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Co-application of Papermill Sludge and Wood Ash Improves Forage Crop Yield and Soil Health in a Greenhouse Trial

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Abstract: Application rates of papermill residual (PMR) in Ontario, Canada, are regulated to a wet weight maximum of 20 Mg ha^{-1} yr^{-1} with the actual rate determined by the concentrations of plant available nitrogen, phosphorous, potassium and 10 regulated metals in the materials and receiving soils. Previous studies report improvements in crop yield and soil properties following PMR application but generally at rates far exceeding 20 Mg ha^{-1} yr^{-1} and after years of repeated application. We investigated whether application of PMR at operational rates could enhance the forage crop yield and improve soil health in three locally sourced agricultural soils in a greenhouse experiment. When papermill sludge (PPMS) was incorporated into the soils, forage yields were comparable to, or greater than, those achieved with mineral fertilizer in two of the three soils. The largest yield increases occurred when PPMS was applied in combination with wood ash. Application of PPMS alone did not produce statistically significant changes in soil health scores, but scores improved by up to ten points when PPMS and wood ash were applied together. When PMR is available locally, land application, particularly when PPMS is applied with wood ash, can reduce dependence on conventional amendments to improve agricultural outcomes.

Key words: Pulp and papermill residuals (PMR), circular economy, soil amendment, soil health.

1. Introduction

Pulp and papermill residuals (PMR) generated during pulp and papermill production have historically been landfilled, but these materials are increasingly being diverted for land application to improve site productivity [1-6]. Two common PMR, pulp and paper mill sludge (PPMS) produced from wastewater treatment, and wood ash (WA), generated from combustion of mill wastes, have well-documented benefits for soil properties and plant productivity [2, 6]. Concerns remain, however, regarding contaminants and heavy metal concentrations in these materials and their persistence in the environment [3, 6]. In their recent review of land application of PPMS, [6] showed clear benefits to crop productivity with no negative ecological impacts under typical field application rates ranging from 10 Mg ha^{-1} up to 200 Mg ha^{-1} (dry weight). In regions with established or emerging agricultural sectors, particularly in northern areas with nearby pulp and papermills, land application of PPMS and WA represents an opportunity to increase the profitability for both the mills and local producers [7]. When properly regulated (e.g., [3, 6, 7]), diverting these materials to agricultural land can reduce disposal costs for generators and decrease reliance on conventional liming materials and mineral fertilizers by agricultural producers, with few environmental risks.

Unlike conventional liming materials and mineral fertilizers, the physical and chemical properties of PPMS and WA vary widely across papermills due to

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differences in production practices and feedstocks [8]. As a result, comparisons across studies require consideration of the properties of these materials. PPMS, which is typically rich in organic matter, can enhance soil aggregation, water holding capacity, infiltration, pH, and nutrient availability in the soil, often resulting in increased plant growth and yield [6]. WA is commonly applied as a liming agent to raise soil pH and supply nutrients such as P and K [9]. The increase in soil pH improves nutrient availability generally reduces the solubility of heavy metals, as many metals are less soluble within the pH range optimal for plant growth [10].

To protect environmental and human health, applications rates for PPMS and WA are often regulated by a governing body [2]. For example, land spreading of PPMS in the European Union is regulated by Directives 86/278/EEC and 91/692/EEC but these directives can be regulated differently by Member States [6]. Spreading in the European Union is covered by the European Waste Catalogue Codes 03 03 05, 03 03 10 & 03 03 11 [6]. In the United States of America and Canada, regulations differ from state to state and province to province [2]. In all jurisdictions, allowable rates consider the properties of the materials, soil characteristics of the receiving environment, and crop requirements with the goal of protecting environmental and human health [11, 12].

In Ontario, Canada, PPMS and WA are classified as non-agricultural source materials (NASM) [11] under the Nutrient Management Act [13] and regulated through Ontario Regulation 267/03 [12, p. 26]. PPMS and WA are classified as Category 3 NASM and require a NASM plan for land application, which is a legal document that demonstrates that the application of NASM mitigates any negative risks to environmental and human health [11]. The maximum permissible annual application rate of any category of NASM is 20 Mg ha^{-1} (wet weight) but the actual rate of application of Category 3 NASM is determined by the quantities of plant available N, P and K, as well as the ten regulated metals. This means that operational rates of application may be lower than the regulatory maximum of 20 Mg ha^{-1} [12]. This maximum rate is at the lower end of the application rates that have been tested in laboratory and field trials, which typically exceed 20 Mg ha^{-1} and can reach up to 450 Mg ha^{-1} [2, 6, 14].

Realized agronomic benefits of PPMS and WA application depend in part on weather during the growing season. As with conventional agricultural amendments, moisture is required to dissolve compounds, facilitate ion transport, and support chemical reactions, such as acid neutralization. Some of the benefits of land application of papermill residuals, like nutrient availability, are dependent upon the decomposition and mineralization of organic compounds in the soil that may not be realized in any given year because of weather conditions. Soil properties also mediate treatment effects. For example, coarse textured soils with low cation exchange capacity (CEC) and low water-holding capacity may produce lower yields than soils with more clay [15, 16]. Coarse textured soils may benefit more from organic amendments that increase their organic matter contents, which can improve aggregation, water holding capacity, infiltration, etc. [4].

The objective of this study was to determine whether operational application rates of PMR, as defined under Ontario's regulations, can improve soil properties and forage crop yield. We evaluated the effects of PPMS and WA applied at regulated rates on forage crop yield and physical, chemical and biological properties of three soils in two greenhouse experiments. We hypothesized that PPMS and WA would increase forage crop yield and improve soil properties and that these effects would differ between the soils included in this study, with the greatest benefits realized in the coarser textured soil. This is the only study that we are aware of that explicitly tests the effectiveness of PMR to increase crop yield and improve soil health at operational rates of application within a regulatory framework.

Table 1 Properties of the soils collected from farms in Slate River Valley, Murillo and South Gillies and used in the greenhouse experiments.

Parameter	Units	Slate River Valley	Murillo	South Gillies
Bulk Density	g cm^{-3}	1.15	1.03	1.02
Sand	%	32	46	22
Silt	%	46	36	27
Clay	%	26	18	41
Texture		loam	loam	clay loam
pH		6.9	6.7	5.7
CEC	Meq 100 g^{-1}	19.2	14.5	22.0
SOM	g kg^{-1}	64	52	82
N	g kg^{-1}	2.7	1.3	2.8
C:N		11.4	13.9	12.3
Extractable P	mg kg^{-1}	46	5	8
Extractable K	mg kg^{-1}	61	117	133
Extractable Mg	mg kg^{-1}	458	411	406
Extractable Ca	mg kg^{-1}	2800	1880	1480
Extractable Na	mg kg^{-1}	24	51	19
Extractable S	mg kg^{-1}	11	7	8
Extractable Zn	mg kg^{-1}	3.3	5.3	4
Extractable Mn	mg kg^{-1}	19	9	17
Extractable Fe	mg kg^{-1}	90	8	116
Extractable Cu	mg kg^{-1}	1.9	2.4	1.5
Extractable B	mg kg^{-1}	0.4	0.5	0.1
Extractable Al	mg kg^{-1}	551	521	1041
Total As	mg kg^{-1}	2	10	2
Total Cd	mg kg^{-1}	0.66	1.09	0.71
Total Cr	mg kg^{-1}	47.03	37.28	45.79
Total Co	mg kg^{-1}	18.05	14.01	21.05
Total Cu	mg kg^{-1}	30.99	31.41	28.67
Total Pb	mg kg^{-1}	8	10	14
Total Hg	mg kg^{-1}	bdl*	0.1	bdl*
Total Mo	mg kg^{-1}	1.4	4.6	1.2
Total Ni	mg kg^{-1}	32.90	27.80	34.53
Total Se	mg kg^{-1}	bdl**	bdl**	bdl**
Total Zn	mg kg^{-1}	87.70	83.05	119.75

*bdl = below detection limit = 0.1 ug g^{-1}, ** bdl = below detection limit = 1.00 ug g^{-1}.

2. Materials and Methods

Two greenhouse experiments were conducted with soils collected from the top 0–15 cm (6 inches) layer from three cultivated fields near Thunder Bay, Ontario, Canada: Slate River Valley (SR) (N 48° 18’ 21.606” W 89° 27’ 32.51”), Murillo (M) (N 48° 27’ 0.24” W 89° 25’ 47.564”), and South Gilles (SG) (N 48° 14’ 18.8880” W 89° 43’ 50.4372”) [17]. Sites were selected to represent local farming systems and to capture variation in soil texture based on historical regional surveys and historical mapping [18, 19]. Immediately after sampling, soils were sieved to 6 mm sieve and stored field-moist at 4oC until the experiment began. Physical and chemical properties of the soils were determined on subsamples of the soils after collection and prior to beginning the experiment (Table 1) to inform fertilizer and PMR application rates following Ontario regulations for these materials [12, 13].

All analyses, except for total N concentrations and the C:N ratio, were performed at A&L Canada Laboratories Inc, London, ON, accredited under the OMAFA Agronomic Test Accreditation Program for available nutrients and for metals to ISO/IEC 17025 standards by Canadian Association for Laboratory Accreditation Inc. (CALA), a requirement under the Nutrient Management Act. Soil pH was determined using the saturated paste method [20], plant available P was measured using a Bray 1 extraction method [21]. Plant available K, Mg, and Ca were determined using a 1 M ammonium acetate extraction method [22], available Zn, Cu, Mn, and Fe using DTPA [23]. Concentrations of ions in all extracts were determined using an ICP-AES. Concentrations of As, Cd, Cr, Co, Cu, Pb, Mo, Ni, Se and Zn were quantified following EPA 3050B/6010 [24, 25] and Hg was determined following EPA 7471B [26]. Soil texture was determined using the hydrometer method [27] and organic matter was determined by loss on ignition [28]. CEC was calculated following [4]. Total N concentrations were determined by flash combustion after grinding to pass through a 53 mm sieve using a Spex ball mill [29].

2.1 Pulp and Papermill Sludge and Wood Ash

Pulp and papermill sludge (PPMS) and wood ash (WA) were sourced locally from Thunder Bay Pulp & Paper (formerly Resolute Forest Products), Thunder Bay, Ontario, Canada. The PPMS is a mixed of primary and secondary pulp and papermill sludge and the wood ash is from the operation's biomass boiler. Composite samples were analyzed at the University of Guelph, which is accredited under the OMAFA Agronomic Test Accreditation Program for nutrients and for metals to ISO/IEC 17025 standards by CALA, a requirement under the Nutrient Management Act. Analyses for dioxins and furans, regulated in some jurisdictions (e.g., [30]) but not in Ontario, were analyzed by ALS Environmental (Burlington, ON) following USEPA 1613B [31]. Chemical properties are presented in Table 2; dioxin and furan concentrations are provided as supplemental material (S1).

2.2 Experimental Design

Application of non-agricultural source materials (NASM) to agricultural soils in Ontario requires a demonstrated beneficial use for land application of these materials (e.g., increases pH, adds organic matter) and that there is an agronomic deficiency in the soil that could be improved with application of the NASM (e.g., sub-optimal pH for maximum crop yield, low N or P). The beneficial use of WA is as a liming agent to increase the pH of the soil, while for the PPMS, its high organic matter content is used to increase the organic matter content of the soil and provide nutrients. Concentrations of regulated metals in the receiving soil cannot exceed regulatory limits. None of the receiving soils exceeded regulatory background concentrations of heavy metals.

Fertilizer recommendations for the forage crop grown in the experiments were derived from provincial (OMAFA) recommendations based the soil test results. Recommendations for NPK were: SR, 140-20-20 kg ha^{-1}; M, 140-110-40 kg ha^{-1}; SG, 140-90-20 kg ha^{-1}. Application rates of PPMS and WA were calculated using OMAFA's Agrisuite nutrient calculator (https://agrisuite.omafra.gov.on.ca/), which calculates the plant available NPK in the materials. Application rates were limited by plant available N and P rather than metal concentrations.

Soil testing indicated that SR and M soils required only PPMS, whereas SG required both liming (WA) and organic matter additions (PPMS). To reflect real-world nutrient management decisions, treatments included no fertilizer, mineral fertilizers alone, PMR alone, and PMR supplemented with mineral fertilizer when needed.

2.2.1 Experiment One: The Effects of Papermill Biosolids Application to Soil on Forage Crop Yield, Soil Properties and Soil Health

Experiment one evaluated the response of SR, M, and SG soils to four treatments: 1. no mineral fertilizer

Table 2 Properties of the wood ash and papermill biosolids from Thunder Bay Pulp and Paper that were used in this study.

Parameter	Unit	Wood Ash	Papermill Biosolids
Moisture	%	29.4	69.5
Dry Matter	%	70.6	30.5
pH		12.2	7.0
Electrical Conductivity	mS cm^{-1}	10.9	0.6
Organic Matter	g kg dry^{-1}	251	938
Total Carbon	g kg dry^{-1}	270	466
Inorganic Carbon	g kg dry^{-1}	10.55	0.88
Organic Carbon	g kg dry^{-1}	260	464
Total Nitrogen	g kg dry^{-1}	1	16
C:N		260	29
Total Kjeldahl Nitrogen	g kg dry^{-1}	0.75	15.62
NH_4-N	g kg dry^{-1}	0.00	1.05
NO_3 and NO_2	g kg dry^{-1}	0.00	0.01
P	g kg dry^{-1}	5.14	3.39
K	g kg dry^{-1}	20.05	1.40
Plant Available Nitrogen	g kg dry^{-1}	0.23	5.43
Plant Available Phosphorous	g kg dry^{-1}	4.71	3.11
Plant Available Potassium	g kg dry^{-1}	21.65	1.51
Neutralizing Value	%$CaCO_3$ equivalents	61.1	Not applicable
Na	g kg dry^{-1}	5.17	3.74
B	mg kg dry^{-1}	89.4	42.2
As	mg kg dry^{-1}	3.0	0.4
Cd	mg kg dry^{-1}	6.4	3.4
Cr	mg kg dry^{-1}	63	16
Co	mg kg dry^{-1}	7.1	0.7
Cu	mg kg dry^{-1}	52	15
Pb	mg kg dry^{-1}	8.7	1.3
Hg	mg kg dry^{-1}	0.148	0.041
Mo	mg kg dry^{-1}	2.3	0.9
Ni	mg kg dry^{-1}	34	7
Se	mg kg dry^{-1}	0.45	0.09
Zn	mg kg dry^{-1}	802	206

and no NASM (control, C), 2. mineral fertilizers (F), 3. PPMS, and 4. PPMS with mineral fertilizers (PPMS+) (Table 3). Maximum PPMS application was limited by N (M, SG) or P (SR). When PPMS did not supply sufficient NPK, supplemental nutrients were added using a combination of ammonium sulphate (20-0-0-24S), urea (46-0-0), environmentally smart N (ESN, 44-0-0), triple superphosphate (TSP, 0-45-0), muriate of potash (MOP, 0-0-60).

Field moist soils were packed into 1L pots (target volume of 750 cm^3), with weights determined using the soil moisture content and soil bulk density at collection. PPMS was incorporated into soil prior to potting. Pots were seeded at 20 kg ha^{-1} with a regionally relevant forage mixture [50% timothy (*Phleum pratense* L.), 40% coated alfalfa (*Medicago sativa* L.) and 10% coated double cut red clover (*Trifolium pratense* L.)]. Mineral fertilizer was applied to the surface after seeding. Pots were arranged in a random block design in the greenhouse at Lakehead University, Thunder Bay. Each treatment was replicated four times. Soil moisture was maintained at 60% water holding capacity by weight through hand watering every three days. The experiment ran from May 18 to September 10, consistent with the regional forage growing season. Temperatures and light conditions inside the greenhouse were consistent with local growing conditions.

2.2.2 Experiment Two: The Effects of Papermill Biosolids and Wood Ash Application to Soil on Forage Crop Yield, Soil Properties and Soil Health

Experiment Two evaluated SG soil responses to wood ash (WA) alone and WA:PPMS mixtures (1:1, 1:3, and 1:4 by weight), in addition to those included in experiment one, applied with and without mineral fertilizers (Table 4). Rational for ratios included ease of logistical transport given the low density of WA. Soil preparation, potting, seeding, fertilization, watering, and greenhouse management followed the procedures described for Experiment One.

Aboveground biomass was harvested 105 days after emergence by clipping at 5 cm above the soil surface. Samples were weighed fresh, dried at 65°C to constant mass in a drying oven, and converted to dry matter yield (Mg ha^{-1}) based on pot surface area. A relative N use coefficient was calculated by as average dry matter yield divided by total applied N [1]. We acknowledge that this is a simplistic calculation that does not consider the input of N from the mineralization of N from soil organic matter but it is a useful metric when considering an amendment's return on investment.

2.3 Soil Analyses

After harvest, pots were weighed and soil height measured to calculate bulk density. Soil was homogenized, subsampled for gravimetric moisture, air-dried, and sieved to 2 mm. Fertility analyses (pH, organic matter by loss on ignition, CEC, and concentrations of P, K, Ca, and Mg) were conducted at A&L Canada Laboratories Inc. (London, ON). Total N was analyzed at Lakehead University. Methods were the same as those used for initial characterization.

Table 3 Application rates of mineral fertilizer and papermill biosolids (PPMS) applied to soils collected from farms in the Slate River Valley (SR), Murillo (M) and South Gillies (GS) in greenhouse experiment one. Rates were informed by soil testing and crop requirements.

Treatment	Application rate (kg ha^{-1})	SR	M	SG
Mineral Fertilizers	ammonium sulphate	100	100	100
	urea	174	174	174
	environmentally smart nitrogen	91	91	91
	triple super phosphate	44	244	244
	muriate of potash	33	67	50
PPMS	papermill biosolids	4762	9091	9091
PPMS + Mineral Fertilizers	papermill biosolids	4762	9091	9091
	ammonium sulphate	47	0	0
	urea	83	0	0
	environmentally smart nitrogen	44	91	0
	triple super phosphate	0	160	160
	muriate of potash	26	53	36

Table 4 Application rates of mineral fertilizers (F), papermill biosolids (PPMS), and wood ash (WA) applied to the South Gillies (SG) soil in greenhouse experiment two. The application rates of the papermill residuals are ratios are mixtures of WA to PPMS (WA:PPMS). Rates were informed by soil testing and crop requirements.

Application rate (kg ha^{-1})	F	PPMS	WA	1:1	1/3: 2/3	1/4: 3/4
wood ash (WA)	0	0	1734	1648	1570	1500
papermill biosolids (PPMS)	0	9091	0.0	1648	4716	6000
papermill residuals (total)	0	9091	1734	3296	6287	7500
ammonium sulphate	100.0	0	100	86	75	66
urea	174	0	174	152	134	116
environmentally smart nitrogen	2	0	91	81	71	62
triple super phosphate	244	160	226	211	198	186
muriate of potash	50	36	0	0	0	0

Wet aggregate stability (WAS), a measurement of the resistance of the soil structure to external forces (mechanical and physio-chemical), was determined using an Eijkelkamp wet sieving apparatus (Eijkelkamp Agrisearch Equipment 08.12. Giesbeek, Netherlands) following [32]. Soil biological activity was assessed using the Solvita burst CO_2-C (BURST) and Solvita labile amino N (SLAN) tests [33, 34]. For BURST, a 30-cc scoop of soil subsample was transferred to a 50 mL plastic beaker. Nine mL of distilled water was added to the beaker, which was placed inside a 475 mL jar. A Solvita BURST paddle was inserted into each beaker and the jars were immediately sealed and transferred to an incubator where they were left undisturbed for 24 h at 22ºC. A Solvita digital colorimeter reader was used to record the CO_2 evolved and expressed as mg CO_2-C kg^{-1} soil. For SLAN, a 4 g subsample was weighed into a 50 mL beaker. Ten mL of 2 N NaOH was added to the beaker, which was placed in a 250 mL jar with a SLAN paddle. The jar was sealed immediately and transferred to an incubator where they were left undisturbed for 24 h at 22ºC. A Solvita digital colorimeter reader was used to record the NH_3-N evolved and expressed as mg NH_3-N kg^{-1} soil. Permanganate oxidizable carbon (POXC), a labile fraction of C that is readily available for microbial decomposition, was measured following [35] and quantified spectrophotometrically at 550 nm using a Biotek 800 TS plate reader.

Soil health scores were calculated following [36] using Ontario-scoring functions for BURST, SLAN and SOM [36]. Q-Q plots were used to check for outliers and there were none. Observations were binned by textural groups following the Comprehensive Assessment of Soil Health [36, 37]. We used cumulative normal distributions as the scoring functions for BURST, SLAN, and SOM, with the means and standard deviations reported by [36] for our textural classes. This approach assigns a score ranging from 0–100. To calculate a SH score out of 100, we assigned an equal weighting of the scores for BURST, SLAN and SOM (i.e., unweighted average). Soil health scores were interpreted as very low (< 40), low (40–55), medium (55–70), high (70–85), and very high (> 85) following the approach of [37].

2.4 Statistical Analyses

All statistical analyses were in SPSS (version 29.0.2.0, IBM). Data met ANOVA assumptions of normality and homoscedasticity. In Experiment One, a two-way ANOVA assessed the effects of site and treatment on yield and soil parameters. When site by

Table 5 Linear contrasts assessing the effects of nutrient amendments on response variables [17]. Nutrient amendments included no amendments (control = C), mineral fertilizer (F), wood ash (WA), papermill biosolids (PPMS), and ratios of WA:PPMS. Contrasts were invoked only when a significant treatment effect was detected in Experiment Two.

Question	Contrast
L1: Does the application of soil amendments affect the response variable?	[C]-1/11*[all amendments]
L2: Does the response to mineral fertilizers differ from all other amendments or combinations?	[F]-1/10*[treatments containing WA and/or PPMS]
L3: Does the response differ when WA treatments are applied with mineral fertilizers versus without?	[WA treatments without F]-[WA treatments with F]
L4: Does the response differ when PPMS treatments are applied with mineral fertilizers versus without?	[PPMS treatments without F]-[PPMS treatments with F]
L5: Does the response differ between WA only and PPMS only amendments?	[WA + WA&F]-[PPMS + PPMS&F]
L6: Does the response differ between WA only and WA applied in combination with PPMS?	[WA + WA&F]-1/3*[WA:PPMS treatments]
L7: Does the response differ between PPMS only and PPMS applied in combination with wood ash?	[PPMS + PPMS&F]-1/3*[WA:PPMS treatments]
L8: Does the response differ between the 1:1 (WA:PPMS) and 1:3 (WA:PPMS) applications?	[1:1 + 1:1&F]-[1:3 + 1:3&F]
L9: Does the response differ between the 1:1 (WA:PPMS) and 1:4 (WA:PPMS) applications?	[1:1 + 1:1&F]-[1:4 + 1:4&F]
L10: Does the response differ between the 1:3 (WA:PPMS) and 1:4 (WA:PPMS) applications?	[1:3 + 1:3&F]- [1:4 + 1:4&F]

treatment interactions occurred, one-way ANOVA was performed to assess the effect of treatment within each site. Tukey-Kramer least significant difference tests were used for post-hoc comparisons.

In Experiment Two, treatment effects were evaluated using one-way ANOVA. Significant results were further examined using linear contrasts [38] to test hypotheses regarding amendment type and combinations effects (Table 5).

3. Results

3.1 Forage Yield and Nitrogen Use Efficiency

3.1.1 Experiment 1: Site-Specific Responses to Papermill Biosolids and Fertilizers

There was significant interaction between site and treatment for dry matter yield ($F = 2.53$, $p = 0.03$) and for the relative N efficiency coefficient ($F = 24.330$, $p < 0.001$). Treatment effects on yield were not significant in the M soil ($F = 2.776$, $p = 0.087$), where the mean yield was 2.0 Mg ha^{-1}. In contrast, treatment significantly affected yield in both SR ($F = 2.242$, $p = 0.003$) and SG ($F = 7.556$, $p = 0.004$). In the SR soil, the highest yields occurred when PPMS was applied with mineral fertilizers, while in SG all amendments increased yield relative to the unamended control (Fig. 1).

Across sites, there were no differences in yield when no amendments were applied ($F = 3.673$, $p = 0.068$) or when only mineral fertilizers were applied ($F = 2.165$, $p = 0.171$). However, yields differed significantly among sites when PPMS was applied alone ($F = 31.153$, $p < 0.001$) or in combination mineral fertilizers ($F = 21.629$, $p < 0.001$), with the M soil producing markedly lower yield than SG and SR.

Relative N efficiency coefficient also varied by site and treatment. In SR, PPMS alone had the highest relative N efficiency coefficient (50.3 kg DM kg N^{-1}) exceeding the fertilizers alone (22.7 kg DM kg N^{-1}) and the PPMS+fertilizer treatments (28.3 kg DM kg N^{-1}) ($F = 53.887$, $p < 0.001$). In the M soil, efficiency was higher but not significant with mineral fertilizers (21.7 kg DM kg N^{-1}) than PPMS alone (9.9 kg DM kg N^{-1}) ($F = 4.235$, $p = 0.051$). There was no effect of treatment on the relative N efficiency coefficient in SG (26.7 kg DM kg N^{-1}) ($F = 2.256$, $p = 0.161$). Overall, relative N efficiency coefficients were similar be-tween SG (26.7 kg DM kg N^{-1}) and SR (25.8 kg DM kg N^{-1}) but were substantially lower in M (15.7 kg DM kg N^{-1}).

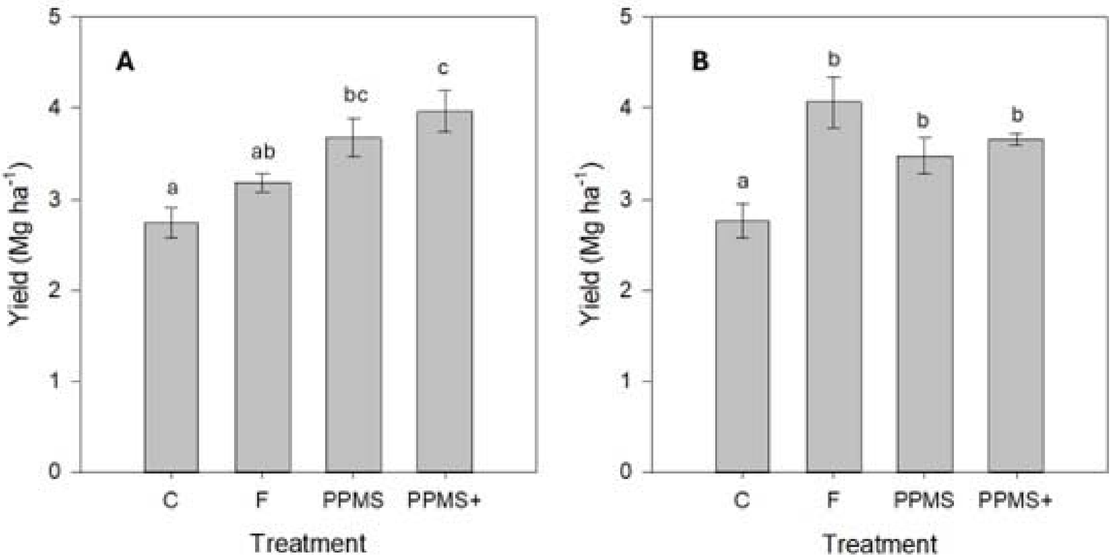

Fig. 1 Dry matter yield for treatments applied to soils from the A) Slate River Valley and B) South Gillies. Bars represent means (n = 4) ± standard error. Treatments include the control (C), mineral fertilizer (F), papermill biosolids (PPMS) and papermill biosolids with mineral fertilizer (PPMS+) in experiment one. Different lower-case letters indicate significant differences among the treatments.

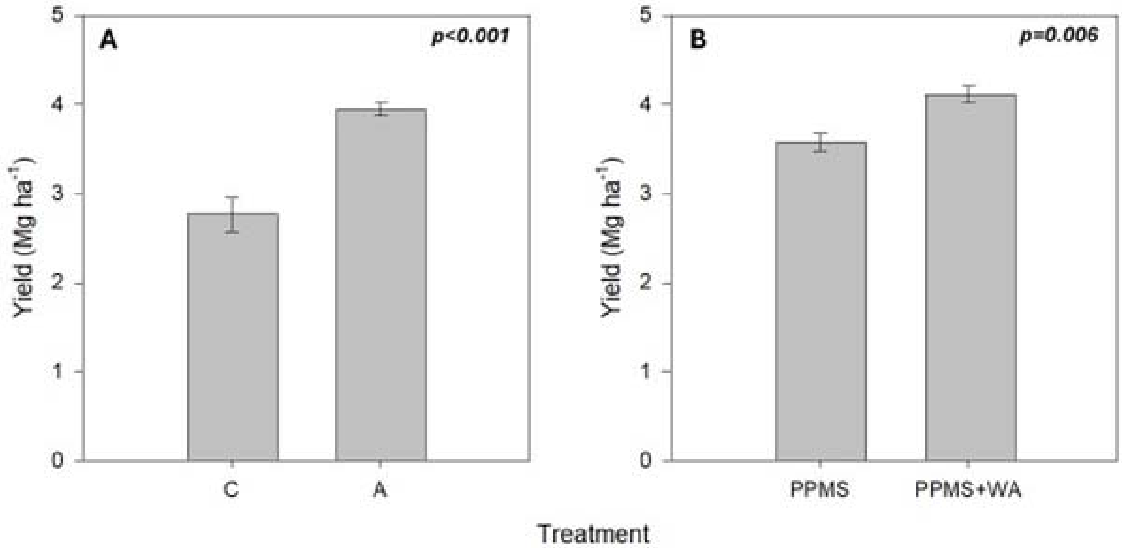

Fig. 2 Dry matter yield in A) the control (C) vs. soils receiving nutrient amendments (A) (L1) and B) soil receiving papermill biosolids (PPMS) and mixtures of PPMS and wood ash (WA) (L7) from greenhouse experiment two. Bars represent means (n = 4) ± standard error.

3.1.2 Experiment 2: Highest Forage Yields with Wood Ash and PPMS Applied Together

In Experiment Two, which evaluated WA and mixtures of WA and PPMS, amendments had a significant effect on yield ($F = 3.258$, $p = 0.004$), but not on the relative N use efficiency coefficient ($F = 1.102$, $p = 0.389$), which averaged 28.2 kg DM kg N^{-1}. Linear contrasts showed that all amendments increased yield relative to the control (L1, Fig. 2 A). Mixtures of PPMS and WA produced higher yields more than PPMS alone (L7, Fig. 2 B), and yield did not differ among mixture ratios.

3.2 Soil Health and Physical, Chemical and Biological Indicators

3.2.1 Experiment 1: No Effect of PPMS on Soil Health Scores and Variable Effects on Soil Indicators

There was no effect of amendment treatment on soil health score, although scores differed significantly among sites (Table 6). Mean scores were SR:91 (very high), SG:79 (high), and M:66 (medium).

BURST rates also differed by site (Table 6), with SG having the lowest (90.7 mg C-CO_2 kg^{-1} d^{-1}) compared to SR (114.8 mg C-CO_2 kg^{-1} d^{-1}) and M (112.6 mg C-CO_2 kg^{-1} d^{-1}). There was site by treatment interactions for SLAN, SOM, and WAS (Table 6). SLAN did not differ by treatment in M (mean 220.6 mg N-NH_3 kg^{-1} d^{-1}) but was highest in the PPMS+ treatment in SR (Table 7) and lowest in the PPMS+ treatment in SG (Table 7). SOM concentration was unaffected by treatment in SR (53.9 g kg^{-1}) but increased with PPMS in both M and SG (Table 7). WAS was unaffected by treatment in SG (mean 47%), whereas in SR and M soils the mineral fertilizers alone had the lowest WAS and PPMS increased WAS in M (Table 7). POXC did not differ among sites or treatments (Table 6; mean 700.5 mg kg^{-1}).

There was no significant interaction between site and nutrient treatment on the post-harvest concentrations of P, K, Ca and Mg (Table 6). There was no effect of nutrient amendment treatment on post-harvest concentrations of Ca and Mg and between sites, SR (2210 mg Ca kg^{-1}, 526 mg Mg kg^{-1}) > M (1823 mg Ca kg^{-1}, 402 mg Mg kg^{-1}) > SG (1656 mg Ca kg^{-1}, 300 mg Mg kg^{-1}). There was a significant effect of site and treatment for post-harvest concentrations of P and K. PPMS+ had the highest concentration of P and the treatments with mineral fertilizers had the highest concentration of K (Fig. 3). The lowest concentrations of P and K were in the M soil

Table 6 Two-way ANOVA results assessing the effects of site and nutrient amendment treatment on soil health score, soil health indicators, and other physical and chemical properties of the soils in experiment one. Significant effects are indicated in bold.

	Treatment		Site		Treatment*Site	
Parameter	*F*	*p*	*F*	*p*	*F*	*p*
Soil Health Score	0.928	0.437	72.709	*<0.001*	0.975	0.456
BURST	0.382	0.767	5.357	*0.009*	1.38	0.249
Solvita Labile Amino Nitrogen	1.515	0.227	85.223	<0.001	2.489	*0.041*
Soil Organic Matter	3.78	0.019	275.298	<0.001	3.927	*0.004*
Permanganate Oxidizable Carbon	1.8	0.165	1.363	0.256	1.363	0.256
Wet Aggregate Stability	1.819	0.161	48.186	<0.001	2.991	*0.018*
Bulk Density	3.682	0.021	286.505	<0.001	3.243	*0.012*
pH	35.487	<0.001	404.854	<0.001	5.898	*<0.001*
CEC	1.57	0.212	43.143	*<0.001*	1.832	0.120
Total N	5.069	0.005	1643.612	<0.001	4.267	*0.002*
C:N	7.437	<0.001	627.985	<0.001	3.024	*0.017*
P	5.217	*0.004*	2147.395	*<0.001*	0.508	0.798
K	10.431	*<0.001*	93.478	*<0.001*	2.279	0.058
Ca	0.829	0.487	36.098	*<0.001*	0.889	0.513
Mg	0.399	0.754	189.386	*<0.001*	1.588	0.179

Table 7 Soil amendment application effects on soil properties in experiment one. Treatments included control (C), mineral fertilizers (F), pulp and papermill sludge (PPMS), and PPMS + F and sites included soils collected from Slate River Valley (SR), Murillo (M), and South Gillies (SG). Values are means (n = 4) and values with different letters differ significantly at the 5% risk threshold based on Tukey's test.

Soil Property	Treatment	SR		M		SG	
SLAN (mg kg^{-1} d^{-1})	C	336.7	b	196.6		359.2	a
	F	325.2	b	199.5		374.3	a
	PPMS	338.7	b	229.7		371.1	a
	PPMS + F	379.	a	256.7		330.6	b
Organic Matter (g kg^{-1})	C	54.5		43.5	a	58.3	b
	F	54.8		43.0	a	62.3	ab
	PPMS	53.0		45.8	b	66.5	a
	PPMS + F	53.3		44.5	b	64.5	a
Water Stable Aggregates (%)	C	77.6	a	40.5	b	60.9	
	F	60.1	b	36.9	c	70.5	
	PPMS	70.1	a	48.0	a	72.0	
	PPMS + F	71.5	a	50.1	a	61.1	
Bulk Density (g cm^{-3})	C	0.98	b	1.16		0.97	
	F	1.00	b	1.20		0.97	
	PPMS	0.99	b	1.19		1.00	
	PPMS + F	1.03	a	1.16		1.02	
pH	C	6.8	b	6.7	a	5.5	b
	F	6.6	c	6.2	b	5.4	c
	PPMS	7.0	a	6.7	a	6.1	a
	PPMS + F	7.0	a	6.5	b	5.6	b
Total Nitrogen (g kg^{-1})	C	2.8		1.3	b	2.9	b
	F	2.8		1.3	b	3.0	ab
	PPMS	2.8		1.4	a	3.2	a
	PPMS + F	2.7		1.4	a	3.2	a
C:N Ratio	C	11.4		13.9		11.3	b
	F	11.4		13.9		11.4	b
	PPMS	11.3		14.1		11.6	b
	PPMS + F	11.3		14.5		12.1	a

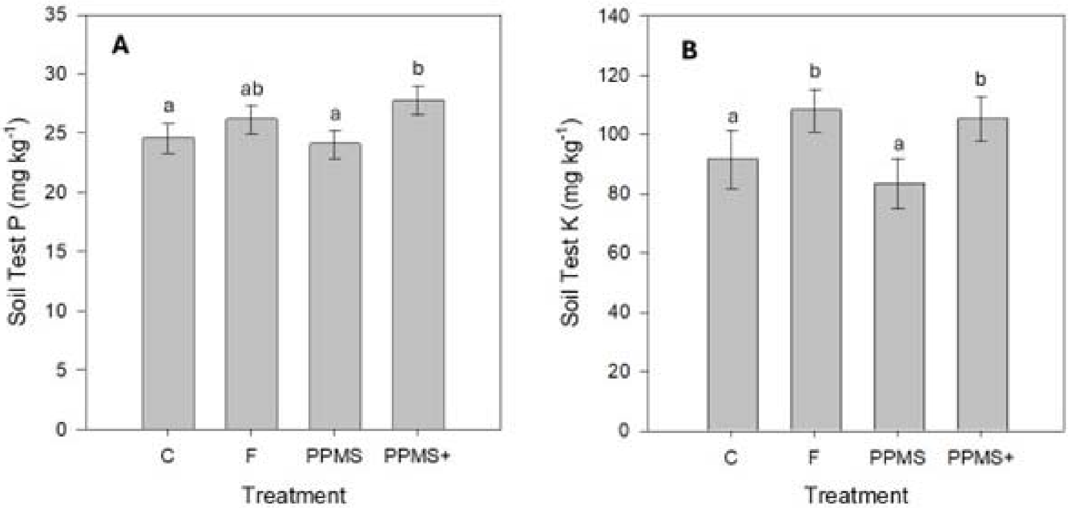

Fig. 3 The effects of nutrient amendment treatments on A) soil test P and B) soil test K. Treatments include control (C), mineral fertilizer (F), papermill biosolids (PPMS) and papermill biosolids with mineral fertilizer (PPMS+) in experiment one. Bars are means (n = 4) ± standard error. Different lower-case letters indicate significant differences at p < 0.05.

Table 8 One-way analysis of variance results evaluating the effects of nutrient amendment treatment on soil health scores, soil health indicators, and other soil properties in greenhouse experiment two. Significant differences are bolded.

Parameter	F	p-value
Soil Health Score	6.941	<0.001
BURST	7.117	<0.001
Solvita Labile Amino Nitrogen	3.114	0.005
Soil Organic Matter	5.353	<0.001
Permanganate Oxidizable Carbon	3.738	0.001
Wet Aggregate Stability	1.580	0.147
pH	11.485	<0.001
Bulk Density	4.762	<0.001
N	14.004	<0.001
C:N	6.187	<0.001
P	12.960	<0.001
K	12.038	<0.001
Mg	2.380	0.025
Ca	1.543	0.159
CEC	1.420	0.206

(4.6 mg kg^{-1}, 62.5 mg kg^{-1}) and the highest concentrations were in SR (58.9 mg kg^{-1}, 1171 mg kg^{-1}). Concentrations in SG were lower in SG (13.4 mg kg^{-1}) compared to SR but similar for K (111.6 mg kg^{-1}).

Bulk density, pH, N, and the C:N ratio showed significant site by treatment interactions (Table 6). Treatment effects on bulk density occurred only in SR (Table 7), where PPMS+ slightly increased bulk density. In SG and M, mineral fertilizers lowered soil pH, and PPMS alone increased pH in M. PPMS+ marginally increased N concentrations and the C:N ratio in SG, and PPMS increased N concentration in M without affecting the C:N ratio (Table 7).

3.2.2 Experiment 2: Consistent Improvements in Soil Health and Soil Indicators with PMR Application

Amendments significantly affected soil health scores, BURST, SLAN, SOM, POXC, but not WAS (mean 63%) (Table 8). Any amendment increased the soil health score by two points; PMR increased the score by four points. Adding fertilizers to WA or PPMS alone did not alter scores (L3, p = 0.267; L4, p

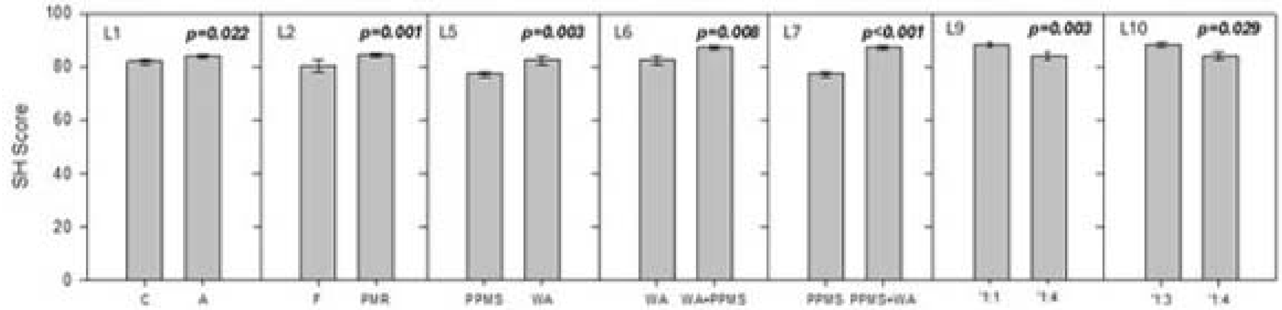

Fig. 4 Linear contrast results (means and standard error of the mean) from experiment two for soil health (SH) scores. Comparisons in the contrast analysis included the control (C), amendment application (A), mineral fertilizers (F), papermill residuals (PMR), pulp and papermill biosolids (PPMS), wood ash (WA) and combinations of WA and PPMS in ratios WA:PPMS of 1:1, 1:3, and 1:4. Differences are significant at $p < 0.05$.

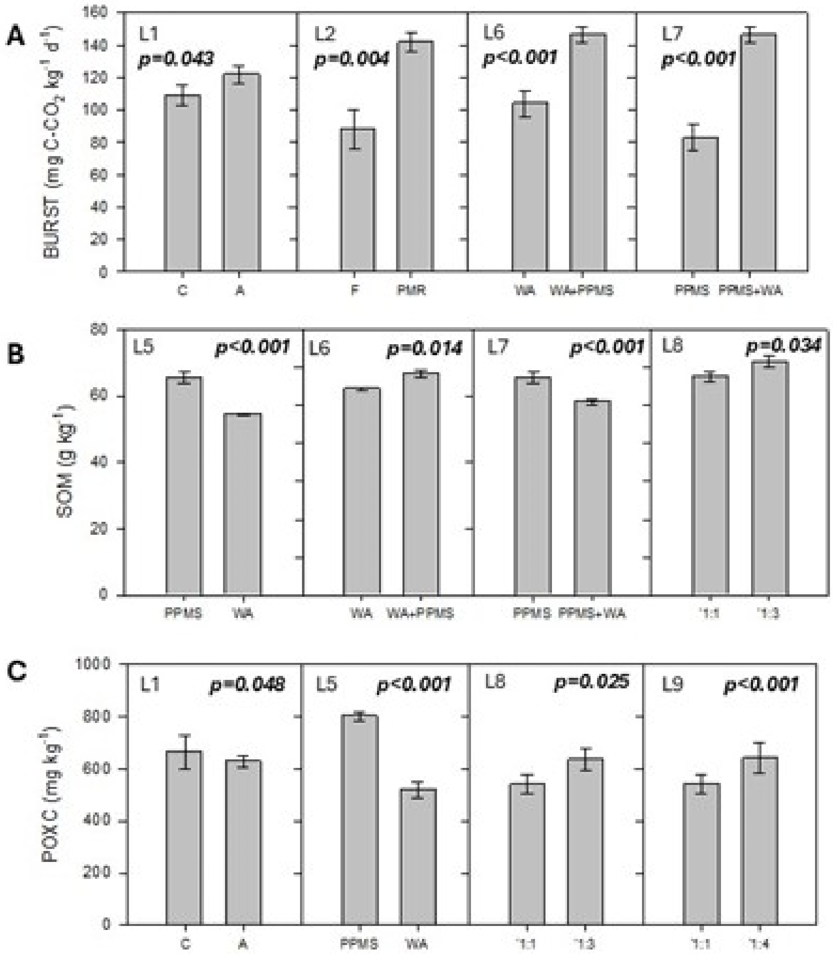

Fig. 5 Linear contrast results (means and standard error of the mean) from Experiment Two for A) BURST, B) soil organic matter (SOM), and C) permanganate oxidizable carbon (POXC). Comparisons in the contrast analysis included the control (C), amendment application (A), mineral fertilizers (F), papermill residuals (PMR), pulp and papermill biosolids (PPMS), wood ash (WA) and combinations of WA and PPMS in ratios WA:PPMS of 1:1, 1:3, and 1:4. The contrasts (L values) represent the hypotheses being tested (Table 5). Differences are significant at $p < 0.05$.

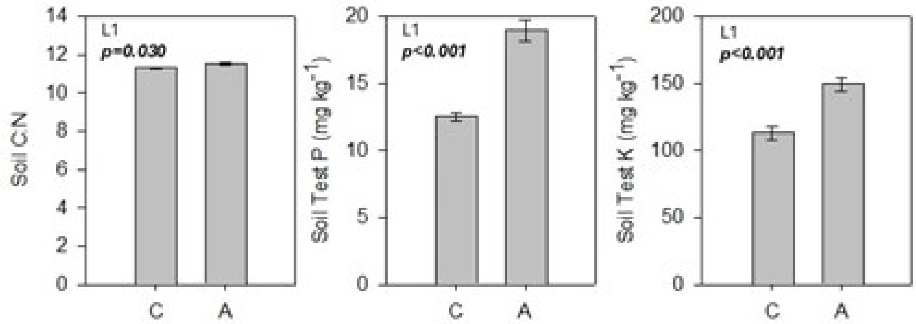

Fig. 6 Linear contrast results (means and standard error of the mean) for the soil C:N ratio, soil test P and soil test K, which is comparing the control and amendment treatments. (C, control, no amendment; A, soil receiving amendment). Differences are significant at $p < 0.05$.

Table 9 Linear contrast results (means) from Experiment Two for soil pH, bulk density, soil test P, K, total nitrogen, and the C:N ratio. Treatment comparisons in the contrast analysis included application of mineral fertilizers (F), papermill residuals (PMR), pulp and papermill biosolids (PPMS), wood ash (WA) and combinations of WA and PPMS in ratios WA:PPMS of 1:1, 1:3, and 1:4, as explained in Table 5. The header in the table refers to the hypothesis the contrast (L) is testing. Values with different letters differ significantly at the 5% risk threshold.

Soil Property	L2		L3		L4		L5		L6	
	F	PMR	WA	WA+F	PPMS	PPMS+F	PPMS	WA	WA	WA+PPMS
pH	5.4b	5.6a	5.7a	5.5b	5.8a	5.6b	5.8a	5.5b	5.5b	5.6a
Bulk Density	0.97	0.98	0.97	0.96	0.97	0.97	1.01a	0.99b	0.99	0.96
P	13.8b	19.4a	21.4	20.3	18.6	18.8	13.8b	22.3a	22.3	20.4
K	122.8b	151.8a	150.5b	176.5a	136.6b	162.4a	105.3b	161.3a	161.3	164.3
Total N	3.0	2.9	2.9	2.8	3.0	3.0	3.2a	2.7b	2.7b	2.9a
C:N	11.4	11.5	11.5	11.4	11.5	11.6	11.8a	11.5b	11.5	11.4
Soil Property	L7		L8		L9		L10			
	PPMS	PPMS+WA	1:1	1:3	1:1	1:4	1:3	1:4		
pH	5.8a	5.6a	5.6	5.6	5.6	5.7	5.6	5.7		
Bulk Density	1.01a	0.96b	0.94	0.95	0.94b	0.98a	0.95b	0.98a		
P	13.8b	20.4a	16.1b	20.6a	16.1b	24.4a	20.6b	24.4a		
K	105.3b	164.3a	157.5b	181.6a	157.5	153.6	181.6a	153.6b		
Total N	3.2a	2.9b	2.9b	3.2a	2.9a	2.7b	3.2a	2.7b		
C:N	11.8a	11.4b	11.3	11.4	11.3b	11.6a	11.4	11.6		

= 0.368). Mixtures of WA and PPMS increased soil health scores by five points relative to WA alone and by ten points relative to PPMS alone (Fig. 4). Among mixtures, the 1:4 (WA:PPMS) ratio produced scores four to five points lower than the 1:1 and 1:3 mixtures (Fig. 4), which did not differ from each other (L8, $p = 0.382$).

All amendments increased BURST (L1, Fig. 5 A), with greater effects for PMR compared to mineral fertilizers (L2), and for mixtures, compared to single amendments (L6–7). Mixtures also slightly increased SLAN relative to WA alone (L6, $p < 0.001$) and the 1:3 ration produced higher SLAN than 1:1 (L8, $p = 0.002$). PPMS increased SOM concentrations more than WA (L5, Fig. 5 B) and SOM concentrations were higher when PMR were applied alone rather than as mixtures (L6–7). The 1:3 mixture had higher SOM concentrations than the 1:1 and 1:4 mixtures (L8–10). All amendments reduced POXC relative to the control (L1). PPMS alone produced higher POXC than WA alone (L5, Fig. 5 C), and the 1:3 and 1:4 mixtures had higher POXC than the 1:1 mixture (L8–9), with 1:4 slightly exceeding 1:3 (L10).

Amendments significantly affected soil pH, bulk density, concentrations of N, P, K, Mg and the C:N ratio, but not CEC (19.3 Meq 100 g^{-1}) or concentrations of Ca (1739 mg kg^{-1}) (Table 8). Amendments increased the concentrations of P, and K, and the C:N ratio (L1, Fig. 6). PMR increased pH, and concentrations of P and K more than mineral fertilizers (L2, Table 9), and applying WA or PPMS without supplemental fertilizers increased pH and K (L3–4, Table 9). Compared to WA, PPMS increased soil pH, bulk density, N, and the C:N ratio, whereas WA resulted in higher concentrations of P, K and Mg (L5, Table 9). Mixtures of WA and PPMS increased soil pH, N, and the C:N ratio and lower soil bulk density relative to WA alone (L6, Table 9). PPMS alone produced higher pH, bulk density, N, and C:N ratio, but lower concentrations of P, K, and Mg than the mixtures (L7, Table 9). Mixture comparisons showed: 1:3 had higher concentrations of N, P and K compared to 1:1 (L8, Table 9); 1:4 had higher bulk density, C:N ratio (11.3 vs. 11.6, $p = 0.007$) and P concentrations, but lower N concentrations than 1:1 (L9, Table 9); and 1:3 had lower bulk density and P concentration and higher concentrations of C, N and K compared to 1:4 (L10, Table 9).

4. Discussion

The goal of our study was to determine whether operational application rates of PMR, as defined by Ontario regulations, can produce measurable benefits to soil properties and forage crop yield. By applying PMR at rates far below those commonly reported in the literature (e.g., [6], we observed improvements to crop yield, soil health scores, and several soil properties, although the magnitude and direction of effects differed among soils. These results indicate that PMR, when applied within agronomic recommendations and regulatory limits, can contribute to improved crop performance and soil conditions.

We examined the effects of PPMS applied at regulated rates to three soils differing in chemical and physical properties in a greenhouse experiment. We hypothesized that PPMS would increase forage crop yield and improve soil properties, with the greatest benefits realized in the coarser textured soils typically associated with lower productivity. Unexpectedly, no amendment increased dry matter yield in the coarser textured Murillo (M) soil, despite its low P and K concentrations and coarser texture. Although dry matter yield was slightly higher, though not significantly so, under mineral fertilizers, none of the PMR treatments improved performance in this soil. Moisture limitations at the microscale may have inhibited decomposition and nutrient release from the PPMS, even though bulk soil moisture was maintained at optimal levels. Reduced dry matter yield and lower relative N efficiency under PPMS support the interpretation that PPMS constrained growth in this soil, and follow-up studies will be required to identify the underlying mechanisms. We acknowledge this as a limitation of the study.

In the other two soils, dry matter yields were comparable to those reported in field studies with similar forage crops [1, 38, 39]. This suggests that that forage nutrient requirements can be met through the application of PPMS in most soils and that producers may consider substituting PPMS for mineral fertilizers. In one soil (SR), nitrogen use efficiency was highest under PPMS, whereas no treatment effect was observed in the SG soil. As noted by [40] the N efficiency coefficient approach does not account for mineralization of soil organic N during the growing season or the existing soil N stores. Total N concentrations were similar in the two soils, but the texture differed, which may have affected the mineralization of pre-existing N stores during the experiment that could explain the difference in response.

Only one soil required a pH adjustment to support optimal crop growth; this requirement was met with the application of WA alone or in combination with PPMS. WA and PPMS applied together consistently increased dry matter yield, and the mixture ratios had no measurable effect, underscoring the importance of simultaneously correcting soil pH and nutrient supply.

Yields did not increase further with supplemental fertilizers, indicating that PMR alone may be sufficient to meet nutrient requirements. These yield responses were accompanied by improvements in soil health scores and soil health indicators.

Soil health scores ranged from medium for the M soil, to high in SG and very high in SR, consistent with values reported in [41] for soils in the area, acknowledging that they measured a broader suite of indicators. In experiment one, PPMS alone did not affect soil health scores; however, in experiment two where WA and PPMS were applied alone and in combination to the SG soil there were significant improvements to soil health. The greatest increases occurred with PPMS and WA (ratios 1:1 and 1:3; WA:PPMS), which elevated the score into the very high range. This demonstrates that PMR can rapidly enhance soil function under favourable moisture conditions, particularly in acidic soils requiring lime. Properties of the soils in our study are consistent with soils that have not been severely degraded, and larger improvements may be realized in more degraded systems [42].

Turner et al. [6] reported that PPMS can increase CEC, pH, aggregate stability, organic matter/organic carbon, and nutrients, and decrease in bulk density. In contrast, our study detected no effect on CEC and only limited effects on pH, aggregate stability, organic matter/organic carbon and bulk density, likely reflecting the relatively good condition of local soils. Application of mineral fertilizer decreased pH and WAS, consistent with previous findings [42], while PPMS tended to increase SOM, without altering bulk density. PMR generally increased soil test P and K, with limited effects on Ca and Mg.

Our PMR application rate (9 Mg ha^{-1}) was substantially lower than those reported in the literature (20–225 Mg ha^{-1}) [6]. Repeated annual applications at operational rates could, over several years, produce cumulative benefits more consistent with published studies, while remaining far below the regulatory thresholds for heavy metals. We acknowledge that some PMRs can contain other trace metals and chemicals that can accumulate and impact environmental and human health (e.g., per- and polyfluoralkyl substances). The composition of PMR is mill-specific, and regulatory compliance does not necessarily account for all possible contaminants or associated risks. Based on site-specific PMR metal concentrations and applications rates used in this study, cumulative metal loading would not limit annual application., and neither would the dioxins and furans, which were measured but not currently regulated in Ontario. The primary uncertainty is weather, particularly soil moisture availability, which is a key control on decomposition, nutrient mineralization, and thus timing and magnitude of PMR derived benefits.

5. Conclusions

Although the benefits of land applying PMR to improve crop yield and soil properties are well established, most published studies have applied PMR at rates far exceeding Ontario's maximum annual permissible annual rate of 20 Mg ha^{-1} (wet weight). Concerns remain regarding the presence of heavy metals and other toxins, such as dioxins and furans. In our study, application rates were constrained by the concentrations of plant available N and P in the PMR, and the resulting metal additions were lower than the standard error of the soils' background metal concentrations. Despite these comparatively low application rates, we observed clear agronomic benefits, most notably forage yield, that exceeded those achieved with mineral fertilizers in the greenhouse trial. These results indicate that land application of PMR in the Thunder Bay area has the potential to reduce reliance on mineral fertilizers and conventional liming materials. Furthermore, diverting PMR from landfills would provide economic advantages to both the producers of these materials and local growers, while simultaneously improving soil conditions and generating broader environmental benefits. We acknowledge that these results are based on a greenhouse experiment where temperature and moisture conditions are optimized and that validation in the field is required to confirm our findings.

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