

**Effects of Wood Ash Amendments on Greenhouse Gas Production and Soil Solute
Dynamics in Hydromorphic Forest Soils of Northwestern Ontario**

by

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ABSTRACT

Wood ash recycling can return nutrients to forest soils, but responses in hydromorphic settings remain poorly characterized. This study examined how ash type and application rate affect pH and dissolved base cations, whether riparian and upland soils differ in methane (CH₄) concentrations, and how gas responses change during anaerobic incubation. Two boiler ashes (PB3 and PB6) were applied at nominal rates of 0, 5, 10, 20 and 50 t ha⁻¹ to five soil composites from northwestern Ontario: two riparian, two upland and one buffer source. Laboratory microcosms were incubated under N₂-flushed conditions for 60 days. Headspace CH₄ and carbon dioxide (CO₂) were measured on Days 10 and 34, and filtered-water chemistry was assessed on Day 60. Ash-amended pH values exceeded the corresponding untreated controls in all five soils by 0.78–2.53 units, including at the lowest rate, supporting the expectation that ash reduces acidity. Dissolved base-cation responses depended on the element, soil and ash: sodium increased in many amended treatments, while calcium and magnesium varied among soils and rates. The hypothesis of higher riparian CH₄ concentrations was partly supported: both riparian sources exceeded their paired upland sources at Day 10, but Black Spruce upland exceeded Black Spruce riparian at Day 34. Temporal gas responses varied among soils and treatments, consistent with the third hypothesis; CO₂ also increased in untreated jars, and neither gas showed a uniform response to increasing ash rate. Exploratory mercury measurements often showed lower filtered-water total mercury (THg) in amended samples, but methylmercury was not measured. Limited field replication and chemistry coverage, together with departures from gas-model assumptions, constrain inference. The findings identify soil- and ash-specific responses for field testing and water-quality monitoring, without establishing an operational application rate.

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CAUTION TO THE READER

This MScF thesis has been through a formal process of review and comment by two faculty members, two committee members, and an external examiner. It is made available for loan by the Faculty of Natural Resources Management for the purpose of advancing the practice of professional and scientific forestry.

The reader should be aware that the opinions and conclusions expressed in this document are those of the student and do not necessarily reflect the opinions of either the thesis advisor, external examiner, committee members, the faculty or Lakehead University.

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1.0 INTRODUCTION AND BACKGROUND

1.1 The Bioeconomy and Biomass Energy in Canada

Canada's forest sector plays an important role in the national bioeconomy and in efforts to reduce greenhouse gas emissions. Many pulp and paper mills, sawmills, and district heating systems now use woody biomass to produce heat and energy (Natural Resources Canada, 2024; Environment and Climate Change Canada, 2023). Burning this biomass produces large amounts of wood ash, which has traditionally been treated as waste and sent to landfill (Hannam et al., 2016). Recently, wood ash has been viewed as a potential resource rather than a waste product because it contains nutrients that are removed from forests during harvesting. Returning ash to forest soils could reduce disposal costs and help recycle nutrients back into ecosystems (Kim et al., 2022).

In practice, the use of wood ash in Canadian forests has been slower than in Nordic countries. This is partly because ash composition varies depending on fuel type and boiler technology, and because regulations differ among provinces (Hannam et al., 2017; Pitman, 2006). Ash produced from different boilers can vary widely in alkalinity, particle size, and trace metal content (Freire et al., 2015; Puri et al., 2024). When properly tested and managed, however, wood ash can improve soil conditions by increasing pH and supplying base cations such as calcium, magnesium, and potassium, as well as phosphorus. These effects are especially useful in acidic forest soils that have lost nutrients through harvesting and acid deposition (Kim et al., 2022; Hannam et al., 2019). Using ash in forests can therefore provide both economic benefits and ecological benefits in regions where soil buffering capacity has declined (Hannam et al., 2019; Pugliese et al., 2014).

The Canadian research program AshNet was developed by the Canadian Forest Service to coordinate existing and plan new field trials and improve standards for ash use (Hannam et al., 2017). These efforts showed that wood ash is not a uniform product and must be carefully tested before application. Its suitability depends on chemical composition, metal content, and site conditions (Pitman, 2006; Freire et al., 2015). Recent work from the AshNet network examined soil biotic responses to wood ash across eight sites in managed forests spanning multiple Canadian ecozones. Metabarcoding of arthropods, bacteria, fungi, and eukaryotic communities, combined with enzyme assays, showed no consistent shifts in community structure or function from amendments up to 20 Mg ha⁻¹. These results describe the managed forest sites examined; comparable responses in hydromorphic soils cannot be assumed (Smenderovac et al., 2022).

1.2 Nutrient Depletion and Base-Cation Dynamics in Boreal Forests

Boreal forests are often naturally low in available nutrients because of cold temperatures, slow rates of primary production and decomposition, and because many soils form on nutrient-poor parent materials (Kimmins, 1996; Krzic et al., 2021). Harvesting can increase nutrient loss, especially when whole trees are removed. Foliage and small branches contain large amounts of calcium, magnesium, and potassium, and removing them speeds up soil acidification and base-cation depletion (Pugliese et al., 2014). Over repeated harvest cycles, this nutrient loss can reduce forest productivity and affect soil microbes and mycorrhizal fungi that control carbon and nutrient cycling (Cheeke et al., 2017). In parts of eastern Canada, declining soil calcium has been linked to reduced forest health and productivity, leading to interest in wood ash as a possible calcium source (Kim et al., 2022).

Wood ash acts as a liming agent because it contains carbonates and oxides that neutralize acidity (Maresca et al., 2017). When ash dissolves in soil water, it raises pH and adds calcium and other base cations to exchange sites (Wang et al., 1998). Studies in Scandinavia and Canada show that ash application can increase soil pH by 0.5 to 1.5 units and improve tree nutrient status when applied at suitable rates (Huotari et al., 2015; Hannam et al., 2019). Operational guidance also emphasizes setbacks from water bodies to reduce the movement of ash-derived solutes and particulates into surface waters. Biological responses can vary among soils and ash materials (Smenderovac et al., 2022).

Wood ash can also supply phosphorus, which becomes more available as acidity decreases in acidic mineral soils (Escudey et al., 2010). However, ash can temporarily raise salt concentrations and electrical conductivity, which may stress plants and microbes if application rates are too high or if ash is applied without chemical and physical pre-treatment (Freire et al., 2015; Baloch et al., 2024). As a result, published guidance recommends stabilizing ash (for example by pelletizing or weathering it) and applying it in stages based on soil type and site conditions, especially near surface waters (Hannam et al., 2016).

1.3 International Precedents and the Canadian Context

Finland and Sweden have several decades of experience applying wood ash to forest soils, providing useful guidance for Canadian programs (Pärn et al., 2010). Studies in these countries show that treated ash can raise soil pH and increase calcium and magnesium in ways similar to low-severity fire, without damaging soil physical structure, meaning particle arrangement and aggregate stability (Pitman, 2006; Huotari et al., 2015). Nordic and Canadian guidance

emphasize careful site selection and setbacks from water bodies to reduce ash-derived solute movement, metal leaching, and runoff (Hannam et al., 2016; Pitman, 2006; Huotari et al., 2015).

Applying these lessons in Canada requires attention to local hydrology, including connections between wetlands and the surrounding landscape (Hokanson et al., 2020). Canadian studies show that large quantities of ash are available from biomass and pulp and paper facilities (Meyer, 2022) and that many forests show signs of calcium decline and acidification (Deighton & Watmough, 2020). However, barriers remain, including differences in provincial regulations, limited economic incentives for ash use, and a lack of long-term monitoring data (Hannam et al., 2016). Some multi-site studies suggest that upland soil biological communities show little to no detectable change under well-characterized ash applications at operational rates (Smenderovac et al., 2022), but responses in wetlands and riparian soils are still poorly understood, with limited directly comparable evidence for hydromorphic Canadian forest soils. These environments play a key role in controlling water quality and material export from headwaters, making them especially important for future study.

1.4 Ash Composition, Quality Assurance, and Soil Biogeochemical Responses

Wood ash has a complex chemical composition. Along with organic carbon from incomplete burnout and inorganic carbon, hydrogen, and oxygen, from a soil nutrient and contaminant perspective it is mainly made up of calcium (Ca), potassium (K), magnesium (Mg), and phosphorus (P), but it also contains trace metals and metalloids depending on the type of fuel burned, contamination by soil or sand, boiler design and air-pollution control systems, and ash handling practices such as water quenching or outdoor versus indoor storage (Someshwar, 1996; Pugliese et al., 2014). Recent studies show large variation in ash alkalinity, particle size, and salt

content both within and among facilities, which affects how ash behaves when applied to soils (Freire et al., 2015). For this reason, ash characterization should include not only total element concentrations but also leaching tests, acid neutralization capacity (ANC), and physical properties related to particle size and reactivity.

In acidic mineral soils, ash addition can raise pH, reduce exchangeable aluminum (Al), and increase calcium and magnesium availability (Albuquerque et al., 2022). Higher pH can also increase the availability of phosphorus in strongly to moderately acidic soils (Krzic et al., 2021). Wood ash can increase dissolved nutrient concentrations and crop uptake, although individual elements respond differently (Jian et al., 2025). However, organic and poorly drained soils with higher dissolved organic carbon (DOC) and low-oxygen, reducing conditions may respond differently (Knorr, 2013).

1.5 Environmental Risks

A key concern with soil application of wood ash is the potential release of trace elements (Maresca et al., 2017). Although total metal concentrations in ash often meet guideline limits, the amount that can leach into soil and water depends on pH, dissolved organic carbon (DOC), and redox conditions. After application, short-term increases in salinity and alkalinity can increase the mobility of metals such as Cd and Zn. In saturated, reducing soils, dissolution and reduction of iron and manganese oxides can mobilize metals that were previously associated with soil or ash particles (Knorr, 2013). These risks highlight the importance of cautious application near surface waters and of monitoring both dissolved and particulate metals in runoff and shallow groundwater.

Mercury (Hg) is a concern because its retention in soil and presence in water depend on the chemical environment. In wetland soils, inorganic mercury binds strongly to reduced sulfur groups in organic matter, while competition with inorganic sulfides affects its solubility and distribution between solid and aqueous phases (Skylberg, 2008). These reactions provide a basis for examining whether ash amendment changes the mercury concentration in filtered water. Total mercury (THg) measures the combined mercury forms in the analyzed sample and is the mercury endpoint used in this study.

Filtered-water THg was compared among ash treatments and untreated samples to assess whether ash addition was associated with higher or lower mercury concentrations in the water phase. This measurement addresses one part of mercury mobility; actual export from soil also depends on water movement and retention along flow paths. Methylmercury (MeHg) was not measured, so the study does not test mercury methylation.

For this reason, ash application in parts of the landscape that can hydrologically connect to DOC-rich riparian soils requires careful evaluation (Knorr, 2013), and small, unmapped hydromorphic depressions and headwater wetlands that drain into waters already under mercury-based fish-consumption advisories may represent particularly sensitive locations (Ontario Ministry of the Environment, Conservation and Parks, 2022).

1.6 Riparian and Wetland Systems as Biogeochemical Hotspots

Riparian buffers and forested wetlands influence the movement of dissolved organic carbon and solutes from headwater areas to streams. High water tables, low oxygen conditions and abundant organic matter support anaerobic decomposition. Changes in water levels and redox conditions can affect the release of DOC and iron from wetland soils and their subsequent

transport to streams (Knorr, 2013). These soil–water connections make hydromorphic settings relevant to the study of ash-derived solutes and filtered-water THg. The incubation examines chemical responses within the soil–water mixture; field transport requires separate assessment.

Soil organic carbon storage in freshwater forested wetlands is often poorly represented in carbon accounting frameworks, leading to underestimation of both mitigation potential and vulnerability (Sapkota et al., 2025). Given that wood ash can modify soil pH, ionic strength, and nutrient availability, it may indirectly affect carbon storage and export through changes in microbial activity.

1.7 Knowledge Gaps and Rationale for the Study

Canadian wood-ash research has largely examined managed upland forest settings, including the multi-site AshNet network (Hannam et al., 2017; Smenderovac et al., 2022). Hydromorphic depressions, riparian soils and wetland margins present additional questions because high water content and hydrologic connections can alter the fate of ash constituents. Ash particles or dissolved constituents could reach these settings through wind redistribution, runoff or groundwater transport even when ash is applied elsewhere in a harvested landscape. This study examines the response of selected soils under common anaerobic incubation conditions rather than testing an operational application rule or setback distance.

The limited evidence for wood ash use in hydromorphic Canadian forest soils leaves a specific question about mercury: how do filtered-water THg concentrations differ between amended and untreated soils? Organic-rich wet soils provide a setting in which mercury binding to organic matter and sulfur compounds may influence the amount present in water (Skylberg, 2008). Comparing THg across soil sources, ash types and rates provides an exploratory

assessment of this response alongside pH, base cations and other dissolved elements. The experiment measures endpoint concentrations rather than mercury export from a catchment.

Anaerobic microbial metabolism in boreal wetland soils includes methane production (methanogenesis) carried out by strictly anaerobic methanogenic archaea, as a terminal step of anaerobic decomposition under strongly reduced conditions (Bräuer et al., 2020). When sulfate is available (for example from atmospheric deposition or potentially from wood ash), anaerobic sulfate reducing bacteria (SRB) commonly outcompete methanogens for shared substrates such as H₂ and acetate because sulfate reduction is thermodynamically more favorable and can proceed at lower substrate concentrations than methanogenesis (Sela-Adler et al., 2017). Anaerobic decomposition processes in boreal wetlands often operate near the lower thermodynamic limit for microbial energy gain and depend on tight symbiosis among fermenters, SRB, methanogens, and iron reducers; these communities are highly sensitive to changes in pH, organic matter quality, trace nutrients needed for metalloenzymes such as methyl coenzyme M reductase (MCR), macronutrient availability, and the supply of alternative electron acceptors (Bräuer et al., 2020).

Wood ash chemistry varies substantially with boiler technology and feedstock, leading to large differences in pH and base-cation concentrations among ashes (Pugliese et al., 2014). As a result, ash applications can either improve soil pH and nutrient status and support forest productivity or, at higher loadings, cause excessive alkalinization and increase the mobility of certain metals (Pitman, 2006). Field studies further show that application rate and the hydrological and erosional transport of ash control how far these chemical changes propagate into the soil profile and how strongly microbial communities are altered (Bang-Andreasen et al., 2021). Consequently, when ash constituents are transported into hydromorphic soils and

wetlands, a similarly wide range of potential positive and negative outcomes for wetland soil microbial biogeochemistry and downstream water quality can be expected.

In northwestern Ontario, biomass-energy development provides a regional context for examining alternatives to ash disposal. Federal support for a Whitesand First Nation energy facility illustrates the growing role of woody biomass in community energy systems (Natural Resources Canada, 2023). Reusing ash requires evidence about both nutrient recycling and potential effects on soils connected to surface waters.

This laboratory study examines how two biomass boiler ashes (PB3 and PB6), applied at 0, 5, 10, 20 and 50 t ha⁻¹, affect pH, headspace CH₄ and CO₂ concentrations, filtered-water THg, and other dissolved elements in five soils from northwestern Ontario. The soils include riparian, upland and buffer locations with different moisture contents, organic matter and initial chemistry. All soils received the same high-moisture, N₂-flushed incubation treatment.

1.7.1 Research Questions and Hypotheses

The study first asks how ash type and application rate influence pH and dissolved base-cation concentrations in the sampled soils. The hypothesis is that ash addition increases pH and base-cation concentrations compared with untreated soil, with the size of the response differing between PB3 and PB6. The untreated control and four application rates allow comparisons across doses, while using both ashes at each rate allows their effects to be compared directly. The pH results are assessed descriptively because only one endpoint value is available for each amended treatment and one control value is reported for each soil source.

A second question is whether the sampled riparian and upland soils respond differently to ash under the same anaerobic incubation conditions. The hypothesis is that CH₄ concentrations

are higher in the riparian soils, given their wet field conditions and organic substrates. The two riparian–upland pairs allow this comparison in two local settings, with the buffer soil providing additional context. These five sources provide a starting point for understanding differences among soils, but do not represent all riparian and upland soils in Ontario.

The study also asks whether changes in gas concentrations between Days 10 and 34 depend on soil source, ash type and application rate. The hypothesis is that these changes differ among soils and ash treatments. Sampling the same numbered jars on both dates allows changes within each jar to be assessed. Filtered-water THg measurements provide a separate, exploratory comparison among soil sources, ash types and application rates at the end of the incubation.

The results of this work are intended to support practical management by identifying candidate rate ranges and monitoring indicators for subsequent field validation, rather than establishing safe operational rates or setback distances from this incubation alone. More broadly, this study contributes boreal-specific evidence to discussions on sustainable ash recycling in Canada, including its potential role in policy and Indigenous-led land stewardship.

2.0 METHODS

2.1 Study Area

This study was conducted in northwestern Ontario, Canada, with soils from Crown forest stands under the Dog River–Matawin and Black Spruce Forest Management Units. Black Spruce (BS) and Dog River–Matawin (DM) identify the sampling areas; comparisons focused on landscape position and measured soil properties. The sampled locations lie within the boreal shield ecozone, characterized by glacially derived soil parent materials, conifer-dominated forests, and a cool continental climate (Environment and Climate Change Canada [ECCC],

2024). The Dog River–Matawin Forest lies approximately 55 km northwest of Thunder Bay, while the Black Spruce Forest is located roughly 60 km north of Thunder Bay and extends several hundred kilometers northwest (Fig. 1). “Dog Mat” in tables and figure labels is an abbreviation for Dog River–Matawin.

The sampled stands included black spruce (*Picea mariana*), jack pine (*Pinus banksiana*), trembling aspen (*Populus tremuloides*) and balsam fir (*Abies balsamea*). Field observations identified contrasts between upland positions and riparian or wetland margins (Table 1). These local observations and measured soil properties provide the ecological context for the experiment.

Within the Black Spruce Forest, three locations were selected in consultation with the Ontario Ministry of Natural Resources and Forestry (OMNRF) and Resolute Forest Products to represent a moisture and topographic gradient: upland, buffer, and riparian zones. These sites spanned coarse mineral uplands, transitional slopes with intermittent saturation, and saturated peat-influenced riparian soils. Field sampling was completed on September 30th, 2024. Study locations were accessed via active and secondary forestry roads originating west of Kakabeka Falls (Fig. 1).

Two complementary sampling locations were selected within the Dog River–Matawin (DM) area to expand the range of physiographic and hydrological conditions included in the incubation study. These sites contain upland conifer sites that had been recently harvested and site-prepared/planted, as well as riparian lowland zones underlain by a mosaic of shallow till, peat, and periodically saturated soils. Sampling took place on November 5, 2024 and included perspectives on sampling location from OMNRF, Canadian Forest Service, and Lakehead University aquatic and terrestrial scientists. Across all sites, organic layers were visibly thinner

in upland settings (~5 cm) and extended to over 10 cm depth in upland hydromorphic and riparian areas with accumulating organic matter.

At each location, GPS coordinates, dominant vegetation, and qualitative hydrologic indicators were recorded (Table 1). Soil samples were collected and sealed in clean Ziploc bags and transported to the Lakehead University Soils Laboratory the same day. Samples were stored under refrigeration until laboratory processing.

The region has a legacy of periodic harvesting, wildfire, and site preparation which has likely reduced base cation pools and acidified surface horizons relative to pre-harvest conditions (Hannam et al., 2017). Combined with high organic matter content and seasonal saturation in riparian soils, these conditions provide a relevant testing ground for evaluating whether wood ash amendments can replenish cations, elevate pH, and alter microbial activity relevant to GHG flux and mercury cycling.

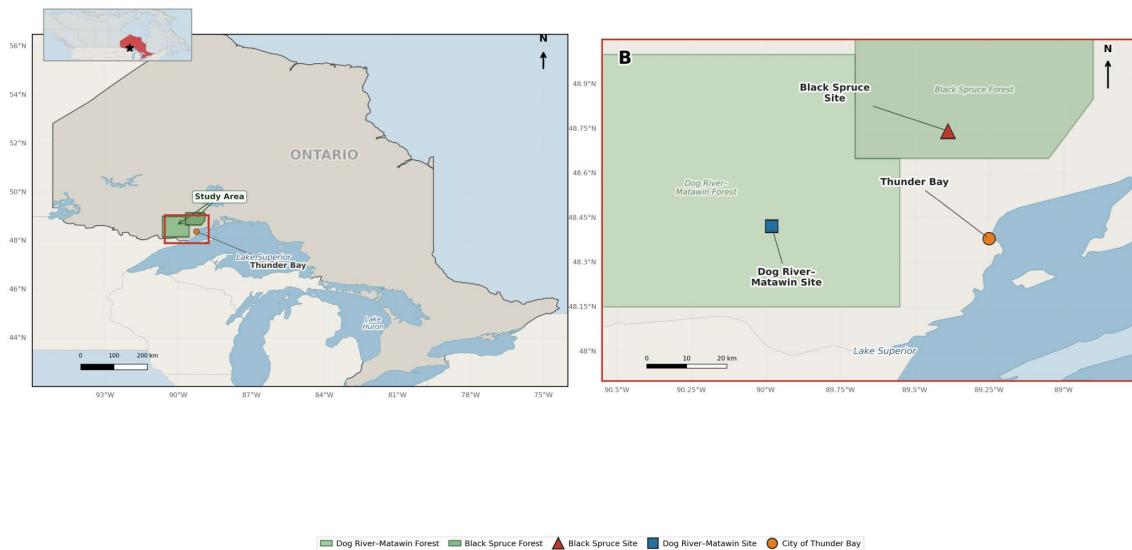


Figure 1. Map of the study area in northwestern Ontario, Canada, showing the Dog River-Matawin Forest and the locations of the sampled sites used in this study.

2.2 Experimental Design

The laboratory experiment compared five homogenized soil composites, representing two riparian sources, two upland sources and one buffer source (Table 1). Each source received PB3 and PB6 at four nonzero nominal rates (5, 10, 20 and 50 t ha⁻¹), with five jars per ash–rate combination, and ten untreated jars. This produced 50 jars per source and 250 jars overall. The two nominal zero-dose ash groups were pooled as one untreated control because neither received ash. The design therefore contained nine physical treatments per soil source. Gas concentrations were measured in the same numbered jars on Days 10 and 34; filtered-water chemistry was assessed after 60 days. The jars provide laboratory replication within each composite, while the five composites provide the soil-source comparisons. Soil preparation is described below, and jar assembly and sampling are detailed in Section 2.4. Laboratory methods were adapted from earlier boreal soil studies in collaboration with the Lakehead University Soils Laboratory, OMNRF staff and the Canadian Forest Service (CFS). Analyses were conducted between October 2024 and 2026.

Table 1. Site descriptions for soils collected by sampling area and landscape position.

Site ID	Sampling area	Landscape Position	Latitude (°N)	Longitude (°W)
BS-S1	Black Spruce Forest	Riparian	48.74194	-89.38805
BS-S2	Black Spruce Forest	Buffer	48.74203	-89.38712
BS-S3	Black Spruce Forest	Upland	48.74215	-89.38635
DM-A1	Dog River–Matawin Forest	Riparian	48.42142	-89.98027

Site ID	Sampling area	Landscape Position	Latitude (°N)	Longitude (°W)
DM-A2	Dog River–Matawin Forest	Upland	48.4207	-89.97806

Table 1 continued. Remaining columns for the same samples.

Site ID	Dominant Vegetation and Ground Cover	Qualitative Hydrologic Indicators
BS-S1	Black spruce with sparse understory shrubs and moss cover	Saturated soil surface adjacent to stream channel; evidence of periodic flooding and shallow water table
BS-S2	Mixed conifer stand with herbaceous understory and discontinuous moss layer	Moderately moist mineral soil; transitional zone between upland and riparian hydrology
BS-S3	Recently harvested clearcut (1–2 years post-harvest) with regenerating shrubs	Well-drained mineral soil; no visible surface water or saturation
DM-A1	Dense shrub and deciduous sapling layer with grasses and mosses adjacent to stream	Standing water and saturated organic soils; visible surface flow
DM-A2	Early successional upland vegetation dominated by herbaceous cover	Dry to moderately moist mineral soil; no standing water

At each location, soil was collected from the upper 0–10 cm using an auger, trowel and spade. Multiple scoops across an approximately 10 × 10 m area were combined to form a composite soil source. Samples were transported to Lakehead University and refrigerated at approximately 4 °C until processing.

One homogenized composite represented each soil source. Scoops collected within a site were subsamples; jars prepared from the composite were laboratory treatment replicates. The design included two riparian sources, two labelled upland and one buffer source. Landscape position, moisture, organic matter and initial chemistry provided the basis for comparing the

soils. Sources from the same area may share environmental history, and collection dates differed between areas. BS-S3 was labelled “upland wet” in the laboratory report and well-drained upland in the field table. It is identified here as BS upland, with its drainage classification treated cautiously. Soil source was included as a fixed factor to compare the five sampled composites. The two riparian–upland comparisons are descriptive because the study has limited independent field replication. Testing these patterns at additional sites would help determine whether landscape position and soil properties predict responses more broadly.

Field-moist soils were processed by removing visible roots, stones and coarse debris, sieving through a 5 mm mesh and thoroughly homogenizing the remaining material. Soils were refrigerated before incubation. Collection dates differed between the two settings, so storage history is a potential source-level influence on subsequent laboratory responses.

Gravimetric moisture was determined from wet and oven-dry subsample masses, and organic matter was estimated by loss on ignition (LOI). Table 3 reports these soil-source characteristics alongside initial pH, carbon and nitrogen. Gas concentrations were not normalized by soil mass.

Baseline soil pH was measured on one representative subsample per site using a 1:4 soil-to-water slurry (10 g soil, 40 mL distilled water). Slurries were shaken manually for ~2 minutes, allowed to settle for ~10 minutes, and measured using a calibrated benchtop pH meter with pH 4.00, 7.00, and 10.00 buffers. Procedures followed those used in boreal microcosm studies by Gupta et al. (2013) and Godin et al. (2012).

To minimize cross contamination among sites, sealed Ziploc bags were used for each soil, and samples were stored under refrigeration on the sampling days at ~4 °C until processing.

During sieving and homogenization, coarse roots and stones were removed using gloved hands. All samples were handled on clean laboratory benches, and sieves were brushed clean and rinsed between soil types. Drying and combustion instruments were monitored for temperature stability, and balances were verified using internal laboratory standards.

Data provenance and exclusions were handled separately for each assay. BS-S3 is reported as BS upland while retaining the uncertainty between its laboratory label (“upland wet”) and field drainage description. Solid-phase characterizations use the laboratory report’s summary columns, with analytical duplicates treated as quality-control observations rather than field replicates. For gases, missing peak areas were excluded, recorded numeric zeros were retained, and only complete Day 10–34 jar pairs entered the repeated-measures models (233 for CH₄ and 221 for CO₂); descriptive figures use available readings. For inductively coupled plasma–optical emission spectrometry (ICP-OES), both chemically distinct records numbered 198 were excluded because neither could be assigned uniquely, and non-numeric records from other projects were omitted. The remaining 156 records were matched to the numbered treatment lookup; non-detects were retained as censored values, not zeros. THg observations were grouped by their recorded source, ash and rate because two records had conflicting sample numbers and replicate labels. Unverified common jar identities prevented a pH–THg correlation. Endpoint pH lacked individual replicate readings. Untreated nominal ash groups were pooled for gas and ICP summaries; both untreated THg observations per source were displayed individually. Assay-specific counts and analytical limitations are detailed in Sections 2.4–2.9 and Tables 4–8.

2.3 Pre-Incubation Soil and Ash Characterization

A preliminary pH screening trial was completed before the full incubation using a wider range of ash additions to help select the final application rates. Those screening results were used for experimental design and are not presented as a separate results dataset.

Prior to incubation, representative subsamples of each field-collected soil ($n = 5$ sites) and both wood ash materials (PB3, PB6) were analyzed at the Lakehead University Environmental Laboratory for solid-phase elemental and nutrient analysis. These analyses provided baseline chemical context for interpreting post-incubation porewater responses to ash amendment. Additionally, the mill where the power boilers operate in Thunder Bay provided product-label information on agricultural liming use. Table 2 presents measured ash chemistry, and Table 3c presents product-label context separately.

Samples were analyzed for a broad suite of major nutrients, trace metals, and macronutrients including total organic carbon (TOC), total nitrogen (TN), sulfur, phosphorus, nitrate/nitrite species, and trace metals relevant to ash dissolution and geochemical cycling. The targeted elemental suite included base cations (Ca, Mg, K, Na), transition metals (Fe, Mn, Zn, Cu, Ni, Cr, Co), metalloids (As, Se), and additional elements associated with both soil mineralogy and wood-ash composition (Al, Ti, V, Pb, Cd, Ba, Sr), as listed in the analytical report (Tables 3a and 3b).

The solid-phase laboratory report provides elemental concentrations in $\mu\text{g g}^{-1}$ and percentages for carbon, nitrogen and sulfur. Original and duplicate determinations are analytical duplicates, not independent field or ash-batch replicates. Tables 2, 3a and 3b use the report's summary-column values, which use the original result for many elements rather than a consistent

mean of duplicates. Quality-control recoveries varied by analyte and included values outside 80–110%; these characterizations are therefore used as compositional context rather than replicated estimates of material variability.

Initial soil and ash chemistry helps explain differences in the pH and dissolved elements measured after incubation. The ash contained higher concentrations of several base cations and nutrients than the soils.

Table 2. Identity and measured chemistry of the two experimental ash materials. Laboratory values are from the summary columns of ML25-0101; they describe these materials, not replicated samples of boiler output.

Measurement or identity	PB3	PB6
Material identity	Power Boiler 3 ash	Power Boiler 6 ash
TOC (%)	23.33	18.56
N (%)	0.11	0.12
S (%)	0.07	1.92
Ca ($\mu\text{g g}^{-1}$)	82,629.7	99,246.6
Mg ($\mu\text{g g}^{-1}$)	10,767.0	12,182.0
K ($\mu\text{g g}^{-1}$)	21,099.6	20,462.8
Na ($\mu\text{g g}^{-1}$)	1,719.0	5,153.8
Total P ($\mu\text{g g}^{-1}$)	4,086.1	7,128.9
pH, ANC, particle size	Not supplied	Not supplied

Product-label values are reported separately in Table 3c. Agricultural Index and application limits are product information, not measurements of neutralizing capacity for the experimental batches. Ash pH, acid-neutralization capacity, particle-size distribution and dissolution rate were not measured in the datasets analyzed here.

2.4 Microcosm Incubations and Microbial Activity Measurements

Microcosms were constructed in 250 mL wide-mouth glass mason jars (Bernardin, Canada) modified for headspace sampling. Lids were fitted with drilled openings sealed with butyl-rubber septa to maintain airtight conditions during incubation and gas sampling. All jars and lids were washed with Alkanox laboratory detergent, rinsed thoroughly with tap water followed by distilled water (5x), and allowed to air dry. Components were handled with nitrile gloves to minimize contamination.

Each microcosm contained 30 g of homogenized field-moist soil mixed with PB3 or PB6 ash at application-rate equivalents of 0, 5, 10, 20 and 50 t ha⁻¹. The conversion used a reference soil depth of 10 cm and bulk density of 1.3 g cm⁻³, equivalent to 1,300 t of soil per hectare. Target ash mass was calculated as $30 \times R/1,300$, where R is the application rate in t ha⁻¹ and the result is in grams. The calculated targets were approximately 0, 0.115, 0.231, 0.462 and 1.154 g ash per jar, respectively, for both ashes. The reference bulk density was a dosing assumption, not a measurement of each soil. This conversion was applied to the 30 g field-moist soil addition; it did not adjust for differences in dry soil mass among sources. Each jar received 60 mL deionized water, giving a 2:1 water-to-soil ratio (2 mL g⁻¹), and was incubated under N₂-flushed conditions.

The incubation comprised 250 numbered jars: five soil sources × two nominal ash groups × five rates × five laboratory replicates. Each nonzero soil × ash × rate treatment contained five jars. The two nominal zero-dose groups contained ten distinct untreated jars per soil and were combined into one physical control group. The physical design therefore comprised five sources × nine treatments: one control and eight ash–dose combinations. Unique numeric jar IDs were

used for matching observations; sample text alone was not unique across soil sources. Available observations and assay-specific sample sizes are reported below and in the accompanying data tables. Records of random assignment, jar placement and handling order were unavailable, so the model did not include blocks.

Following assembly, jars were connected to a manifold and repeatedly evacuated with a two-stage vacuum pump (Yellow Jacket model 93546 through 23-gauge needles) and flushed with ultra-high-purity N₂ gas (99.999%) to remove atmospheric oxygen (Gupta et al., 2013). Jars were allowed to return to atmospheric pressure after the final N₂ flush, and headspace flushing and sealing were performed immediately after soil loading to minimize oxygen exchange.

Incubations proceeded for 60 days under static conditions at 20.5 °C. Headspace samples were collected during the incubation using 10 mL gas-tight syringes with stopcocks and 25-gauge needles inserted through the septa. Removed headspace gas was replaced with high-purity nitrogen to maintain pressure in the microcosms, and 10 mL of gas was analyzed for methane (CH₄) and carbon dioxide (CO₂).

Gas observations were available at Days 10 and 34 for the same numbered jars. CH₄ had 240 and 243 observations at the two dates, respectively, and CO₂ had 226 and 244. Complete pairs were available for 233 CH₄ jars and 221 CO₂ jars. Missing peak areas were excluded rather than converted to zero by downstream formulas. Recorded numeric zeros were retained. No Day 60 gas observations were available for analysis.

2.5 Gas Chromatography Analysis

Gas samples were analyzed at the Lakehead University Soils Laboratory using an SRI Instruments 310 gas chromatograph equipped with a 1 mL sampling loop/injector, a 1/8-inch packed column (Hayesep D packing), and a flame ionization detector (FID) with methanizer (SRI Instruments, Torrance, California, USA). The FID detects methane directly, while the inline nickel-catalyst methanizer converts carbon dioxide to methane so that CO₂ can be quantified as a second FID peak. The GC was operated under standard laboratory settings with controlled detector, injector, and oven temperatures following Godin et al. (2012), except that H₂ was used as both carrier gas and FID fuel. Calibration was performed using a certified mixed-gas standard containing 10.81 ppm CH₄ and 1008 ppm CO₂ in air. Calibration checks were conducted at the beginning of each analytical session, and mid-run verification injections were used to assess detector stability and instrument drift.

Headspace concentrations are reported in parts per million by volume (ppm, also denoted ppmv). Recorded peak areas were converted using $\text{CH}_4 \text{ ppm} = \text{area} \times 10.81/45.55$ and $\text{CO}_2 \text{ ppm} = \text{area} \times 1008/3442$. The factors were applied to both dates. Means and standard errors were calculated across individual jars within each source, ash and rate combination; untreated jars were pooled once per source. Headspace volumes were not recorded. Volume is not needed for this concentration conversion. Gas mass, cumulative production and production rates were not calculated. Concentration is the analytical outcome because it follows directly from calibrated peak areas without estimating gas volume. It describes the incubation atmosphere at sampling, not gross microbial production: consumption, dissolution and replacement of sampled gas can also influence it. The single-point conversion assumes a proportional response through zero; separate run-specific calibration slopes or drift corrections could not be reconstructed from the

supplied records. Applying this factor to concentrations above the standard also assumes that detector response remains linear across the measured range.

Quality control for gas analyses included the use of headspace blanks (i.e., jars without soils) and the certified gas standards. Samples exhibiting implausible retention times or detector response (e.g., leaks, syringe misfires) were rerun.

2.6 Post-Incubation Filtration and pH of Solutes

Microcosms were destructively sampled on Day 60 for filtered-water chemistry. Jars were filtered and analyzed individually; filtrates were not composited across replicate jars. The available records contain fewer results than the original jar allocation, with assay-specific counts reported below. In this thesis, dissolved or porewater concentrations refer to the water passing through the 0.5 μm filter, which may include small colloids.

Filtration was conducted using oven-baked glass-fibre filter papers (0.5 micrometer retention, 2.5 cm diameter; Macherey-Nagel, Düren, Germany) placed into reusable Teflon filter holders. Filters were baked at 105 °C to remove residual volatiles. Filtration was performed using disposable 30-mL polypropylene Luer-lock syringes, each attached to the filter holder assembly. Teflon filter holders were rinsed thoroughly with distilled water before initial use and between each sample to minimize cross-contamination.

Filtrate was collected into new, sterile 50-mL plastic vials, capped immediately and stored under refrigerated conditions (~4 °C) until further analysis. A separate aliquot from each treatment was set aside for total mercury determination, while remaining filtrate was retained for elemental analysis by ICP-OES.

Endpoint pH was measured with a calibrated Denver Instruments UltraBasic meter. The dataset contains 40 amended-treatment values: five soil sources $\times$ two ashes $\times$ four nonzero rates, with one value per combination. Untreated Day 60 pH was 5.63 for BS riparian, 5.71 for BS buffer, 5.24 for BS upland, 6.78 for DM riparian and 5.67 for DM upland. Initial-soil pH is reported separately in Table 3. Individual replicate readings and the number of measurements behind each control value were not recorded in the available pH data. Treatment comparisons are therefore descriptive.

2.7 Total Mercury (THg) Analysis

Total mercury (THg) in filtered porewater samples was analyzed using cold-vapor atomic fluorescence spectrometry following the Brooks Rand BRL standard operating procedure for total mercury in aqueous samples. THg was used to compare mercury concentrations in the filtered-water fraction among ash treatments and untreated samples. It provides an endpoint measure of mercury in that fraction, without distinguishing individual mercury species. MeHg was not analyzed, so THg differences cannot be interpreted as changes in methylation.

For THg analysis, aliquots of approximately 10 mL of each filtrate were transferred into 40-mL borosilicate I-Chem vials with Teflon-lined caps (EPA-certified to Method 1631 standards). Samples were refrigerated at $\sim 4^\circ\text{C}$ prior to digestion. Two procedural blanks consisting of ultrapure water were processed alongside samples to evaluate contamination introduced during filtration, digestion, or handling.

Immediately prior to analysis, residual oxidant was neutralized using hydroxylamine hydrochloride to prevent interference with subsequent reduction by stannous chloride.

Following quenching, samples were analyzed using a Brooks Rand MERX-T automated cold-vapor atomic fluorescence spectrometer (CVAFS) operated according to manufacturer specifications. The instrument reduces Hg^{2+} to volatile elemental mercury (Hg^0) using SnCl_2 , purges liberated Hg^0 into the detector with inert carrier gas (argon), and quantifies vapor-phase mercury via fluorescence response. Aqueous THg concentrations were calculated using calibration responses generated within the MERX-T system and reported as ng L^{-1} , corrected for procedural blanks.

The THg dataset contains 50 observations: 40 amended values and ten untreated values. Each amended soil $\times$ ash $\times$ rate combination has one observation, and each soil has two untreated observations. Sample numbers and replicate labels disagree for two records, so mercury measurements could not be reliably matched with pH measurements from the same jars. THg results were grouped using the recorded soil, ash and rate labels. Laboratory blank and recovery results were not included in the dataset.

2.8 Soluble-Phase Elemental Analysis (Metals, Base Cation Elements, and S)

Elemental chemistry of post-incubation filtrates was characterized using inductively coupled plasma–optical emission spectrometry (ICP–OES) on an Agilent 5800 system at the Lakehead University Instrument Laboratory. Analytes included Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn; detection status is summarized in Table 7. These analyses were used to assess ash dissolution or associated changes in nutrient solubility (base cations, P, S), and potential mobilization or immobilization/precipitation of metals from soils. Two ultrapure-water filtration blanks

accompanied each sample batch to evaluate contamination introduced during filtration, digestion, and storage.

2.9 Statistical Analysis

Data preparation and statistical analyses were conducted in Python and R programs. Statistical significance was assessed at $\alpha = 0.05$. Gas concentrations were transformed as $\ln(1 + \text{concentration in ppm})$. Model assumptions were assessed using residual plots, Shapiro–Wilk tests for residual normality and Brown–Forsythe (median-based Levene) tests for equal residual variance. These checks identified departures from the assumptions, so the ANOVA results are interpreted as exploratory.

2.9.1 pH Responses

Day 60 pH is summarized separately for PB3 and PB6 at 5, 10, 20 and 50 t ha⁻¹, with one untreated endpoint value per soil source at zero dose. Each amended value is compared descriptively with its soil’s endpoint control. The available readings do not provide a measure of variation within each treatment.

2.9.2 Greenhouse Gas Concentrations

Gas concentrations were analyzed using a repeated-measures approach because the same jars were sampled on Days 10 and 34. Jar number was used to match the two readings. The analysis included 233 CH₄ jars and 221 CO₂ jars with measurements on both dates. Concentrations were transformed as $y = \ln(1 + C)$, where C is the concentration in ppm. The analysis separately examined differences among jars and changes within each jar, including soil source, ash treatment and their interactions. This avoids treating two measurements from the

same jar as independent replicates. Jars missing one date were excluded from the statistical tests, but their available readings contributed to the means shown in the figures. This exclusion could affect the results if missing readings were related to the treatment response. With only two dates, no sphericity correction is needed.

With two sampling dates, the transformed readings from each jar were expressed as a scaled average and a scaled change. The sum of the Day 10 and Day 34 readings and the Day 34 minus Day 10 difference were each divided by the square root of two. Separate soil-source $\times$ treatment models were fitted to these responses, with 45 cells representing five sources and nine physical treatments. The first model tests differences among jars; the second tests changes within jars. The intercept in the change model tests the mean difference between dates, weighting treatment cells equally. Source and treatment comparisons also give equal weight to physical treatment cells.

Ash rate was analyzed as five separate treatment levels: 0, 5, 10, 20 and 50 t ha⁻¹ rather than assuming a straight-line response as the rate increased. The rate test therefore has four degrees of freedom. The analysis also compared PB3 with PB6 across the four amended rates and tested whether the difference between the ashes changed with application rate. These tests have one and three degrees of freedom, respectively. The untreated control was included once because no ash was added to it. The model also tested whether these treatment patterns differed among soil sources and between sampling dates. Table 4 reports sums of squares (SS), degrees of freedom (df), mean squares (MS), F statistics, p-values and experimental error. Each test accounts for the other terms in the model; the reported partial sums of squares therefore do not add up to the total sum of squares.

For each amended rate, a model column assigned -2 to untreated jars, $+1$ to both ashes at that rate and 0 to other treatments. Ash type was coded -1 for PB3, $+1$ for PB6 and 0 for untreated jars. Three orthogonal contrasts represented differences in the ash comparison among the four amended rates. Interactions with soil source completed the 45-cell model. Between-jar and within-jar tests used separate experimental error terms.

The repeated-measures framework was retained to account for paired observations from the same jar and to separate differences among jars from changes between sampling dates. Unlike endpoint pH and amended-treatment THg, the gas data contain replicate jars within treatment cells, allowing residual variation to be estimated. This replication supports fitting the model but does not remove the consequences of non-normality or unequal variance. Soil source was treated as fixed because the aim was to compare these five selected composites; a random-source term would not supply the missing independent field replication. The contrast coding also avoids assigning a physical ash type to untreated jars. These are reasons for the model structure, not evidence that conventional F tests are robust to the observed diagnostic failures. No rank-based or permutation reanalysis is presented here; the ANOVA is retained as an exploratory summary alongside the concentration patterns, and the p-values are not treated as confirmatory evidence.

Model checks included Shapiro–Wilk tests for residual normality and Levene tests for differences in residual spread (Table 5). Small treatment groups, recorded zeros, missing readings and unavailable run-specific quality-control records limit the analysis. The tests assess overall patterns and were not adjusted for multiple comparisons; they do not identify which individual ash treatments differ from their controls. Comparisons among soil sources apply to the selected composites rather than to all soils within a sampling area or landscape position.

2.9.3 Porewater Chemistry

Porewater samples were analyzed by inductively coupled plasma–optical emission spectrometry (ICP-OES) at Lakehead University. A total of 30 analytes were measured; however, the results presented here focus on seven elements of primary interest for understanding ash-driven changes in soil porewater chemistry: the base cations calcium (Ca), magnesium (Mg), potassium (K), and sodium (Na), as well as sulfur (S), manganese (Mn) and phosphorus (P).

Of 158 numeric-labelled ICP records, two chemically different entries shared sample number 198 and could not be assigned uniquely. Both entries were excluded without averaging or selecting either one. The remaining 156 records were linked to the numbered soil-source and treatment lookup. Controls were pooled once per source, with available counts in Table 8. Given incomplete treatment coverage, small control groups and censoring, results are descriptive. Non-detects are retained as censored observations; detected-only means and SE are accompanied by detected/total counts and do not estimate the full treatment mean when non-detects occur.

2.9.4 Total Mercury (THg)

THg is summarized descriptively by soil source, ash type and rate using 50 observations. A single observation per amended treatment does not provide an estimate of within-treatment variance. A jar-level pH–THg correlation was not calculated because the two assays could not be linked to verified common jar identifiers. Pooling treatments across soils would also mix source differences with within-source associations.

3.0 RESULTS

3.1 Pre-incubation soil and ash characterization

The five soils differed in initial pH, moisture, LOI, carbon and nitrogen (Tables 2-3c). PB3 had higher TOC than PB6, while PB6 had higher Ca, Mg, S and total P. These differences provide a chemical basis for comparing the two ashes.

Table 3. Initial soil characteristics. pH, moisture and LOI characterize each source. TOC and N follow the solid-phase laboratory report, and C:N is calculated as TOC/N. Values are source characterizations, not field-replicate means.

Sampling area	Landscape Position	Original pH	Gravimetric Moisture (%)	LOI (%)
Black Spruce	Riparian	5.47	140.5	23.5
Black Spruce	Buffer	5.61	67.7	59.6
Black Spruce	Upland	4.79	40.2	27.0
Dog Mat	Riparian	5.73	208.8	71.1
Dog Mat	Upland	5.44	29.7	6.6

Table 3 continued. Remaining columns for the same samples.

Sampling area	Landscape Position	TOC (%)	N (%)	C:N (TOC/N)
Black Spruce	Riparian	11.71	0.95	12.33
Black Spruce	Buffer	20.22	1.53	13.22
Black Spruce	Upland	14.90	1.03	14.47
Dog Mat	Riparian	9.76	0.85	11.48
Dog Mat	Upland	1.86	0.13	14.31

Dog River–Matawin riparian soil had the highest gravimetric moisture (208.8%) and LOI (71.1%), while Dog River–Matawin upland soil had the lowest LOI (6.6%). These contrasts indicate that the incubation included both organic-rich, hydrologically wet soils and more

mineral upland soils, which is important for interpreting ash dissolution and solute chemistry under anaerobic conditions.

Table 3a. Pre-incubation solid-phase major chemistry of soils and ash materials. Values are dry-mass concentrations unless reported as percentages.

Sample/material	TOC (%)	TN (%)	S (%)	Ca ($\mu\text{g g}^{-1}$)
Dog River–Matawin riparian (A1)	9.76	0.85	0.12	8,168.0
Dog River–Matawin upland (A2)	1.86	0.13	0.01	1,908.1
Black Spruce riparian (S1)	11.71	0.95	0.11	6,066.8
Black Spruce buffer (S2)	20.22	1.53	0.13	8,564.8
Black Spruce upland wet (S3)	14.90	1.03	0.07	4,688.8
PB3 ash	23.33	0.11	0.07	82,630
PB6 ash	18.56	0.12	1.92	99,247

Table 3a continued. Remaining columns for the same samples.

Sample/material	Mg ($\mu\text{g g}^{-1}$)	K ($\mu\text{g g}^{-1}$)	Na ($\mu\text{g g}^{-1}$)	Total P ($\mu\text{g g}^{-1}$)
Dog River–Matawin riparian (A1)	4,002.8	450.5	261.1	794.0
Dog River–Matawin upland (A2)	3,977.0	405.1	179.1	473.4
Black Spruce riparian (S1)	5,542.9	568.5	175.2	829.5
Black Spruce buffer (S2)	3,790.9	760.7	150.8	673.1
Black Spruce upland wet (S3)	2,830.9	684.5	70.00	539.7
PB3 ash	10,767	21,100	1,719.0	4,086.1
PB6 ash	12,182	20,463	5,153.8	7,128.9

Table 3b. Pre-incubation solid-phase trace metals and related elements of soils and ash materials. Values are dry-mass concentrations.

Sample/material	Al ($\mu\text{g g}^{-1}$)	Fe ($\mu\text{g g}^{-1}$)	Mn ($\mu\text{g g}^{-1}$)	Cd ($\mu\text{g g}^{-1}$)	Pb ($\mu\text{g g}^{-1}$)
Dog River–Matawin riparian (A1)	14,591	20,096	367.8	0.41	12.94
Dog River–Matawin upland (A2)	20,196	23,977	196.6	<0.20	5.69
Black Spruce riparian (S1)	20,098	44,257	2,213.8	0.95	47.84
Black Spruce buffer (S2)	15,336	17,439	1,774.2	0.91	41.84
Black Spruce upland wet (S3)	9,529.5	17,815	760.4	0.33	17.86
PB3 ash	9,149.8	9,975.0	3,277.4	3.16	3.26
PB6 ash	17,679	12,863	2,588.9	12.83	11.50

Table 3b continued. Remaining columns for the same samples.

Sample/material	Cu ($\mu\text{g g}^{-1}$)	Ni ($\mu\text{g g}^{-1}$)	Zn ($\mu\text{g g}^{-1}$)	Cr ($\mu\text{g g}^{-1}$)	As ($\mu\text{g g}^{-1}$)
Dog River–Matawin riparian (A1)	32.00	21.52	49.92	42.76	3.11
Dog River–Matawin upland (A2)	11.96	27.79	35.69	35.81	1.72
Black Spruce riparian (S1)	54.46	53.77	180.1	55.39	7.49
Black Spruce buffer (S2)	101.4	56.21	231.1	28.19	7.21
Black Spruce upland wet (S3)	21.36	21.39	84.67	20.19	3.47
PB3 ash	35.98	11.43	431.0	14.42	<1.50
PB6 ash	71.24	22.67	1,261.6	35.58	5.78

Baseline solid-phase chemistry showed that the ash materials were strongly enriched in base cations, sulfur, and total phosphorus relative to most soils, while also containing measurable trace/heavy metals of environmental concern (Tables 3a and 3b). PB6 generally had higher Ca, Mg, S, total P, Cd, Zn, Cr, Cu, Ni, Pb, and As than PB3, whereas PB3 had slightly higher Mn. These pre-incubation values provide the context for interpreting the post-incubation porewater chemistry and the low dissolved trace-metal detection frequencies reported below.

Table 3c. Product-label information compiled for PB3-only and combined PB3/6 ash products. These values describe the labelled products, not measurements or approvals for the individual experimental PB6 batch.

Parameter	PB3-only product	Combined PB3/6 product	Source/notes
Agricultural Index (minimum)	25	36	TBPP wood-ash labels
Maximum dry-tonne application rate	20 t ha ⁻¹ per 12 months	7.4 t ha ⁻¹ per 12 months	TBPP wood-ash labels
Feedstock / source description	Power Boiler #3; primarily conifer bark with occasional wood chips	Power Boiler #3 and 6 label material; primarily conifer bark with occasional wood chips	TBPP wood-ash labels
Ash appearance / carbon description	Black, high-carbon ash with combusted and partially combusted wood pieces	Label describes black high-carbon ash; interpreted here as combined PB3/6 product-label information rather than PB6-specific chemistry	TBPP wood-ash labels

Parameter	PB3-only product	Combined PB3/6 product	Source/notes
Use guidance	Use as pH adjustment for low-pH soils; incorporate into soil as soon as possible; observe setbacks from surface water, tile inlets, and wells	Same label guidance	TBPP wood-ash labels
Total P in laboratory solid-phase analysis	4,086.1 $\mu\text{g g}^{-1}$	7,128.9 $\mu\text{g g}^{-1}$	Laboratory solid-phase analysis
Trace/heavy metals of concern in laboratory solid-phase analysis	Cd 3.16; Pb 3.26; Cu 35.98; Ni 11.43; Zn 431.0; Cr 14.42; As <1.50 $\mu\text{g g}^{-1}$	Cd 12.83; Pb 11.50; Cu 71.24; Ni 22.67; Zn 1,261.6; Cr 35.58; As 5.78 $\mu\text{g g}^{-1}$	Laboratory solid-phase analysis

3.2 pH Response

Figure 2 shows 40 ash-specific Day 60 pH values and five untreated Day 60 values. PB3 and PB6 are displayed separately, with one control per source at zero dose. Initial-soil pH is retained in Table 3. Endpoint pH in amended samples ranged from 6.76 to 8.16, compared with untreated Day 60 values of 5.24–6.78. The amended ranges were 7.15–8.16 for BS riparian, 7.56–8.02 for DM riparian, 6.76–7.65 for BS upland, 7.08–7.57 for DM upland, and 7.01–7.37 for BS buffer. All 40 amended values exceeded their corresponding endpoint control by 0.78–2.53 pH units, consistent with an increase in pH following ash addition. pH did not consistently increase as the ash rate rose from 5 to 50 t ha⁻¹. For example, BS buffer pH with PB3 declined from 7.37 at 5 t ha⁻¹ to 7.01 at 50 t ha⁻¹. Differences between PB3 and PB6 also varied among soils and application rates.

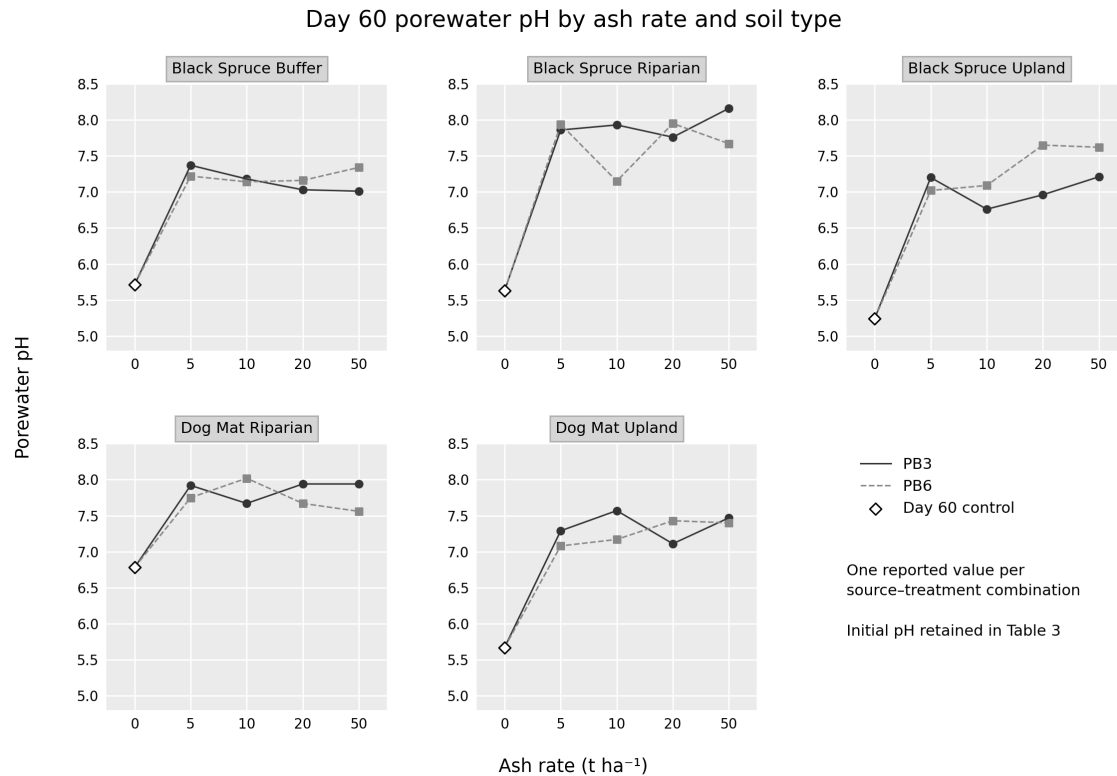


Figure 2. Day 60 pH by soil source, ash type and application rate. Diamond markers at 0 t ha^{-1} show untreated Day 60 controls. Solid lines with circles show PB3, and dashed lines with squares show PB6. One reported value is shown for each soil and treatment combination. Initial pH is reported separately in Table 3. Application rates are spaced equally along the x-axis. BS = Black Spruce; DM = Dog River–Matawin.

3.3 Microbial Activity Responses

Figures 3-6 show mean headspace concentrations $\pm$ SE for PB3 and PB6. The ten untreated jars per soil were combined into one control group. The error bars describe variation among laboratory jars. At Day 10, mean CH_4 concentrations were higher in both riparian soils than in the upland soil from the same area when the nine-treatment means were given equal weight. At Day 34, BS upland had a higher mean than BS riparian, while DM upland remained lower than DM riparian. The riparian–upland pattern therefore changed during incubation. Differences between the two riparian–upland pairs show why landscape position and individual soil

properties both need to be considered when interpreting the response to ash. Differences in CH₄ concentration between PB3 and PB6 varied among soils and rates. Increasing ash rate did not produce the same pattern in every soil. The exploratory repeated-measures analysis identified CH₄ differences among sources and between dates (Table 4). Between-jar ash and rate tests gave $F_{1,188} = 33.51$ ($p < 0.001$) and $F_{4,188} = 2.87$ ($p = 0.024$), respectively. Several source and time interactions were also present. Interpretation of these overall tests follows the limitations summarized in Table 5 and Section 2.9.2.

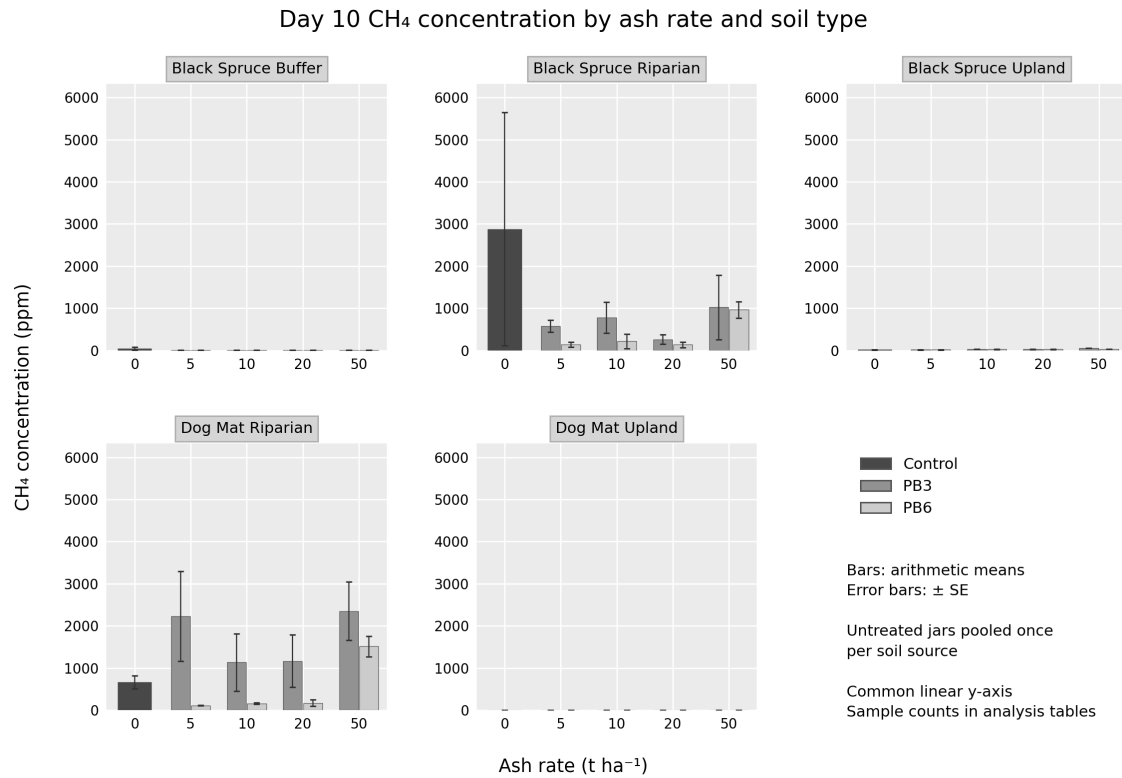


Figure 3. Day 10 CH₄ concentrations (ppm) by soil source, ash type and rate. Bars are arithmetic means $\pm$ SE. PB3 and PB6 are distinguished at nonzero rates; untreated jars are pooled once per source at zero dose. All panels use the same linear y-axis within each figure. Missing peak areas are excluded and numeric zeros are retained. Sample counts accompany the analysis tables.

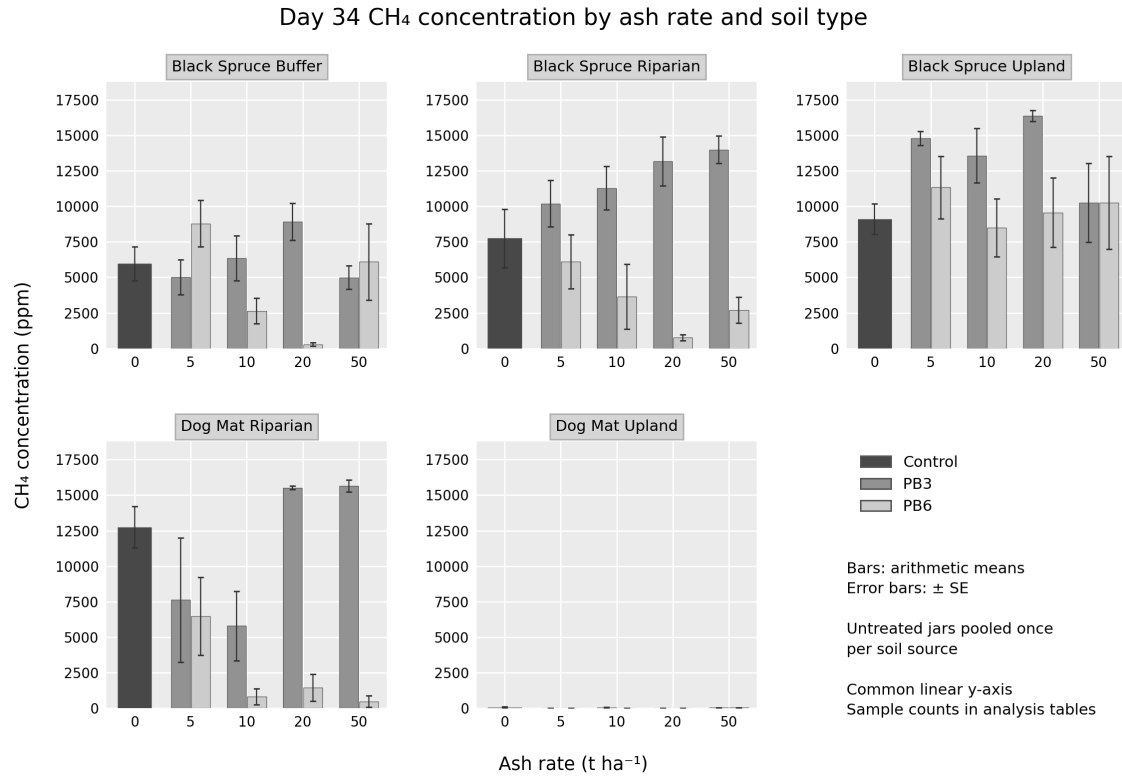


Figure 4. Day 34 CH₄ concentrations (ppm) by soil source, ash type and rate. Bars are arithmetic means $\pm$ SE. PB3 and PB6 are distinguished at nonzero rates; untreated jars are pooled once per source at zero dose. All panels use the same linear y-axis within each figure. Missing peak areas are excluded and numeric zeros are retained. Sample counts accompany the analysis tables.

CO₂ concentrations also varied among soils and ash treatments. BS upland had the highest mean on both sampling dates when the nine treatment means were given equal weight (Figures 5 and 6). Equal-treatment mean CO₂ concentrations ranged from approximately 13,585 to 31,353 ppm among sources at Day 10 and from 19,217 to 34,750 ppm at Day 34. These summaries give each of the nine physical treatments equal weight. The source-specific patterns are shown in Figures 5 and 6. The between-jar F test for CO₂ rate gave $p = 0.0015$. Diagnostic limitations are summarized in Table 5.

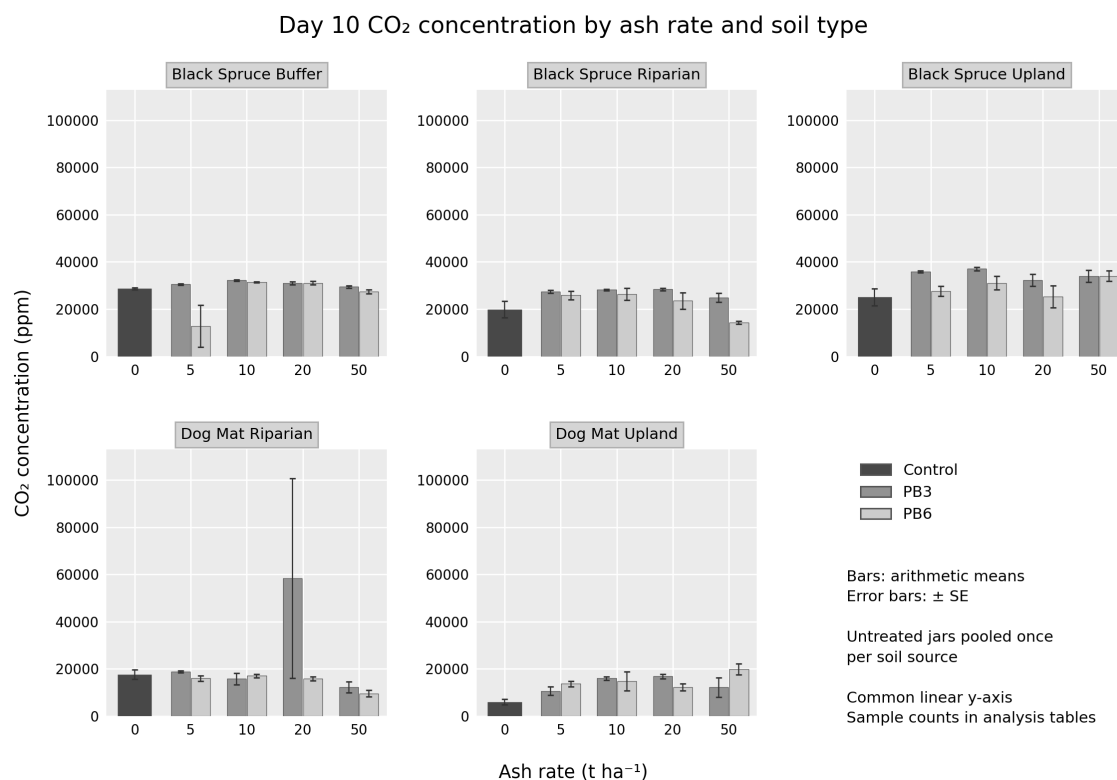


Figure 5. Day 10 CO₂ concentrations (ppm) by soil source, ash type and rate. Bars are arithmetic means $\pm$ SE. PB3 and PB6 are distinguished at nonzero rates; untreated jars are pooled once per source at zero dose. All panels use the same linear y-axis within each figure. Missing peak areas are excluded and numeric zeros are retained. Sample counts accompany the analysis tables.

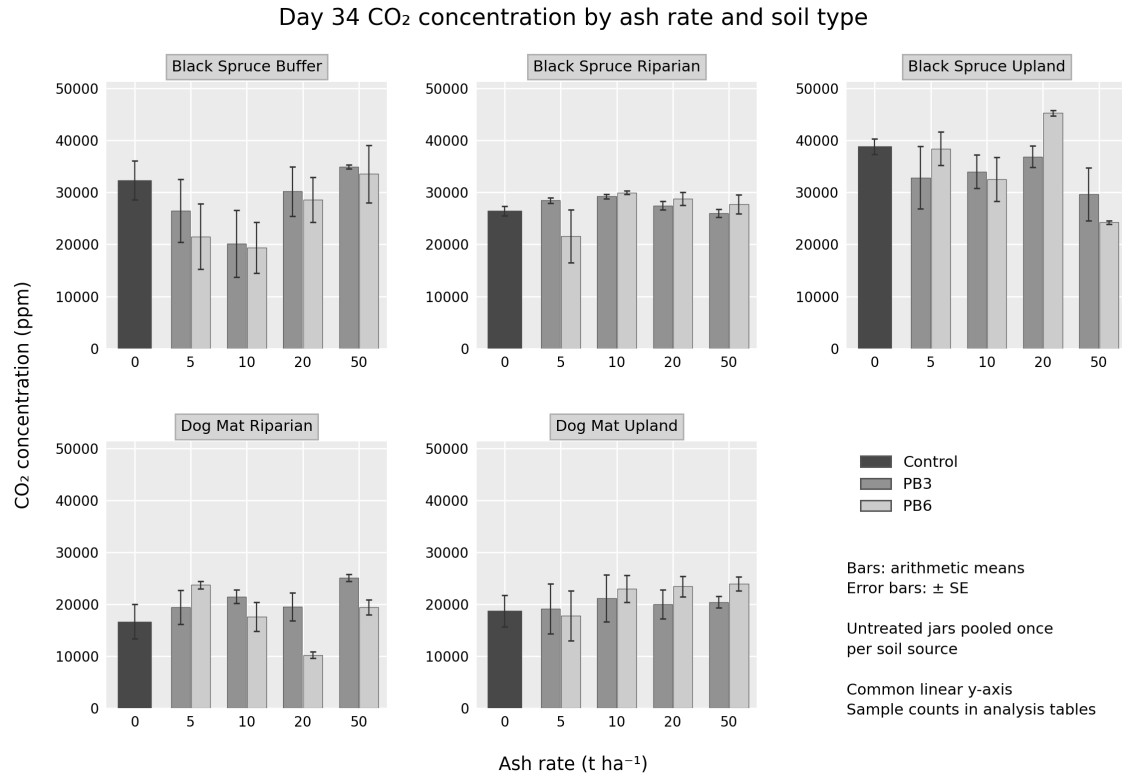


Figure 6. Day 34 CO₂ concentrations (ppm) by soil source, ash type and rate. Bars are arithmetic means $\pm$ SE. PB3 and PB6 are distinguished at nonzero rates; untreated jars are pooled once per source at zero dose. All panels use the same linear y-axis within each figure. Missing peak areas are excluded and numeric zeros are retained. Sample counts accompany the analysis tables.

Table 4. Exploratory repeated-measures ANOVA of gas concentrations on Days 10 and 34, transformed as $\ln(1 + C)$, where C is concentration in ppmv.

Gas / stratum	Term	SS	df	MS	F	Error df	p
CH4 B	Soil source	1.65e+03	4	413	186	188	<0.001
CH4 B	Rate	25.5	4	6.38	2.87	188	0.024
CH4 B	Ash	74.5	1	74.5	33.5	188	<0.001
CH4 B	Ash $\times$ rate	16.3	3	5.42	2.44	188	0.066
CH4 B	Soil $\times$ rate	53	16	3.31	1.49	188	0.107
CH4 B	Soil $\times$ ash	65	4	16.3	7.31	188	<0.001
CH4 B	Soil $\times$ ash $\times$ rate	37.7	12	3.14	1.41	188	0.163

Gas / stratum	Term	SS	df	MS	F	Error df	p
CH4 B	Experimental error	418	188	2.22	—	188	—
CH4 W	Day	1.47e+03	1	1.47e+03	1.08e+03	188	<0.001
CH4 W	Day × soil source	472	4	118	86.9	188	<0.001
CH4 W	Day × rate	14.8	4	3.69	2.71	188	0.031
CH4 W	Day × ash	29.1	1	29.1	21.4	188	<0.001
CH4 W	Day × ash × rate	15.7	3	5.23	3.85	188	0.011
CH4 W	Day × soil × rate	46.8	16	2.92	2.15	188	0.008
CH4 W	Day × soil × ash	14.3	4	3.58	2.63	188	0.036
CH4 W	Day × soil × ash × rate	26	12	2.17	1.6	188	0.096
CH4 W	Experimental error	255	188	1.36	—	188	—
CO2 B	Soil source	36.7	4	9.18	15.5	176	<0.001
CO2 B	Rate	10.8	4	2.71	4.59	176	0.002
CO2 B	Ash	0.759	1	0.759	1.28	176	0.259
CO2 B	Ash × rate	0.324	3	0.108	0.183	176	0.908
CO2 B	Soil × rate	13.7	16	0.857	1.45	176	0.123
CO2 B	Soil × ash	1.87	4	0.467	0.79	176	0.533
CO2 B	Soil × ash × rate	5.32	12	0.444	0.751	176	0.700
CO2 B	Experimental error	104	176	0.591	—	176	—
CO2 W	Day	3.29	1	3.29	4.95	176	0.027
CO2 W	Day × soil source	3.87	4	0.967	1.45	176	0.218
CO2 W	Day × rate	4.23	4	1.06	1.59	176	0.179
CO2 W	Day × ash	0.819	1	0.819	1.23	176	0.269
CO2 W	Day × ash × rate	0.893	3	0.298	0.448	176	0.719

Gas / stratum	Term	SS	df	MS	F	Error df	p
CO2 W	Day × soil × rate	17.6	16	1.1	1.65	176	0.060
CO2 W	Day × soil × ash	0.412	4	0.103	0.155	176	0.961
CO2 W	Day × soil × ash × rate	2.4	12	0.2	0.301	176	0.989
CO2 W	Experimental error	117	176	0.665	—	176	—

The complete-pair models contain 45 soil-source × physical-treatment cells. Separate between-jar and within-jar errors have 188 df for CH₄ and 176 df for CO₂. SS are partial (conditional contrast) sums of squares; MS = SS/df and conventional F = MS/error MS. All p-values are unadjusted and exploratory. The five rate levels are compared with four df; ash × rate has three df because no physical ash type exists at zero dose. Model assumptions and the distinction between concentration and production limit inference. B = between jars; W = within jars. The within-jar model intercept tests Day, with equal weight across the nine physical treatments.

Table 5. Diagnostics for the $\ln(1 + \text{concentration in ppmv})$ repeated-measures strata. Shapiro–Wilk tests describe residual normality; median-centred Levene tests compare residual spread among the 45 source–treatment cells.

Gas	Stratum	Jars	SW W	SW p	Levene F	Levene p
CH ₄	Between jars	233	0.905	<0.001	2.4	<0.001
CH ₄	Within jars	233	0.946	<0.001	1.77	0.005
CO ₂	Between jars	221	0.714	<0.001	0.96	0.548
CO ₂	Within jars	221	0.736	<0.001	0.874	0.695

Residuals departed from normality in all four analyses, and residual variation differed among treatments in both CH₄ analyses. The statistical results are therefore interpreted as exploratory.

3.4 Soluble mercury responses to ash additions

The 50 THg observations range from 1.14 to 26.0 ng L⁻¹ (Figure 7). Source-specific ranges are 1.14–3.05 for BS riparian, 1.74–6.00 for DM riparian, 8.56–26.0 for BS upland, 2.98–18.1 for DM upland, and 6.75–13.2 ng L⁻¹ for BS buffer. BS upland includes the largest measured concentrations. Many amended THg values were lower than the untreated values from the same soil. Differences between PB3 and PB6 varied among soils and rates. Figure 7 shows individual THg measurements, including the two untreated observations per soil at zero dose. The descriptive basis of these comparisons is set out in Section 2.9.4.

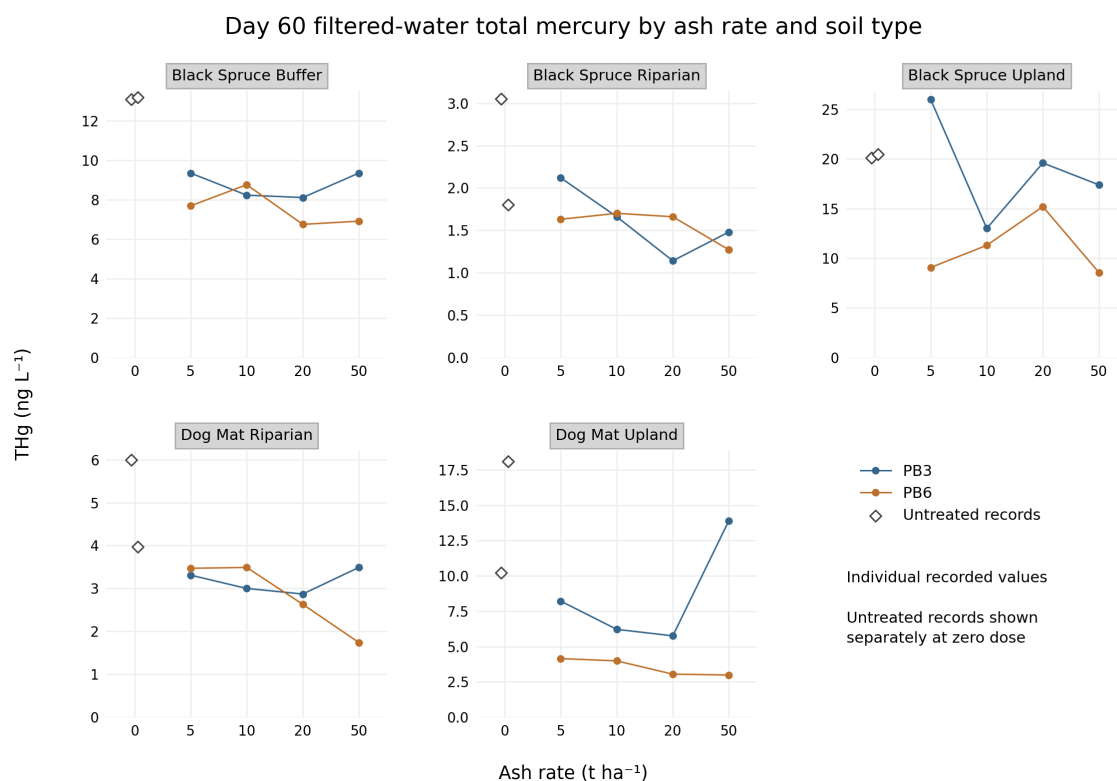


Figure 7. Recorded THg concentrations in filtered water, separated by ash type at nonzero rates. Each amended point is one measured record ($n = 1$); two nominal untreated observations per source are shown individually as diamond markers. Panel y-axis ranges differ.

3.5 Responses of other metals, S, P, and base-cation elements to ash additions

In addition to Ca, Mg, K, Na, S, and Mn, the soluble-phase dataset was screened for trace/heavy metals of environmental concern (Cd, Pb, Cu, Ni, Zn, Cr, and As) and for P. These analytes are included in Table 7 because low or non-detectable concentrations are still relevant for interpreting environmental risk from ash-amended porewater under anoxic incubation conditions. Because most trace/heavy metal observations were below the method detection/reporting limit, these analytes are summarized by detection frequency and detected range rather than by full treatment-level mean tables.

After excluding the two records with ambiguous sample number 198, 156 ICP observations remained. Tables 6 and 7 summarize concentrations and detection frequencies, and Table 8 reports assay availability by source and treatment. Na concentrations increased in many amended treatments, and S concentrations were high in several PB6 treatments. Ca and Mg responses differed among soils and application rates. Several PB6 treatments had high concentrations at 50 t ha⁻¹. These patterns describe the available observations; treatment coverage and sample counts are given in Table 8.

K and Mn responses varied among treatments. Where some results were below the reporting limit, the mean includes only detected concentrations. The detected and total sample counts show how much of each treatment is represented.

3.5.1 Primary post-incubation porewater concentration data

The ICP summary uses numeric Solution Label records linked to the jar and treatment lookup. Both ambiguous entries labelled 198 and non-numeric records from other projects were

excluded. Aqueous ICP concentrations are reported in ppm. Values are retained as reported; no additional dilution multiplier is applied in this analysis.

Table 6. Filtered-water chemistry from 156 ICP records after excluding both ambiguous entries labelled 198. Controls are pooled once per source. Values are detected-only mean $\pm$ SE with detected/total n; one detection has no SE. Concentrations are in ppm. Non-detects are not substituted with zero.

Soil	Ash	Rate	Ca	Mg	K
BS buffer	—	0	43.6 1/1	13.7 1/1	15.3 1/1
BS buffer	PB3	5	56.7 $\pm$ 1.59 2/2	17.4 $\pm$ 0.175 2/2	25.9 $\pm$ 0.845 2/2
BS buffer	PB3	10	57.2 $\pm$ 3.16 2/2	17.4 $\pm$ 0.715 2/2	35.2 $\pm$ 0.66 2/2
BS buffer	PB3	20	78.3 $\pm$ 3.48 3/3	24.3 $\pm$ 1.13 3/3	102 $\pm$ 37.9 3/3
BS buffer	PB3	50	43.6 $\pm$ 1.34 2/2	13.7 $\pm$ 0.26 2/2	64.5 $\pm$ 48.8 2/2
BS buffer	PB6	5	52.1 $\pm$ 1.18 2/2	15.9 $\pm$ 0.295 2/2	27.1 $\pm$ 2.42 2/2
BS buffer	PB6	10	63.7 $\pm$ 1.4 4/4	19.5 $\pm$ 0.759 4/4	107 $\pm$ 62.2 4/4
BS buffer	PB6	20	80 $\pm$ 8.62 5/5	26.6 $\pm$ 2.44 5/5	133 $\pm$ 47 5/5
BS buffer	PB6	50	68.7 $\pm$ 8.04 3/3	38.9 $\pm$ 0.551 3/3	172 $\pm$ 13 3/3
BS riparian	—	0	43.1 $\pm$ 1.23 5/5	22.4 $\pm$ 0.479 5/5	17.3 $\pm$ 11.5 5/5
BS riparian	PB3	5	52.3 $\pm$ 2.11 5/5	26.5 $\pm$ 1.5 5/5	31.2 $\pm$ 13.3 5/5

Soil	Ash	Rate	Ca	Mg	K
BS riparian	PB3	10	51.9 ± 2.67 4/4	31.6 ± 1.13 4/4	44 ± 15.5 4/4
BS riparian	PB3	20	50.6 ± 4.23 5/5	33.2 ± 1.2 5/5	75 ± 24.9 5/5
BS riparian	PB3	50	64.1 ± 3.98 5/5	41.3 ± 0.768 5/5	131 ± 22.5 5/5
BS riparian	PB6	5	46.4 ± 5.56 5/5	22.5 ± 3.1 5/5	15.3 ± 0.804 5/5
BS riparian	PB6	10	51.8 ± 7.02 5/5	27.2 ± 4.16 5/5	55.3 ± 23.6 5/5
BS riparian	PB6	20	53.2 ± 10.9 5/5	31.8 ± 0.292 5/5	76.6 ± 13.1 5/5
BS riparian	PB6	50	57.3 ± 13.9 5/5	35.3 ± 0.426 5/5	145 ± 15.7 5/5
BS upland	—	0	77.1 ± 4.65 3/3	23.7 ± 1.95 3/3	305 ± 139 3/3
BS upland	PB3	5	75.1 ± 2.53 2/2	22.3 ± 1.03 2/2	32.7 ± 0.275 2/2
BS upland	PB3	10	92.3 ± 2.47 3/3	27.5 ± 0.735 3/3	176 ± 133 3/3
BS upland	PB3	20	92.2 ± 6.62 4/4	28.6 ± 2.63 4/4	122 ± 59.4 3/4
BS upland	PB3	50	133 ± 3.6 2/2	43.6 ± 1.79 2/2	268 ± 142 2/2
BS upland	PB6	5	87.8 ± 1.73 4/4	26.8 ± 0.443 4/4	126 ± 86.4 4/4
BS upland	PB6	10	106 ± 3.75 2/2	31.7 ± 1.68 2/2	55.8 ± 3.4 2/2

Soil	Ash	Rate	Ca	Mg	K
BS upland	PB6	50	90.2 ± 0.1 2/2	45.8 ± 0.01 2/2	152 ± 0.1 2/2
DM riparian	—	0	44.8 ± 3.56 9/9	12.1 ± 0.873 9/9	41 ± 26.1 9/9
DM riparian	PB3	5	51.3 ± 10.8 4/4	13.5 ± 2.78 4/4	62.2 ± 53 4/4
DM riparian	PB3	10	51.8 ± 10.1 5/5	13.8 ± 2.78 5/5	55.7 ± 38.7 5/5
DM riparian	PB3	20	64.9 1/1	22 1/1	38.6 1/1
DM riparian	PB3	50	73.2 ± 6.31 4/4	26 ± 3.26 4/4	136 ± 35.5 4/4
DM riparian	PB6	5	67.4 ± 0.956 4/4	17 ± 0.46 4/4	50.1 ± 36.6 4/4
DM riparian	PB6	10	73.3 ± 4.93 4/4	18.2 ± 0.647 4/4	69.8 ± 42.8 4/4
DM riparian	PB6	20	109 ± 13 4/4	21.8 ± 2.67 4/4	85.1 ± 32 4/4
DM riparian	PB6	50	171 ± 5.63 3/3	22 ± 1.4 3/3	148 ± 4.63 3/3
DM upland	—	0	8.57 ± 1.34 5/5	2.92 ± 0.547 5/5	41.7 ± 39.9 5/5
DM upland	PB3	10	30.4 1/1	8.09 1/1	158 1/1
DM upland	PB3	20	35.9 1/1	7.81 1/1	18.3 1/1
DM upland	PB3	50	79.2 ± 6.79 5/5	23.9 ± 0.79 5/5	134 ± 38.3 5/5

Soil	Ash	Rate	Ca	Mg	K
DM upland	PB6	5	28.2 ± 2.48 4/4	7.52 ± 0.671 4/4	29 ± 21.7 4/4
DM upland	PB6	10	73.7 ± 4.82 4/4	15.4 ± 0.768 4/4	57.4 ± 37.4 4/4
DM upland	PB6	20	151 ± 14.9 4/4	24.6 ± 1.71 4/4	83.9 ± 32.9 4/4
DM upland	PB6	50	321 ± 6.97 4/4	34.6 ± 1.16 4/4	174 ± 33.1 4/4

Table 6 continued. Remaining columns for the same samples.

Soil	Ash	Rate	Na	S	Mn	P
BS buffer	—	0	2.92 1/1	1.66 1/1	0.0695 1/1	<MDL 0/1
BS buffer	PB3	5	4.64 ± 0.144 2/2	2.19 ± 0.008 2/2	3.66 ± 2 2/2	0.109 1/2
BS buffer	PB3	10	4.82 ± 0.203 2/2	1.72 ± 0.0515 2/2	0.438 ± 0.425 2/2	<MDL 0/2
BS buffer	PB3	20	7.32 ± 0.198 3/3	1.63 ± 0.0707 3/3	2.89 ± 1.46 3/3	0.11 1/3
BS buffer	PB3	50	2.94 ± 0.0135 2/2	1.74 ± 0.001 2/2	2.67 ± 2.66 2/2	<MDL 0/2
BS buffer	PB6	5	7.67 ± 0.654 2/2	1.77 ± 0.0335 2/2	1.23 ± 0.798 2/2	0.106 1/2
BS buffer	PB6	10	14.3 ± 0.118 4/4	1.99 ± 0.0742 4/4	2.5 ± 1.13 4/4	0.111 ± 0.0067 2/4
BS buffer	PB6	20	28 ± 1.46 5/5	1.96 ± 0.0934 5/5	1.53 ± 0.654 5/5	0.122 ± 0.00575 2/5

Soil	Ash	Rate	Na	S	Mn	P
BS buffer	PB6	50	56 ± 3.42 3/3	12.5 ± 6.54 3/3	0.138 ± 0.00798 3/3	0.133 ± 0.0217 3/3
BS riparian	—	0	4.57 ± 0.211 5/5	1.09 ± 0.0794 5/5	0.488 ± 0.379 5/5	<MDL 0/5
BS riparian	PB3	5	5.48 ± 0.272 5/5	0.859 ± 0.0971 5/5	1.59 ± 0.755 5/5	<MDL 0/5
BS riparian	PB3	10	6.76 ± 0.501 4/4	0.78 ± 0.0361 4/4	0.877 ± 0.468 4/4	<MDL 0/4
BS riparian	PB3	20	8.15 ± 0.129 5/5	0.889 ± 0.0543 5/5	0.632 ± 0.218 5/5	<MDL 0/5
BS riparian	PB3	50	13 ± 0.183 5/5	1.3 ± 0.177 5/5	0.334 ± 0.136 5/5	<MDL 0/5
BS riparian	PB6	5	7.91 ± 0.281 5/5	0.951 ± 0.0627 5/5	1.54 ± 0.726 5/5	0.194 1/5
BS riparian	PB6	10	14.4 ± 0.522 5/5	2.72 ± 1.9 5/5	1.55 ± 0.843 5/5	<MDL 0/5
BS riparian	PB6	20	25.4 ± 1.35 5/5	14.5 ± 13.7 5/5	0.788 ± 0.353 5/5	<MDL 0/5
BS riparian	PB6	50	45.1 ± 0.39 5/5	41.8 ± 7.34 5/5	0.168 ± 0.13 5/5	<MDL 0/5
BS upland	—	0	4.84 ± 0.396 3/3	5.06 ± 0.284 3/3	9.23 ± 1.24 3/3	0.279 ± 0.0708 3/3
BS upland	PB3	5	9.55 ± 0.686 2/2	5.28 ± 0.271 2/2	2.18 ± 2.03 2/2	0.121 ± 0.0068 2/2
BS upland	PB3	10	6.09 ± 0.23 3/3	4.35 ± 0.141 3/3	5.66 ± 1.95 3/3	0.192 ± 0.0147 3/3

Soil	Ash	Rate	Na	S	Mn	P
BS upland	PB3	20	8.37 ± 0.31 4/4	4.93 ± 0.14 4/4	4.31 ± 1.5 4/4	0.197 ± 0.0247 4/4
BS upland	PB3	50	14.2 ± 0.905 2/2	5.55 ± 0.459 2/2	6.43 ± 0.071 2/2	0.269 ± 0.0264 2/2
BS upland	PB6	5	10.8 ± 0.185 4/4	5.12 ± 0.185 4/4	3.33 ± 1.82 4/4	0.227 ± 0.0231 4/4
BS upland	PB6	10	17.3 ± 0.83 2/2	5.19 ± 0.437 2/2	4.11 ± 2.61 2/2	0.214 ± 0.0396 2/2
BS upland	PB6	50	53.8 ± 0.025 2/2	5.25 ± 0.013 2/2	2.3 ± 0.005 2/2	0.28 ± 0.0031 2/2
DM riparian	—	0	3.14 ± 0.104 9/9	1.19 ± 0.0945 9/9	0.577 ± 0.183 9/9	<MDL 0/9
DM riparian	PB3	5	4.7 ± 0.583 4/4	1.75 ± 0.0844 4/4	0.686 ± 0.465 3/4	<MDL 0/4
DM riparian	PB3	10	5.28 ± 0.469 5/5	1.6 ± 0.244 5/5	0.536 1/5	<MDL 0/5
DM riparian	PB3	20	7.1 1/1	0.935 1/1	0.471 1/1	<MDL 0/1
DM riparian	PB3	50	13.5 ± 0.495 4/4	1.44 ± 0.198 4/4	0.132 ± 0.129 4/4	0.15 ± 0.0253 2/4
DM riparian	PB6	5	8.59 ± 0.202 4/4	1.14 ± 0.109 4/4	0.784 ± 0.26 4/4	<MDL 0/4
DM riparian	PB6	10	14.4 ± 0.535 4/4	8.32 ± 5.66 4/4	0.455 ± 0.25 4/4	<MDL 0/4
DM riparian	PB6	20	25.1 ± 0.986 4/4	45.8 ± 14.8 4/4	1.1 ± 0.403 3/4	0.129 1/4
DM riparian	PB6	50	57.7 ± 1.37 3/3	183 ± 18 3/3	1.31 ± 0.0709 3/3	<MDL 0/3

Soil	Ash	Rate	Na	S	Mn	P
DM upland	—	0	3.07 ± 0.253 5/5	1.39 ± 0.119 5/5	0.431 ± 0.18 4/5	<MDL 0/5
DM upland	PB3	10	4.93 1/1	1.21 1/1	1.09 1/1	<MDL 0/1
DM upland	PB3	20	6.66 1/1	3.49 1/1	0.652 1/1	<MDL 0/1
DM upland	PB3	50	14.6 ± 0.254 5/5	4.73 ± 1.4 5/5	1.18 ± 0.438 5/5	0.108 1/5
DM upland	PB6	5	8.15 ± 0.356 4/4	18.4 ± 2.92 4/4	0.198 ± 0.182 3/4	<MDL 0/4
DM upland	PB6	10	16.2 ± 0.503 4/4	63 ± 8.33 4/4	0.691 ± 0.416 4/4	<MDL 0/4
DM upland	PB6	20	29.3 ± 2.71 4/4	122 ± 12.7 4/4	1.94 ± 0.512 4/4	<MDL 0/4
DM upland	PB6	50	65.4 ± 0.751 4/4	315 ± 8.5 4/4	4.06 ± 0.294 4/4	<MDL 0/4

Table 7. Detection frequencies from 156 unambiguous ICP records. Concentrations are in ppm. Detected ranges exclude censored observations. Both ambiguous sample-198 records are excluded.

Analyte	Detected / total	Below MDL (%)	Detected range
P	37/156	76.3	0.102–0.421
Cd	0/156	100.0	Not detected
Pb	0/156	100.0	Not detected
Cu	42/156	73.1	0.0055–0.0254
Ni	35/156	77.6	0.0106–0.0396
Zn	39/156	75.0	0.0116–0.107
Cr	1/156	99.4	0.005–0.005
As	0/156	100.0	Not detected

Cd, Pb and As were below the reporting limit in all 156 included records; Cr was detected once. P, Cu, Ni and Zn were detected in a minority of records (Table 7).

Table 8. Available unambiguous ICP records by soil source, nominal ash group, and rate. Five jars were allocated per listed combination. Zero-dose nominal groups are pooled once for Table 6. An absent assay is not a measured zero. Both records labelled 198 are excluded.

Soil	Nominal group	0	5	10	20	50
BS buffer	PB3	1	2	2	3	2
BS buffer	PB6	0	2	4	5	3
BS riparian	PB3	0	5	4	5	5
BS riparian	PB6	5	5	5	5	5
BS upland	PB3	1	2	3	4	2
BS upland	PB6	2	4	2	0	2
DM riparian	PB3	5	4	5	1	4
DM riparian	PB6	4	4	4	4	3
DM upland	PB3	2	0	1	1	5
DM upland	PB6	3	4	4	4	4

4.0 DISCUSSION

4.1 pH Response to Wood Ash Amendment

Wood ash raised Day 60 pH above the matched untreated value in all five soils, including at 5 t ha⁻¹. Amended values ranged from 6.76 to 8.16, compared with 5.24–6.78 in the untreated controls (Figure 2). This supports the hypothesis that ash would reduce soil acidity. The response was not consistently larger at higher rates: several intermediate and high rates produced similar or lower pH values than the lower rates. The main result is therefore a clear difference between amended and untreated samples, rather than a uniform increase with each additional ash dose.

This response is consistent with the liming action of wood ash. Alkaline compounds in ash neutralize acidity as they react with soil water, while ash also supplies base cations such as Ca, Mg and K (Pitman, 2006; Maresca et al., 2017). The measured enrichment of base cations in the two ashes relative to the soils supports this explanation (Tables 2 and 3a). The amount of each element, however, is only part of the explanation. The mineral forms in which it occurs affect dissolution and the neutralizing capacity of the ash (Maresca et al., 2017). Thorough mixing and the addition of water in this experiment provided close contact between the ash and soil, allowing these reactions to occur throughout the mixture.

The magnitude of the pH response varied among soils and could reflect differences in exchangeable acidity, organic matter and mineral composition. BS upland had an untreated endpoint pH of 5.24 and amended values of 6.76–7.65, whereas DM upland had an untreated endpoint of 5.67 and amended values of 7.08–7.57. Soil organic matter and mineral surfaces can buffer changes in solution pH, so initial acidity alone does not determine the response to a given ash addition (Krzic et al., 2021). The different soil responses are consistent with this explanation, although buffering capacity was not measured directly. These comparisons use the Day 60 controls; the initial-soil measurements in Table 3 describe the starting materials.

The similar endpoint values at several rates suggest that increasing the amount of ash may provide little additional pH change in some soils. Soil buffering and differences in ash dissolution offer possible explanations (Maresca et al., 2017; Krzic et al., 2021), alongside variation among jars and measurements. From a management perspective, the response at 5 t ha⁻¹ makes lower application rates worth testing in the field. It does not establish a suitable operating rate, because the persistence of the pH change and its effects on vegetation and water chemistry also need to be considered.

4.2 Greenhouse Gas Concentrations

4.2.1 Methane (CH₄)

The CH₄ results partly support the expectation of higher concentrations in riparian soils. Both riparian sources had higher means than their paired upland sources at Day 10, but BS upland exceeded BS riparian by Day 34. This change shows that the relationship between landscape position and methane depended on the stage of incubation. It also explains why treating all upland soils as a single low-methane group would overlook an important response in the BS upland composite.

The expectation of higher riparian CH₄ was based on the supply of organic substrates and the low-oxygen conditions commonly found in wet soils. Decomposition supplies the compounds used by methanogens, and differences in substrate availability can help explain variation in methane dynamics among peatland soils (Basiliko et al., 2007). In this experiment, all soils were incubated under the same N₂-flushed conditions. Differences between sources therefore reflect their responses to a shared laboratory environment, including possible differences in organic matter quality and microbial communities. The later increase in the relative importance of BS upland is consistent with a response that developed during incubation, rather than one determined solely by its landscape label.

Ash could affect methane through several processes acting at the same time. Reducing acidity may change microbial activity and the availability of substrates, but methanogen communities differ in their physiology and environmental responses (Bräuer et al., 2020). Changes in the supply of alternative electron acceptors could also influence competition for

hydrogen and acetate. Sela-Adler et al. (2017) showed in estuarine sediments that sulfate reduction and methanogenesis can share these substrates and occur together, even when sulfate reduction is dominant. This provides a possible explanation for why a chemical change need not produce a simple methane response. It is not evidence that sulfate reduction controlled these jars: the ICP measurements represent total S, and neither sulfate reduction nor microbial community composition was measured.

The soil- and date-dependent patterns therefore suggest that ash effects on methane depend on the receiving soil and the progress of anaerobic decomposition. A higher pH alone is not enough to predict the direction of the response. The repeated-measures analysis accounts for the two observations from each jar. Taken together, the results support examining soil properties and incubation time alongside ash type and rate, rather than expecting a single methane response across all five sources.

4.2.2 Carbon Dioxide (CO₂)

CO₂ concentrations increased between Days 10 and 34 in several soil and ash treatments. The clearest example was DM upland, where all eight amended treatment means increased over this period. Its untreated mean also rose, from approximately 5,977 to 18,702 ppm. Thus, the rise in CO₂ was not confined to ash-amended jars. The results are consistent with continued carbon decomposition during incubation, but an increase through time must be considered alongside the untreated response when assessing the contribution of ash.

Comparisons at the same sampling date provide a more direct description of the ash response. At Day 10, all eight amended means exceeded the untreated mean in both upland sources. In DM upland, the amended means ranged from approximately 10,637 to 19,847 ppm,

compared with 5,977 ppm in the control. By Day 34, the control had increased and the separation was less pronounced. These patterns are consistent with an early response to ash in these soils, followed by changes in both amended and untreated jars. They do not establish that every amended treatment differed statistically from its control, because the analysis tested overall effects rather than individual treatment–control comparisons.

Ash-induced changes in pH and nutrient supply suggest possible biological explanations for the higher concentrations in some treatments. Changes in acidity can alter the release and retention of dissolved organic matter, while microbial activity affects both its production and breakdown (Kalbitz et al., 2000). Where ash makes suitable substrates or nutrients more available, decomposition could contribute to higher CO₂ concentrations. Differences in the amount and quality of organic matter could also help explain why the response varied among soils and over time. Dissolved organic carbon and microbial activity were not measured directly, so their contributions to the CO₂ response could not be separated.

The repeated-measures model found an overall difference between sampling dates for CO₂ ($p = 0.027$). The exploratory rate test gave $p = 0.0015$ (Table 4). The most supported interpretation is that CO₂ changed during incubation and that some amended groups had higher concentrations than their controls, particularly at Day 10 in the upland soils. There was no consistent increase with ash rate across the experiment. The concentration patterns support examining possible changes in decomposition, subject to the measurement limitations in Section 4.5.

4.3 Total Mercury and Porewater Chemistry

4.3.1 Total Dissolved Mercury (THg)

Filtered-water THg was often lower in amended samples than in the untreated records, although the response varied among soils and ash rates. BS upland contained the highest observed THg value, and the relative difference between PB3 and PB6 changed among treatments. The results therefore suggest that ash addition can be associated with changes in the amount of mercury present in filtered water, but they do not show a uniform response to either ash or a consistently stronger effect at higher rates.

Changes in mercury binding and solubility offer possible explanations for these observations. Mercury binds strongly to sulfur-containing groups in organic matter, while mineral surfaces and inorganic sulfur compounds also affect its distribution between water and solids (Ullrich et al., 2001; Skyllberg, 2008). Ash changes the chemical environment in which these reactions occur. Greater retention by soil or ash surfaces could therefore contribute to lower filtered-water THg in some treatments. However, dissolved organic complexes can also retain mercury in the water phase, so the direction of the response cannot be predicted from pH alone. The present records cannot establish a paired jar-level relationship between pH and THg.

These observations are relevant to ash use near wetlands and streams because changes in mercury retention could affect transport from soils. Lower filtered-water THg is not sufficient evidence of reduced methylmercury production or downstream risk. Methylation depends on microbial activity and mercury availability as well as the surrounding sulfur and organic matter chemistry (Ullrich et al., 2001; Skyllberg, 2008). The next step is to test whether the observed THg differences are repeatable and whether they coincide with changes in MeHg under field conditions.

4.3.2 Porewater Base Cation and Elemental Chemistry

The porewater results show that ash addition changed the supply of dissolved elements, with Na increasing in many amended treatments and Ca and Mg responses varying more among soils. Dissolution of ash is a direct potential source of these elements. The amount released depends on both the quantity added and the mineral forms present, while soil reactions can retain part of that input (Pitman, 2006; Maresca et al., 2017). This provides a basis for expecting different responses among elements even when they are supplied in the same ash treatment.

The measured ash chemistry helps explain differences between PB3 and PB6. PB6 contained more total Na and S than PB3 (Table 2), consistent with the large dissolved concentrations in several PB6 treatments. DM upland provides a clear example: at 50 t ha⁻¹ PB6, mean Ca, Na and S concentrations were approximately 321, 65.4 and 315 ppm, compared with untreated means of 8.57, 3.07 and 1.39 ppm, respectively (Table 6). In this soil, the PB6 means for all three elements increased across the four amended rates. This is consistent with a larger ash input supplying more soluble material, although the dissolved concentrations also reflect retention and other reactions within the soil mixture.

Soil properties may help explain why the same ash produced different porewater responses among sources. DM upland had the lowest LOI (6.6%), whereas DM riparian had the highest (71.1%). Organic matter provides sites that can retain cations, and differences in these sites may contribute to the contrast in dissolved concentrations. Mineral composition and pH also affect retention, so LOI alone cannot establish which soil has the greater exchange capacity. The strong DM upland response is a reason to examine these properties together in future trials. The total S measurements also require distinction from sulfate: sulfur speciation would be needed to connect these results to sulfate reduction or mercury methylation.

The high concentrations at some rates have practical implications for ash selection and water-quality monitoring. Returning Ca and other nutrients to depleted forest soils is one purpose of ash recycling, but a high concentration in filtered water does not directly measure plant uptake. It can also identify an element that could be transported if water moves through the treated soil. Field hydrology, retention along flow paths and vegetation uptake will determine how much reaches groundwater or streams. The PB6 results make Na and S particularly useful monitoring targets, alongside the base cations, when testing ash under field conditions.

4.4 Soil Properties and Site Context

The five soil sources provided a gradient in initial pH, moisture and organic matter for examining differences in ash response. LOI ranged from 6.6% in DM upland to 71.1% in DM riparian, with further differences in TOC and N documented in Table 3a. These properties are relevant because organic matter supplies substrates for microbial activity, contributes to nutrient retention and binds mercury. They provide more useful ecological context than the administrative boundaries in which the sites were located.

The results did not follow one consistent riparian-upland pattern. The higher CH₄ concentrations in both riparian sources at Day 10 supported the initial expectation, but the BS upland response at Day 34 showed that this relationship could change during incubation. Similarly, the strong dissolved-element response in DM upland indicates that mineral soils can show substantial chemical changes after ash addition. Soil moisture, organic matter quality, acidity and mineral composition should therefore be considered together when selecting sites or predicting their response.

The relatively similar bulk C:N ratios do not rule out differences in nitrogen availability, because these ratios do not measure the forms of carbon and nitrogen available to microorganisms. Nor do the five composites separate the individual effects of soil properties. The value of this comparison is that it identifies shared responses, such as the rise in pH, as well as differences that need testing across more independent sites. This approach can contribute to broader guidance based on measured soil conditions without assuming that every soil requires an entirely separate recommendation.

4.5 Limitations and Considerations

Only one composite was collected from each soil source. Laboratory jar replication therefore estimates treatment variation within these composites, while broader riparian-upland comparisons require more independent sites. Gas-model residuals departed from normality in all four analyses, and residual variation differed among treatments in both CH₄ analyses. These departures limit the reliability of the conventional ANOVA p-values. Missing measurements and incomplete records of treatment allocation and jar placement also limit the statistical conclusions. Available readings were retained in descriptive figures, whereas the repeated-measures models used complete pairs; the two summaries can therefore differ.

Homogenization, ash mixing and high water content simplify field exposure. Each jar received 60 mL water per 30 g field-moist soil. This provided more direct contact between ash and soil than surface spreading, but did not reproduce rainfall-driven dissolution or transport through intact profiles. Equal field-moist soil masses also contained different amounts of dry soil and organic matter. Differences among soil sources may partly reflect these unequal amounts.

Initial-soil pH and Day 60 filtrate pH differ in preparation, so the treatment comparisons use matched-time untreated controls. Replicate-level endpoint pH measurements were unavailable.

The N₂-flushed jars represent imposed anaerobic conditions rather than field emissions from upland or riparian soils. Gas concentrations were calculated using the recorded single-point calibration factors. Run-specific quality-control records were unavailable. Headspace volumes were not recorded during the experiment. Headspace volume is not required to express the calibrated readings in ppm, but gas amounts and production rates would require that volume and an account of gas withdrawal or replacement. The reported temporal changes are therefore differences in sampled concentration, not rates of carbon loss per unit soil mass. No recorded headspace volumes are therefore available for retrospective conversion of the concentration measurements to gas amounts. The nominal 250 mL jar capacity and 60 mL water addition alone are insufficient to recover it: the volume occupied by field-moist soil, native soil water and the actual sealed jar capacity also matter. A defensible reconstruction would require checking those quantities and accounting for gas withdrawal and N₂ replacement. Such a reconstruction could support estimates of headspace gas amounts and net accumulation, but would still not measure gross microbial production or field flux.

The available THg dataset contains one observation per amended treatment, and mercury records could not be reliably paired with pH records. ICP results had incomplete treatment coverage and included values below reporting limits; two records with conflicting sample labels were excluded. These data support descriptive chemical comparisons, but do not determine mercury methylation or downstream effects. The Day 60 ICP measurements also provide only one point in time: earlier release, retention and changes in dissolved concentrations were not measured.

Finally, ash properties vary among sources, combustion facilities and feedstock materials (Pitman, 2006; Maresca et al., 2017). The results apply to the two ash materials tested and should not be transferred directly to other batches with different chemistry, particle size or carbon content. Characterization of both the ash and receiving soils, followed by field measurements, is needed before operational application.

4.6 Conclusions and Management Implications

Wood ash increased Day 60 pH in all five soils, including at the lowest amended rate, supporting the hypothesis that it would reduce acidity. The response was not uniformly larger at higher rates. Porewater chemistry also changed, with large Ca, Na and total S concentrations in several PB6 treatments. These results show why both ash composition and receiving-soil properties matter when evaluating ash as a soil amendment.

Gas responses varied with soil source and sampling date. Higher CH₄ concentrations in riparian soils at Day 10 partly supported the landscape-based hypothesis, but the pattern was not maintained at Day 34. CO₂ increased in several treatments, including all amended DM upland groups, while increases in untreated jars showed that incubation time also mattered. Higher amended CO₂ means in the upland soils at Day 10 are consistent with an early response to ash, although non-normal residuals limit interpretation of the general rate effect. These findings point to changes in carbon processing that depend on the soil and stage of incubation.

THg was often lower in amended samples, making mercury retention a useful focus for further work. Direct MeHg measurements are needed to determine whether this pattern has implications for methylation and downstream exposure. Together with the high dissolved-

element concentrations in some treatments, this supports including water chemistry in field assessments of ash use, particularly where treated areas connect to wetlands or streams.

The findings suggest four priorities for further work:

1. Test lower and higher application rates under field conditions before recommending an operating range.
2. Measure the chemistry, neutralizing capacity, particle size and leaching behaviour of each ash batch.
3. Measure transport from treated soils and its effects on water chemistry, MeHg and gas exchange.
4. Sample multiple independent riparian and upland sites, with separate estimates of field and laboratory variation.

Recycling wood ash can return nutrients removed during harvesting and reduce disposal in landfills. This study provides laboratory evidence of chemical responses relevant to that use, while identifying soil and ash combinations that merit closer examination. Longer-term field measurements across sites and seasons would help determine where these benefits can be achieved while protecting soil and water quality.

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