b) total nitrogen concentrations, (c) normalized hot water extractable carbon, and (d) normalized microbial biomass carbon in the litter moss layer. Different letters indicate a significant difference between treatment means of wood ash application rates (Tukey's test, $p < 0.05$). Error bars indicate standard error ($n = 5$ for the control and $N = 4$ for all other treatments)

et al., 1995; Reed et al., 2017). Perkiömäki (2004) reported an increase in microbial activity following wood ash application in terms of both mineralization rate and respiration rate in the humus (analogous to our forest floor) at their boreal site. It is, however, plausible that the number of statistical tests has introduced at least one false positive at alpha = 0.05. This is supported by additional inconsistencies in our analyses, where significant differences were noted for mineralizable C at the Mistik site (central Saskatchewan) and HWEC at the Pineland site, though there were no differences in MBC at either of these sites.

Although not a primary driver behind the wood ash applications in this study, pH was measured because of its role in regulating microbial activity and the solubility of many elements (e.g., Bååth et al., 1995). Although pH did increase at all sites with wood ash application, these increases were not always significant. In fact, there were no significant changes in soil pH in any of the soil layers at Aleza Lake North, Haliburton (central Ontario), Island Lake, nor any in the mineral soil at Pineland or the forest floor at the 25th Side Road and Eastern Township sites. Generally, the changes in soil pH are longer-term and are evident in deeper layers suggesting slow downward

transfer of base cations (Gömöryová et al., 2016; Reid & Watmough, 2014).

Our results suggest that the physio-chemical effects of wood ash application are relatively short lived in these soils and that higher rates of wood ash application or repeated applications may be required to produce changes in site productivity or soil quality. The Eastern Townships site had the highest rate of wood ash application in this study at 20 Mg ha^{-1}, which is beyond what is recommended in Europe (Hannam et al., 2019), and there appears to be no detrimental effects to soil quality based on the measured attributes. Differences were most pronounced in the LM layer, with none being evident in the mineral soil. The 25th Side Road also had a relatively high wood ash treatment (10 Mg ha^{-1}), where no measurable effects have been detected up to five years after application.

From our study, there were few indications that wood ash application at the rates applied (0.7–20.0 Mg ha^{-1}) would result in measurable effects to soil C or N pools. Our results suggest that the effects from wood ash application were inconsistent, infrequent, and small. Where it makes good economic sense, diverting wood ash from the landfill and applying it to forest soils at rates that do not exceed regulated concentrations for nutrients and heavy metals (i.e., tested and certified wood ash sources) should be encouraged in Canada.

5 | CONCLUSIONS

The application of wood ash at eight sites across a broad geographic range did not produce appreciable change in the SOM attributes examined in this study. The response of soil C fractions to wood ash application may be better explained by intrinsic site factors not examined here, as opposed to the type of wood ash used and rate of application. The sensitivity of labile C pools was not consistent across the sites and selection of indicators will likely need to be site specific. The observed changes, or lack thereof, suggest that the application of wood ash to forest soils maintains, or may even enhance, soil quality. Though few labile C fractions were responsive to wood ash application, these fractions may nevertheless assist with monitoring the impact of wood ash application which might not be detectable otherwise. Even though all indicators did not respond to the application of wood ash, they do support the finding that wood ash application either had no effect or enhanced soil quality (i.e. direction of change in indicator with ash application was consistent with an improvement to the soil property). Coupled with meta-analysis findings that wood ash utilization generally improves tree growth at longer (10 y) timescales across northern forests, this study supports the prudent use of wood ash as a forest soil amendment.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are openly available in the Dryad data repository (https://datadryad.org) at https://doi.org/10.5061/dryad.fbg79cnwr.

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REFERENCES

Armstrong, R. A. (2014). When to use the Bonferroni correction. *Ophthalmic and Physiological Optics*, *34*(5), 502–508. https://doi.org/10.1111/opo.12131

Bååth, E., Frostegård, Å., Pennanen, T., & Fritze, H. (1995). Microbial community structure and pH response in relation to soil organic matter quality in wood-ash fertilized, clear-cut or burned coniferous forest soils. *Soil Biology and Biochemistry*, *27*, 229–240. https://doi.org/10.1016/0038-0717(94)00140-V

Bera, T., Vardanyan, L., Inglett, K. S., Reddy, K. R., O'Connor, G. A., Erickson, J. E., & Wilkie, A. C. (2019). Influence of select bioenergy by-products on soil carbon and microbial activity: A laboratory study. *Science of the Total Environment*, *653*, 1354–1363. https://doi.org/10.1016/j.scitotenv.2018.10.237

Brais, S., Bélanger, N., & Guillemette, T. (2015). Wood ash and N fertilization in the Canadian boreal forest: Soil properties and response of jack pine and black spruce. *Forest Ecology and Management*, *348*, 1–14. https://doi.org/10.1016/j.foreco.2015.03.021

Bünemann, E. K., Bongiorno, G., Bai, Z., Creamer, R. E., De Deyn, G., de Goede, R., Fleskens, L., Geissen, V., Kuyper, T. W., Mäder, P., Pulleman, M., Sukkel, W., van Groenigen, J. W., & Brussaard, L. (2018). Soil quality—A critical review. *Soil Biology and Biochemistry*, *120*, 105–125. https://doi.org/10.1016/j.soilbio.2018.01.030

Cardoso, E. J. B. N., Vasconcellos, R. L. F., Bini, D., Miyauchi, M. Y. H., dos Santos, C. A., Alves, P. R. L., de Paula, A. M., Nakatani, A. S., Pereira, J. D. M., & Nogueira, M. A. (2013). Soil health: Looking for suitable indicators. What should be considered to assess the effects of use and management on soil health? *Scientia Agricola*, *70*, 274–289. https://doi.org/10.1590/S0103-90162013000400009

Carter, M. R., & Gregorich, E. G. (Eds.). (2008). *Soil sampling and methods of analysis*. CRC Press/Taylor & Francis.

Conant, R. T., Smith, G. R., & Paustian, K. (2003). Spatial variability of soil carbon in forested and cultivated sites: Implications for change detection. *Journal of Environmental Quality*, *32*, 278–286. https://doi.org/10.2134/jeq2003.2780

Demeyer, A., Voundi Nkana, J. C., & Verloo, M. G. (2001). Characteristics of wood ash and influence on soil properties and nutrient uptake: An overview. *Bioresource Technology*, *77*, 287–295. https://doi.org/10.1016/S0960-8524(00)00043-2

Doran, J. W., & Parkin, T. B. (1994). Defining and assessing soil quality. Defining soil quality for a sustainable environment (pp. 1–21), SSSA Special Publication. https://doi.org/10.2136/sssaspecpub35.c1

Emilson, C. E., Hannam, K., Aubin, I., Basiliko, N., Bélanger, N., Brais, S., & Hazlett, P. (2018). *Synthesis of current AshNet study designs and methods with recommendations towards a standardized protocol. Information report GLC-X-22*. Retrieved from https://cfs.nrcan.gc.ca/publications?id=39388

Gaudreault, C., Lama, I., & Sain, D. (2020). Is the beneficial use of wood ash environmentally beneficial? A screening-level life cycle assessment and uncertainty analysis. *Journal of Industrial Ecology*, *24*, 1300–1309. https://doi.org/10.1111/jiec.13019

Ghani, A., Dexter, M., & Perrott, K. W. (2003). Hot-water extractable carbon in soils: A sensitive measurement for determining impacts of fertilisation, grazing and cultivation. *Soil Biology and Biochemistry*, *39*, 1231–1243. https://doi.org/10.1016/S0038-0717(03)00186-X

Goh, K. M., Bruce, G. E., Daly, M. J., & Frampton, C. M. A. (2000). Sensitive indicators of soil organic matter sustainability in orchard floors of organic, conventional and integrated apple orchards in New Zealand. *Biological Agriculture & Horticulture*, *17*(3), 197–205. https://doi.org/10.1080/01448765.2000.9754842

Gömöryová, E., Tóthová, S., Pichler, V., Homolák, M., Kriššák, V., & Gömöry, D. (2016). Wood ash effect on chemical and microbiological properties of topsoil in a Norway spruce stand one year after the treatment. *Folia Oecologica*, *43*, 156–163.

Gregorich, E. G., Beare, M. H., McKim, U. F., & Skjemstad, J. O. (2006). Chemical and biological characteristics of physically uncomplexed organic matter. *Soil Science Society of America Journal*, *70*, 975–985. https://doi.org/10.2136/sssaj2005.0116

Gregorich, E. G., Monreal, C. M., Carter, M. R., Angers, D. A., & Ellert, B. H. (1994). Towards a minimum data set to assess soil organic matter quality in agricultural soils. *Canadian Journal of Soil Science*, *74*, 367–385. https://doi.org/10.4141/cjss94-051

Hannam, K. D., Fleming, R. L., Venier, L., & Hazlett, P. W. (2019). Can bioenergy ash applications emulate the effects of wildfire on upland forest soil chemical properties? *Soil Science Society of America Journal*, *83*(s1), S201. https://doi.org/10.2136/sssaj2018.10.0380

Hannam, K. D., Venier, L., Allen, D., Deschamps, C., Hope, E., Jull, M., Kwiaton, M., McKenney, D., Rutherford, P. M., & Hazlett, P. W. (2018). Wood ash as a soil amendment in Canadian forests: What are the barriers to utilization? *Canadian Journal of Forest Research*, *48*, 442–450. https://doi.org/10.1139/cjfr-2017-0351

Hannam, K. D., Venier, L., Hope, E., McKenney, D., Allen, D., & Hazlett, P. W. (2017). AshNet: Facilitating the use of wood ash as a forest soil amendment in Canada. *The Forestry Chronicle*, *93*, 17–20. https://doi.org/10.5558/tfc2017-006

Hansen, M., Saarsalmi, A., & Peltre, C. (2016). Changes in SOM composition and stability to microbial degradation over time in response to wood chip ash fertilisation. *Soil Biology and Biochemistry*, *99*, 179–186. https://doi.org/10.1016/j.soilbio.2016.05.012

Haynes, R. J. (2005). Labile organic matter fractions as central components of the quality of agricultural soils: An overview. In D. L. Sparks (Ed.), *Advances in agronomy* (Vol. *85*, pp. 221–268). Elsevier Academic Press Inc.

Hope, E. S., McKenney, D. W., Allen, D. J., & Pedlar, J. H. (2017). A cost analysis of bioenergy-generated ash disposal options in Canada. *Canadian Journal of Forest Research*, *47*, 1222–1231. https://doi.org/10.1139/cjfr-2016-0524

Huotari, N., Tillman-Sutela, E., Moilanen, M., & Laiho, R. (2015). Recycling of ash—For the good of the environment? *Forest Ecology and Management*, *348*, 226–240. https://doi.org/10.1016/j.foreco.2015.03.008

Jacobson, S., Lundström, H., Nordlund, S., Sikström, U., & Pettersson, F. (2014). Is tree growth in boreal coniferous stands on mineral soils affected by the addition of wood ash? *Scandinavian Journal of Forest Research*, *29*(7), 675–685. https://doi.org/10.1080/02827581.2014.959995

Janzen, H. H., Janzen, D. W., & Gregorich, E. G. (2021). The 'soil health' metaphor: Illuminating or illusory? *Soil Biology and Biochemistry*, *159*, 108167. https://doi.org/10.1016/j.soilbio.2021.108167

Karlen, D. L., Mausbach, M. J., Doran, J. W., Cline, R. G., Harris, R. F., & Schuman, G. E. (1997). Soil quality: A concept, definition, and framework for evaluation (a guest editorial). *Soil Science Society of America Journal*, *61*(1), 4–10. https://doi.org/10.2136/sssaj1997.03615995006100010001x

Karltun, E., Saarsalmi, A., Ingerslev, M., Mandre, M., Andersson, S., Gaitnieks, T., Ozolinčius, R., & Varnagiryte-Kabasinskiene, I. (2008). Wood ash recycling—Possibilities and risks. In D. Röser, A. Asikainen, K. Raulund-Rasmussen, & I. Stupak (Eds.), *Sustainable use of forest biomass for energy: A synthesis with focus on the baltic and nordic region* (pp. 79–108). Springer. https://doi.org/10.1007/978-1-4020-5054-1_4

Kuhnel, A., Wisemeier, M., Sporlein, P., Schilling, B., & Kogen-Knabner, I. (2019). Influence of drying vs. freezing of archived soil samples on soil organic matter fractions. *Journal of Plant Nutrition and Soil Science*, *182*, 772–781.

Larson, W. E., & Pierce, F. J. (1991). Conservation and enhancement of soil quality. Evaluation for sustainable land management in the developing world : Proceedings of the International Workshop on Evaluation for Sustainable Land Management in the Developing World, Chiang Rai, Thailand, 15–21 September, 1991. http://agris.fao.org/agris-search/search.do?recordID=US201301762465

McFee, W. W., Kelly, J. M. (Eds.). (1995). *Carbon forms and functions in forest soils*. Soil Science Society of America Inc. https://doi.org/10.1002/ldr.2743

Natural Resources Canada. (2017). *Bioenergy from biomass [WWW document]*. https://www.nrcan.gc.ca/forests/industry/bioproducts/13323accessed

Natural Resources Canada. (2020). Bioenergy from biomass. https://www.nrcan.gc.ca/our-natural-resources/forests-forestry/forest-industry-trade/forest-bioeconomy-bioenergy-biop/bioenergy-biomass/13323

Papendick, R. I., & Campbell, G. S. (1981). Theory and measurement of water potential. In J. F. Parr, W. R. Gardner, & L. F. Elliott

(Eds.), *SSSA Special Publications* (pp. 1–22). Water Potential Relations in Soil Microbiology. Soil Science Society of America. https://doi.org/10.2136/sssaspecpub9.c1

Paré, D., Rochon, P., & Brais, S. (2002). Assessing the geochemical balance of managed boreal forests. *Ecological Indictors*, *1*, 293–311.

Perkiömäki, J. (2004). Wood ash use in coniferous forests, a soil microbiological study into the potential risk of cadmium release, Forest Research Institute. Research papers 917. Metsäntutkimuslaitoksen tiedonantoja. *Joensuun Tutkimuskeskus, Joensuu*.

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

R Core Team. (2020). *R: A language and environment for statistical computing*. R Foundation for Statistical Computing. https://www.R-project.org/

Reed, E. Y., Chadwick, D. R., Hill, P. W., & Jones, D. L. (2017). Critical comparison of the impact of biochar and wood ash on soil organic matter cycling and grassland productivity. *Soil Biology and Biochemistry*, *110*, 134–142. https://doi.org/10.1016/j.soilbio.2017.03.012

Reeves, D. W. (1997). The role of soil organic matter in maintaining soil quality in continuous cropping systems. *Soil & Tillage Research*, *43*(1–2), 131–167. https://doi.org/10.1016/S0167-1987(97)00038-X

Reid, C., & Watmough, S. A. (2014). Evaluating the effects of liming and wood-ash treatment on forest ecosystems through systematic meta-analysis. *Canadian Journal of Forest Research*, *44*, 867–885. https://doi.org/10.1139/cjfr-2013-0488

Rice, C. W., Moorman, T. B., & Beare, M. (1997). Role of microbial biomass carbon and nitrogen in soil quality. *Methods for assessing soil quality* (pp. 203–215). John Wiley & Sons, Ltd. https://doi.org/10.2136/sssaspecpub49.c12

Saarsalmi, A., Mälkönen, E., & Kukkola, M. (2004). Effect of wood ash fertilization on soil chemical properties and stand nutrient status and growth of some coniferous stands in Finland. *Scandinavian Journal of Forest Research*, *19*(3), 217–233. https://doi.org/10.1080/02827580410024124

Saarsalmi, A., Smolander, A., Kukkola, M., Moilanen, M., & Saramäki, J. (2012). 30-Year effects of wood ash and nitrogen fertilization on soil chemical properties, soil microbial processes and stand growth in a Scots pine stand. *Forest Ecology and Management*, *278*, 63–70. https://doi.org/10.1016/j.foreco.2012.05.006

Shepard, R. K. (1997). Response of young black spruce (Picea mariana (Mill.) B.S.P.) to a mixture of wood ash and secondary papermill sludge. Maine agricultural and forest experiment station miscellaneous report no. 405. https://digitalcommons.library.umaine.edu/aes_miscreports/22/

Soil Classification Working Group. (1998). *The Canadian system of soil classification* (3rd ed.) (p. 187). Agriculture and Agri-Food Canada Publication.

Titus, B. D., Brown, K., Helmisaari, H.-S., Vanguelova, E., Stupak, I., Evans, A., Clarke, N., Guidi, C., Bruckman, V. J., Varnagiryte-Kabasinskiene, I., Armolaitis, K., de Vries, W., Hirai, K., Kaarakka, L., Hogg, K., & Reece, P. (2021). Sustainable forest biomass: A review of current residue harvesting guidelines. *Energy, Sustainability and Society*, *11*, 10. https://doi.org/10.1186/s13705-021-00281-w

Van Bich, N., Eyles, A., Mendham, D., Dong, T. L., Ratkowsky, D., Evans, K. J., Hai, V. D., Thanh, H. V., Thinh, N. V., & Mohammed, C. (2018). Contribution of harvest residues to nutrient cycling in a tropical *Acacia mangium* Willd. plantation. *Forests*, *9*, 577. https://doi.org/10.3390/f9090577

Ventura, M., Panzacchi, P., Muzzi, E., Magnani, F., & Tonon, G. (2019). Carbon balance and soil carbon input in a poplar short rotation coppice plantation as affected by nitrogen and wood ash application. *New Forests*, *50*(6), 969–990. https://doi.org/10.1007/s11056-019-09709-w

Weigel, A., Eustice, T., van Antwerpen, R., Naidoo, G., & Schulz, E. (2011). Soil organic carbon (SOC) changes indicated by hot water extractable carbon (HWEC). *Proceedings of the South African Sugar Technologists' Association*, *84*, 210–222.

Zagal, E., Munoz, C., Quiroz, M., & Cordova, C. (2009). Sensitivity of early indicators for evaluating quality changes in soil organic matter. *Geoderma*, *151*(3–4), 191–198. https://doi.org/10.1016/j.geoderma.2009.04.004

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