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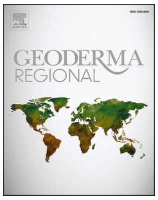

The amendment value of pulp and paper mill sludges in Finnish coarse-textured soil

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ABSTRACT

As a source of exogenous organic matter, pulp and/or paper mill sludges (PPMS) may have beneficial effects on crop productivity and soil chemical and physical properties. This study's aim was to assess the impacts of two different PPMS materials on crop yields, the quality of percolation water, and soil chemical and hydraulic properties in a three-year field experiment on a silt loam soil in East Central Finland. Fresh (FPMS) and lime-stabilized (LPMS) sludges were applied once at rates of 21–28 fresh-Mg ha^{-1} in the spring prior to the sowing of grass ley under barley as a cover crop and incorporated into the upper 7 cm soil layer. Supplemental nitrogen (N) was applied at levels of 40 and 80 kg ha^{-1}. A decrease of barley grain yield due to N immobilization was observed at the N level 40 kg ha^{-1}, but the standard N application rate (80 kg ha^{-1}) connected with a moderate C:N ratio (FPMS 27:1, LPMS 24:1) was adequate to avoid significant yield losses. In the second and third year following the PPMS applications, there was a tendency for positive residual effects on the total dry matter yield of grass ley, which could be attributed to slow mineralization of sludge-N. The application of LPMS increased the pH in surface soil by 0.5–0.7-units and Ca concentration by 240–660 mg L^{-1} of soil relative to the non-amended control over the study period. In the year of PPMS applications, the amendments produced a significant increase (about 2.0 g kg^{-1}) in the total carbon (C) concentrations in the uppermost 10 cm soil layer relative to the non-amended soil. During the following years, the change in soil C was no longer measurable, indicating relatively fast decomposition of sludge-C. Saturated hydraulic conductivity tended to be 1.4 to 2.3 times higher in the PPMS-treated soils than in the non-amended soil. Except for the decline in readily plant-available water, the other common water retention parameters were not significantly affected by the PPMS amendments. There were significant positive treatment effects on the amount of water retained between −13 and − 316 kPa matric potentials, suggesting an increase in medium-sized pores contributing to water storage in the soil. To maintain or enhance the beneficial direct and indirect effects of PPMS on crop yields and soil physico-chemical properties, repeated applications of PPMS are required, possibly combined with the use of organic fertilizers, especially during grass ley years.

1. Introduction

Various organic materials are increasingly used as fertilizers and soil conditioners in different crop production systems to respond to the demands of circularity (e.g., Harder et al., 2021) and maintenance of soil health (Lal, 2016). The application of organic amendments such as livestock manure, biosolids, biowastes, crop residues, green manures and various sidestreams may have multiple beneficial effects on soil biological, chemical, and physical properties, especially when amending degraded coarse-textured soils (Diacono and Montemurro, 2010; Larney and Angers, 2012). Sandy soils are widely distributed throughout the world and cover 31 % of the total land area (Huang and Hartemink, 2020). At the European scale, topsoils with relatively high sand contents are found in the Nordic and Baltic countries (Ballabio et al., 2016). Besides a lower intrinsic specific surface area, cation exchange capacity (CEC), and soil organic carbon (SOC) content, most coarse-textured soils

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are characterized by a lower field capacity, permanent wilting point, and plant-available water capacity, whereas finer-textured soils are characterized by a lower gas permeability and saturated hydraulic conductivity (Yost and Hartemink, 2018; Huang and Hartemink, 2020). The relative importance of SOC in contributing to CEC or water retention is higher in coarse- than in fine-textured soils (Minasny and McBratney, 2018), and concerning attainable crop yields, sandy soils may therefore potentially benefit more from organic inputs and increasing SOC than clayey soils (Hijbeek et al., 2017).

As a source of exogenous organic matter (OM), sidestreams originating in the forest industry have been reused successfully in agriculture as a soil amendment, also providing a desired alternative solution to incineration and landfilling (Camberato et al., 2006; Faubert et al., 2016; Turner et al., 2022). The type, composition, and quantity of sidestreams the forest industry generates vary substantially among different production processes and mills. The main residual sidestreams of Finnish pulp and/or paper mills are different types of sludges (PPMS) such as fiber sludge from the cleaning and processing of raw materials, and primary sludge and biosludge from primary and secondary wastewater treatments (Hassan et al., 2019). On a dry matter (DM) basis, nearly 580,000 Mg of PPMS (includes fiber, coating, sewage, and deinking sludges) are generated in Finland annually, containing approximately 230 Mg of phosphorus (P) and 1160 Mg of nitrogen (N), the proportion of soluble N being 30 Mg. The majority of PPMS biomasses (c. 66 %) are typically incinerated for energy production, while smaller quantities are used in earthworks, and merely a minor part is applied to soil as fertilizer or soil conditioning products (Marttinen et al., 2018). Excluding manures, only about 5 % and 2 % of all total P and N fertilization used in Finnish agriculture is based on recycled nutrients (Marttinen et al., 2018), indicating potential for increasing the utilization of various sidestreams. Recently, there have also been challenges with the availability of inorganic fertilizers and especially with their high and volatile prices (Hebebrand and Laborde, 2023), which encourages part of the mineral fertilization to be replaced with nutrient-rich PPMS, for example.

In recent years/decades, the land application of PPMS to agricultural soils has become more common due to their potential to improve crop productivity and soil quality by increasing soil organic matter (SOM) content, fertility, pH, microbial activity, aggregation, and nutrient and water holding capacity (Camberato et al., 2006; Faubert et al., 2016; Turner et al., 2022). As SOM components are important for soil structural development and stability due to their binding and cementing actions (Tisdall and Oades, 1982; Dexter, 1988), the suitability of PPMS applied on clay soil as a water protection measure has been explored via rainfall simulations with a positive outcome on percolate quality (Muukkonen et al., 2009; Rasa et al., 2021).

In field conditions, various PPMS have induced an increase in aggregate stability and macroaggregate size (Nemati et al., 2000; N'Dayegamiye, 2009). Moreover, a sludge-induced increase in soil water holding capacity, especially in plant-available water retention following the PPMS application is expected to have a favorable influence on crop growth (Camberato et al., 2006). Beneficial amendment effects on soil water retention characteristics were obtained in sandy soils by Zibilske et al. (2000), Foley and Cooperband (2002), and Räty et al. (2023). Potential negative agronomical issues related to the land application of PPMS are typically associated with an accumulation of heavy metals in soils and N immobilization, leading to short-term N-availability limitation (Camberato et al., 2006; Faubert et al., 2016; Turner et al., 2022). However, a tendency for N immobilization depends on the quality and amount of sludge and soil temperature, for example, and might be mitigated by a co-application of supplemental N, delayed planting of non-legumes, planting of legumes, or composting of primary sludge (Camberato et al., 2006; Turner et al., 2022). Furthermore, Nemati et al. (2000) detected a decline in water stable aggregate stability following sludge applications for well-aerated sandy loam soil, in contrast with the positive effects recorded in more clayey soils. This adverse effect on structural stability was attributed to increased soil microbial activity, the decomposition of the sludge amendment, and the destruction of structure-supporting organo-mineral linkages through a priming effect.

In this study, a three-year field experiment was established to assess the expected positive and possible negative impacts of two different types of nutrient-rich PPMS on productivity, yield quality, and the risk of nutrient losses in a boreal silt loam soil in North Savo in East Central Finland. The sludge amendments were included in part of rotational leys typical of the area by applying them as a single dose prior to the sowing of grass ley under a barley cover crop in the spring. The effects of the PPMS amendments on crop yield quantity and quality, selected element content in soil and its release to percolating water, and soil hydraulic conductivity and water holding capacity were measured to provide science-based practical information for farmers about the impacts of PPMS materials on crop productivity and the environmental nutrient loading in coarse-textured arable soils.

2. Material and methods

2.1. Study site

A field experiment was conducted in 2020–2022 on the premises of Natural Resources Institute Finland (Luke) in Kuopio, Maaninka, in the province of North Savo in East Central Finland (63°8.42′N, 27°19.30′E). On the study site, the topsoil (0–20 cm) was silt loam, and the 20–40 and 40–60 cm layers of subsoil consisted of silt loam and sandy loam respectively. According to the WRB system (IUSS Working Group WRB, 2022), the experimental field was classified as Stagnic Regosols (Loamic, Aric, Drainic, Ochric) (Yli-Halla and Räty, 2024). The mean values of selected soil properties on the experimental site for the 0–20, 20–40, and 40–60 cm soil layers are summarized in Supplementary Table 1. Briefly, the mean topsoil pH (H_2O) was 6.0, the potential CEC was 12.6 cmol(+) kg^{-1}, and the total C and N content was 18.0 and 1.34 g kg^{-1} respectively.

The growing degree day (GDD) sum, starting when the daily mean temperature rises permanently above +5 °C (threshold 10 days), ranged from 1379 °C days in 2022 to 1487 °C days in 2021, and lasted an average of 165 days (Finnish Meteorological Institute, 2022). The mean annual precipitations in 2020, 2021, and 2022 were 678, 624, and 586 mm respectively, and the respective mean annual temperatures were 5.9, 3.7, and 4.5 °C (Supplementary Fig. 1). In 2020, the mean annual precipitation and temperature exceeded the long-term average by 59 mm and 2.2 °C, whereas 2021 and 2022 were close to the long-term averages in the period 1991–2020 (Finnish Meteorological Institute, 2022; Jokinen et al., 2021).

2.2. Experimental design

The experimental setup included two types of organic soil amendments originating in the sidestreams of the pulp and/or paper mill industry. Fresh and non-processed sludge (FPMS) was received from Stora Enso Oyj, Varkaus, Finland, and lime-stabilized sludge (LPMS) from Fortum Waste Solutions Oy, Kuopio, Finland (Table 1). The application rates of the sludges were based on the maximum permissible cadmium (Cd), using the analysis results originating in previous batches of the sludge materials. Under Finnish legislation, the threshold value for Cd content is 1.5 mg kg^{-1} DM, and the land application rate is limited to 7.5 g Cd ha^{-1} during a five-year period (1.5 g Cd ha^{-1} yr^{-1}) in agriculture (MMMa 964/2023, 2023). The amounts of C were then equalized among the sludge treatments. On a fresh mass basis, the single application rates for LPMS and FPMS corresponded to 28 and 21 Mg ha^{-1} respectively. Two levels of supplemental N mineral fertilizer (40 and 80 kg ha^{-1}) were used. In addition to these four soil amendment treatments (FPMS-40 N, FPMS-80 N, LPMS-40 N, and LPMS-80 N), four treatments (Min-N) receiving increasing mineral N fertilizer application

Table 1

Chemical composition (means ± standard deviations; $n = 3$) on a dry matter (DM) basis of two pulp and paper mill sludge materials applied in the field experiment in the spring of 2020, and application rates of the sludges, nutrients, and heavy metals within the sludge materials in 2020. The rates are presented on a dry matter basis, using the means of the chemical content of the sludges. FPMS = fresh pulp and paper mill sludge, and LPMS = lime-stabilized pulp and paper mill sludge.

	Chemical composition			Application rates		
	Units	FPMS	LPMS	Units	FPMS	LPMS
Application rate				(kg ha^{-1} DM)	9429	9884
DM[1]	(%)	45 ± 0.8	35 ± 0.7			
BD[2]	(g cm^{-3})	0.67 ± 0.02	1.11 ± 0.02			
pH_{H2O}[3]		6.9 ± 0.3	9.4 ± 2.3			
C:N ratio		27:1	24:1			
C[4]	(g kg^{-1} DM)	403 ± 1.1	354 ± 9.9	(kg ha^{-1} DM)	3284	2942
N total[5]	(g kg^{-1} DM)	15 ± 0.0	15 ± 0.6	(kg ha^{-1} DM)	141	145
N soluble[6]	(g kg^{-1} DM)	1.7 ± 0.06	1.3 ± 0.15	(kg ha^{-1} DM)	16	13
H_2O-TP[7]	(g kg^{-1} DM)	0.05 ± 0.00	0.04 ± 0.00	(kg ha^{-1} DM)	0.52	0.41
$NaHCO_3$-TP[7]	(g kg^{-1} DM)	0.17 ± 0.00	0.27 ± 0.02	(kg ha^{-1} DM)	1.6	2.7
NaOH-TP[7]	(g kg^{-1} DM)	0.85 ± 0.02	0.26 ± 0.04	(kg ha^{-1} DM)	8.0	2.5
HCl-TP[7]	(g kg^{-1} DM)	0.51 ± 0.01	1.7 ± 0.11	(kg ha^{-1} DM)	4.9	17
Hedley-P_{Sum}[7]	(g kg^{-1} DM)	1.6 ± 0.03	2.2 ± 0.15	(kg ha^{-1} DM)	15	22
P total[8]	(g kg^{-1} DM)	2.5 ± 0.00	3.0 ± 0.15	(kg ha^{-1} DM)	24	30
K[8]	(g kg^{-1} DM)	0.91 ± 0.06	1.13 ± 0.16	(kg ha^{-1} DM)	8.6	11
Ca[8]	(g kg^{-1} DM)	27 ± 0.0	66 ± 1.2	(kg ha^{-1} DM)	255	652
Mg[8]	(g kg^{-1} DM)	2.5 ± 0.12	2.6 ± 0.15	(kg ha^{-1} DM)	23	26
S[8]	(g kg^{-1} DM)	10 ± 0.3	2.3 ± 0.1	(kg ha^{-1} DM)	96	22
Na[8]	(g kg^{-1} DM)	1.1 ± 0.12	2.1 ± 0.06	(kg ha^{-1} DM)	11	21
Mn[8]	(g kg^{-1} DM)	1.3 ± 0.05	1.1 ± 0.13	(kg ha^{-1} DM)	12	10
Zn[8]	(g kg^{-1} DM)	1.6 ± 0.19	0.66 ± 0.07	(kg ha^{-1} DM)	15	6.5
Cu[8]	(mg kg^{-1} DM)	414 ± 34	50 ± 4.2	(kg ha^{-1} DM)	3.9	0.5
B[8]	(mg kg^{-1} DM)	23 ± 7.6	6.3 ± 1.53	(g ha^{-1} DM)	214	63
As[9]	(mg kg^{-1} DM)	1.3 ± 0.58	<1.0	(g ha^{-1} DM)	13	9.9
Cd[9]	(mg kg^{-1} DM)	2.1 ± 0.15	0.6 ± 0.06	(g ha^{-1} DM)	20	5.7
Cr[9]	(mg kg^{-1} DM)	70 ± 5.6	8.0 ± 1.00	(g ha^{-1} DM)	660	79
Hg[9]	(mg kg^{-1} DM)	0.2 ± 0.03	<0.05	(g ha^{-1} DM)	1.6	<0.5
Ni[9]	(mg kg^{-1} DM)	20 ± 2.6	<4.0	(g ha^{-1} DM)	189	<40
Pb[9]	(mg kg^{-1} DM)	36 ± 14	2.7 ± 0.58	(g ha^{-1} DM)	339	26

Indicators of pathogens were determined from FPMS material prior to field applications: salmonella; not verifiable and Eschericia coli; < 10 pmy g^{-1}.

[1] Dry matter (DM) content was determined gravimetrically by drying a sample in an oven at 105 °C according to SFS 3008:1990.

[2] Laboratory compacted bulk density (BD) was conducted in accordance with SFS EN 13040:2008 (mod).

[3] pH was measured in a sludge-water suspension in an extraction ratio of 1:5 v:v, shaking for 1 h (SFS-EN 13037:2011).

[4] Total C determination was based on the Dumas method of dry combustion using a Leco© TruMac CN determinator.

[5] Total N was determined by the modified Kjeldahl method using H_2SO_4/K_2SO_4 digestion with Se or $CuSO_4$ as a catalyst (EN 13654–1 mod.; EN 13342 mod.).

[6] Soluble N was determined by the modified Kjeldahl method (SFS 5505:1988 mod.), in which nitrate and nitrite are reduced with Devarda's alloy and organic matter is digested with H_2SO_4 in the presence of a Cu catalyst.

[7] Using the Hedley P fractionation procedure (Hedley et al., 1982) as modified by Sharpley and Moyer (2000), P reserves were sequentially extracted with deionized H_2O, 0.5 M $NaHCO_3$ (pH 8.5), 0.1 M NaOH, and 1.0 M HCl in a soil: solution ratio of 1:60 (w:v). Each P fraction was presented as total P (TP = inorganic P + organic P fractions) and Hedley-P_{Sum} as the sum of fractionated P pools.

[8] Concentrations of total P, K, Ca, Mg, S, Na, Mn, Zn, Cu and B were determined with a microwave-assisted acid digestion (HNO_3), which was followed by ICP-OES/ICP-MS analysis.

[9] Heavy metals (As, Cd, Cr, Hg, Ni, Pb) were determined in accordance with SFS-EN ISO 11885:2009; SFS-EN ISO 17294-2:2005.

rates of 0, 40, 80, and 120 kg ha^{-1} were included to obtain a N yield response curve, and to serve as control treatments without sludge application (Min-40 N and Min-80 N). All the treatments were arranged on the experimental field in a randomized complete block design with four replicates, including non-experimental plots between each experimental plot of 22.5 m^2 (1.5 m × 15 m). The residual effects of PPMS were investigated in the second and third experimental years following PPMS applications to soil.

The sludge materials were evenly applied to the soil surface of the predetermined plots on June 1, 2020. Mineral fertilizers were applied on the soil surface (Tume RL 1500 spreader), after which the plots were harrowed to a depth of about 7 cm. On June 2, 2020, the experimental field was sown with a mixture of timothy (*Phleum pratense* L., cv. Tryggve, seed rate 15 kg ha^{-1}) and meadow fescue (*Festuca pratensis* Huds., cv. Minto, seed rate 6 kg ha^{-1}) using barley (Brage, seed rate 188 kg ha^{-1}) as a cover crop. Therefore, cereal cover crop was harvested in 2020 and grass ley in 2021–2022. Table 1 presents the application rates of nutrients within PPMS materials in 2020. The application rates of mineral fertilizers for the barley in 2020 and the grass ley in 2021–2022 are given in Supplementary Table 2.

2.3. *Yields and plant sampling and analyses*

On September 14, 2020, the barley grain yields were collected from each treatment plot by harvesting an area of c. 12 m^2 (1.5 m × c. 8 m) with a plot combine harvester (Wintersteiger nurserymaster). The grain was dried and weighed after sorting. The grain DM content was determined by drying at 100 °C for 20–24 h, and the grain yields were calculated at 15 % moisture content. Of the grain quality factors, test weight (TW; kg hL^{-1}) and thousand grain weight (TGW; g) were determined. The crude protein (CP; g kg^{-1} DM) content of the grain was estimated using a near-infrared (NIR) reflectance spectroscopy technique, and the grain N content was calculated by dividing the protein content by an N-to-protein conversion factor of 6.25. For P, K, S, Ca, and Mg analysis, ground grain samples were microwave digested in concentrated HNO_3, and the elements were measured using ICP-OES spectrometry. The content of Cd was determined from grain samples in the Min-80 N, LPMS-80 N, and FPMS-80 N plots in accordance with SFS-EN ISO 17294-2:2016.

During grass ley growing years 2021–2022, the plots were harvested twice per growing season (Haldrup 1500 plot harvester) to a stubble height of 7 cm from an area of 12 m^2 (1.5 m × c. 8 m) and weighed with an onboard weighing system. Separate grass samples were collected from each plot for laboratory analyses. The first and second cuts took place on June 15 and August 4, 2021, and on June 22 and August 1, 2022, respectively. The DM content was determined from fresh samples by drying at 60 °C for 40–48 h. Digestibility value (D value) and the content of CP were analyzed using the NIR technique (Foss NIRSystems XDS analyzer), and the content of CP was converted into N using an N-to-protein conversion factor of 6.25. N uptake was calculated by DM yield x N content x 1000^{-1} for barley grains and grass. The content of P, K, Ca, and Mg was determined using an X-ray fluorescence (XRF) analytical technique, and S was determined following microwave digestion as above.

2.4. Soil sampling and analyses

Composite soil samples were taken from each plot by an auger at depths of 0–20 and 20–40 cm in October 2020, and September 2021, by bulking 10–12 subsamples into one sample per plot within each soil layer. In October 2022, soil samples were taken only from the plow layer (0–20 cm). Easily available (soluble) total N and inorganic N (NH_4^+-N and NO_3^--N) were extracted from fresh soil samples taken in 2020 and 2021 with 2 M KCl at a soil-to-solution ratio of 1:5 (w:v) for 2 h, and the concentrations of soluble total N, NH_4-N, and NO_3-N were analyzed from filtered samples with a Skalar San^{++} autoanalyzer. Soluble organic N (SON) was taken as the difference between soluble total N (after oxidative digestion) and inorganic N. Soil pH and electrical conductivity (EC) were measured in a soil-water suspension (1,2.5, v,v). Soil total C and N were determined with the dry combustion (Dumas) method using a Leco® TruMac CN determinator. Additionally, two replicate profile samples were taken from each plot in the Min-80 N, LPMS-80 N, and FPMS-80 N treatments by an auger (4.8 cm in diameter) to a depth of 20 cm or 40 cm in October 2020–2022. The soil core samples were sliced into 10 cm segments and dried at 40 °C for about seven days and then analyzed for soil C and N as above.

To analyze soil plant-available nutrient contents, the air-dried and sieved (a 2 mm sieve) samples were extracted with acid ammonium acetate (AAAc; 0.5 M CH_3COONH_4, 0.5 M CH_3COOH, pH 4.65, 1:10 v:v, 1 h; Vuorinen and Mäkitie, 1955). The concentrations of P were analyzed with a Thermo Scientific Aquakem analyzer and Ca, Mg, K, Na, and S with an ICP-OES. In Finnish soil testing, soil is measured out to analysis on a volume basis to overcome the considerable variation in the volume weights of the soil deriving from a large range of OM contents. The volume weight of dried and ground (< 2 mm) fine mineral soil, as used in the present study, is on average 1.0 kg L^{-1} (range 0.8–1.3 kg L^{-1}; Keskinen et al., 2016), which leads to equal values in mass-based conversion.

2.5. Leaching experiment

Soil monoliths (30 cm in diameter and about 40 cm in depth, four replicates per treatment) were drilled from the experimental plots in PVC cylinders (0.5 m in length) during October 2020. These monoliths were capped and stored in darkness at +2 °C until taken to the leaching experiments. At the beginning of the leaching experiment, each soil core was first saturated from below (via a drainage hole) for one day. After saturation, the drainable water was released, and the soil column was drained for 24 h.

Rainfall was simulated using a simple stationary droplet maker. The container filled with collected filtered natural rainwater was 0.5 m above the soil core, and droplets were allowed to form freely through needle holes on the bottom of the water container. Rain intensity was set to the typical Finnish level, 5 mm h^{-1} (Kuusisto, 1980). Simulated rainfall was applied for 4.5 h per day for four days to form 25 mm of rain per day and 100 mm in total. Percolation water was collected and weighed daily and united to form one collective sample representing the whole test period. Samples were stored at −18 °C for later analysis.

Water samples were analyzed for total N (unfiltered samples, SFS 3031 mod.; Skalar San^{++} autoanalyzer), NO_3-N (0.2 μm filtered samples, SFS 3030 mod., Skalar San^{++} autoanalyzer), Ca (ICP), Mg (ICP), S (ICP), total P (unfiltered samples, SFS 3026 mod., Skalar San^{++} autoanalyzer), dissolved P (0.2 μm filtered samples, SFS 3025 mod., Skalar San^{++} autoanalyzer), total suspended solids (TSS; gravimetric analysis, SFS-EN 872), and dissolved organic C (DOC; 1.6 μm filtered samples, Shimadzu TOC-V CSH analyzer).

2.6. Measurements of soil hydraulic properties

In September 2020, near-surface soil cylinder samples (2–7 cm depth) were taken in six (c. 250 cm^3) and five (c. 195 cm^3) replications from the Min-80 N, LPMS-80 N, and FPMS-80 N treatments to determine soil water retention characteristics and saturated hydraulic conductivity respectively. Prior to the measurements, the samples were saturated with standing tap water for two weeks by adjusting the water level to the midpoint of the cylinder.

The saturated hydraulic conductivity (K_{sat}) was determined by the principle of constant head method (Klute and Dirksen, 1986). Water was delivered to each sample cylinder first allowed to soak overnight before measurement. Then a constant head of water was maintained on the samples by allowing water to flow in and excess water to drain out. During the measurement period (lasting 18–364 min, mean 240 min), the amount of collected water (20.3–22.7 °C, mean 21.5 °C) was recorded gravimetrically several times (5–26, mean 14), and the measurements were continued until the flowrate was stabilized. The K_{sat} (cm min^{-1}) was calculated based on the application of Darcy's law using the equation: $K_{sat} = V_w \times l / (A \times t \times h)$, where V_w is the quantity of water that flows through the sample of cross-sectional area A (40.5 ± 0.30 cm^2) per unit of time t (min), and h is the hydraulic head difference (7.2 ± 0.35 cm) imposed across the sample length l (4.8 ± 0.07 cm). The change in the sample height was recorded with a digital vernier caliper.

The conventional soil water retention curve, i.e., the pF curve, was determined at 13 decreasing matric potentials of −0.1, −0.3, −3.2, −6.3, −10, −13, −20, −32, −63, −100, −316, −1585, and −39,811 kPa, using the procedure described in detail by Räty et al. (2023). Briefly, the water retention characteristics between −0.1 and −0.3 kPa and between −3.2 and −316 kPa were measured using a box with plastic foam at the bottom and an overpressure method with pressure plate extractors (Soilmoisture Equipment Corp., USA) respectively, with equilibration times ranging from three to 21 days. After equilibration at each matric potential, the water content of the sample was measured gravimetrically. Changes in the sample height (vertical shrinkage) and diameter (horizontal shrinkage) were measured after each drying step at six points per sample using a digital vernier caliper and at four points using a feeler gauge respectively. At the matric potentials of −1585 and −39,811 kPa, the soil water retention was determined by a relative humidity (i.e., vapor pressure) method in the desiccator containing the saturated salt solution (−1585 kPa; $(NH_4)_2C_2O_4$ and −39,811 kPa; NaCl; equilibration time 21 days), with three laboratory replicates.

The total porosity (TP) was taken as the volumetric water content at a − 0.1 kPa matric potential, i.e., in near-saturated conditions, field capacity (FC) at −10 kPa, and permanent wilting point (PWP) at −1585 kPa. Drainable water (DW), readily plant-available water (RPAW), and plant-available water (PAW) were defined as water held between −0.1 kPa and −10 kPa, between −10 kPa and − 316 kPa, and between −10 kPa and −1585 kPa respectively. The pore size ranges were also divided into wide coarse (wCP; > −6.3 kPa), fine coarse (fCP; −6.3 to −32 kPa), medium-sized (MSP; −32 to −1585 kPa), and fine pores (FP; < −1585 kPa), with the respective equivalent pore diameters of >46 μm, 46–9.1 μm, 9.1–0.2 μm, and < 0.2 μm (Blume et al., 2016). Furthermore, the BD was presented by dividing the mass of the oven-dried soil after −316 kPa by the volume of the sample at −0.1 kPa. The soil particle density (PD) was determined with two replicates based on a stoppered bottle pycnometer method as in Räty et al. (2023).

2.7. Statistical analyses

The experimental data were analyzed with linear mixed models to study the differences between the three treatments: LPMS, FPMS, and

Min-N control. N levels of 40 and 80 N were analyzed separately. The Min-0 N and Min-120 N treatments were only used for constructing functions presented in Figs. 1 and 2. The used fixed and random effects varied according to the models, depending on the sub-data structure for the specific research problem. The basic experimental design for the PPMS treatments was carried out with four randomized blocks in all the sub-data, but the total number of observations varied between 12 and 923.

PPMS treatments (Min-40 N (control), LPMS-40 N, FPMS-40 N or Min-80 N (control), LPMS-80 N, FPMS-80 N), pressure (13 values), measurement year (2 or 3 years), harvest time (twice for grass), soil depth (2 or 4 depths), and their interactions were used as categorical fixed effects in the models. Replicate, treatment plot, measurement year, harvest time, soil depth, sample, and pressure were used to define an appropriate correlation and variance structure (using random effects and specific covariance structures) for normally distributed response variable values.

The Bonferroni method (with a significance level of 0.05) was used in multiple comparisons of estimated class means for the PPMS treatments (partitioned comparisons if interactions with the treatment were included in the model). The statistical analyses were performed with the GLIMMIX procedure and REG procedure of SAS software (version 9.4).

The results of the monolith leaching experiment were analyzed with the MIXED procedure of SAS 9.4, with the treatment set as a fixed factor in the model, and the replicate was a random factor. Pairwise comparisons were made using the Tukey-Kramer test.

3. Results

3.1. Yields

The linear increasing yield response curve of barley on the increasing Min-N applications indicated N limitation of the grain yields (Fig. 1). According to the response curve, the grain yields obtained in LPMS-40 N, FPMS-40 N, LPMS-80 N, and FPMS-80 N were equal to yields obtainable with Min-N fertilizer application levels of 1, 28, 66, and 69 kg ha^{-1} respectively. In 2020, when the PPMS were spread, the grain yield of spring barley was an average of 2950 kg ha^{-1} for the Min-40 N level and 3900 kg ha^{-1} for the Min-80 N level (Supplementary Table 3). The LPMS-40 N treatment reduced the grain yield compared with Min-40 N control (−860 kg ha^{-1}; −26 %) and FPMS-40 N (−540 kg ha^{-1}; −18 %). The difference between FPMS-40 N and Min-40 N control was −320 kg ha^{-1} (−10 %) and not statistically significant. The N uptake of the LPMS-40 N treatment was lower than Min-40 N control (10.7 kg ha^{-1}; 24 %; Table 2). The yields at the higher N level (80 kg ha^{-1}) were comparable in all treatments. In contrast with the Min-40 N level, the N uptake of the FPMS-80 N treatment was lower than Min-80 N control (6.2 kg ha^{-1}; 11 %; Table 2).

In 2021, one year after PPMS spreading, the total DM yield of grass was an average of 5830 kg ha^{-1} year^{-1} for the Min-40 N level and 7660 kg ha^{-1} year^{-1} for the Min-80 N level (Supplementary Table 3). According to the yield response curve, FPMS-40 N and LPMS-40 N gave the same DM yield in the first cut as Min-N application levels 82 and 71 kg ha^{-1} respectively (Fig. 2). In the first cut, FPMS-80 N and LPMS-80 N exceeded the yield response curve, and in the second cut, LPMS-40 N, FPMS-40 N, and LPMS-80 N were located at the same point as the corresponding Min-N. In the second cut, the DM yield of FPMS-80 N was as high as the DM yield of Min-N 106 kg ha^{-1}. The DM yields of PPMS treatments tended to be greater than Min-N controls (370–700 kg ha^{-1}, 8–18 %). Although the differences appeared large enough to be practically significant, they were not statistically significant. In the second cut, the DM yields of LPMS and FPMS were at the same level as the corresponding Min-N control. The N yield of FPMS-40 N and LPMS-40 N was higher than Min-40 N control (+15 kg ha^{-1} and + 14 kg ha^{-1} respectively; Supplementary Table 4) in the first cut, but at the Min-80 N level and in the second cut, the corresponding differences were nonsignificant.

In 2022, two years after PPMS spreading, the total DM yield of the grass was an average of 6540 kg ha^{-1} year^{-1} for the Min-40 N level and 8230 kg ha^{-1} year^{-1} for the Min-80 N level (Supplementary Table 3). In the first cut, LPMS-80 N exceeded the yield response curve, while the yields in LPMS-40 N, FPMS-40 N, and FPMS-80 N corresponded to those attainable with Min-N fertilizer application levels of 51, 71, and 92 kg ha^{-1} respectively (Fig. 2). In the second cut, according to the yield response curve, the yield of LPMS-40 N, FPMS-40 N, LPMS-80 N, and FPMS-80 N corresponded to yields attainable with Min-N fertilizer application levels of 43, 27, 91, and 111 kg ha^{-1} respectively. The PPMS treatments tended to produce higher yields in both the first and second cuts compared to the Min-N controls, but the differences were not statistically significant.

3.2. Plant analysis

The use of PPMS did not affect the TW of barley in 2020, but both FPMS and LPMS decreased the TGW when the lower Min-N supplement level of 40 kg ha^{-1} was used (Table 2). The N content of barley grains

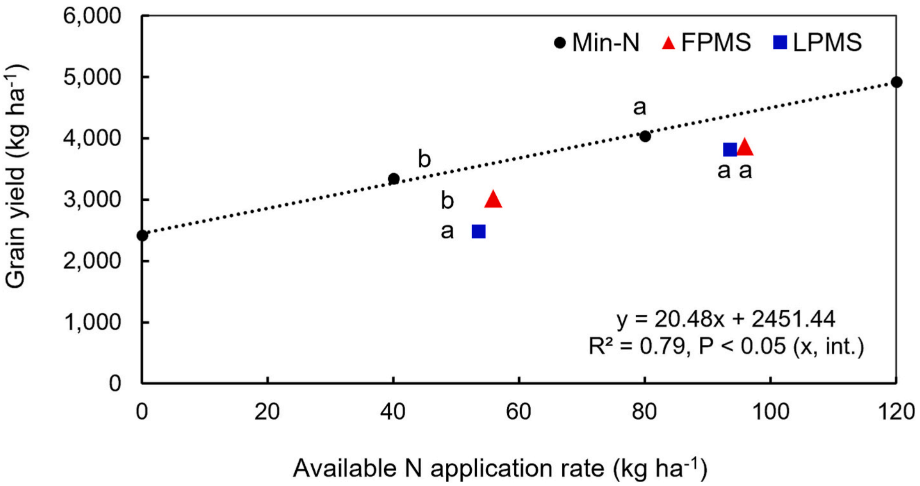

Fig. 1. The grain yield response curve (kg ha^{-1}, at 15 % moisture content) of increasing mineral nitrogen (Min-N) application rates of 0, 40, 80, and 120 kg N ha^{-1} and grain yields of LPMS and FPMS in cover crop barley in 2020. FMPS = fresh pulp and paper mill sludge, and LPMS = lime-stabilized pulp and paper mill sludge, which were applied as a single application in June 2020. Different letters indicate statistically significant differences ($P \leq 0.05$) separately at N-levels Min-40 N and Min-80 N. For Min-0 N and Min-120 N, the means of four replicates were used, not including the statistical model.

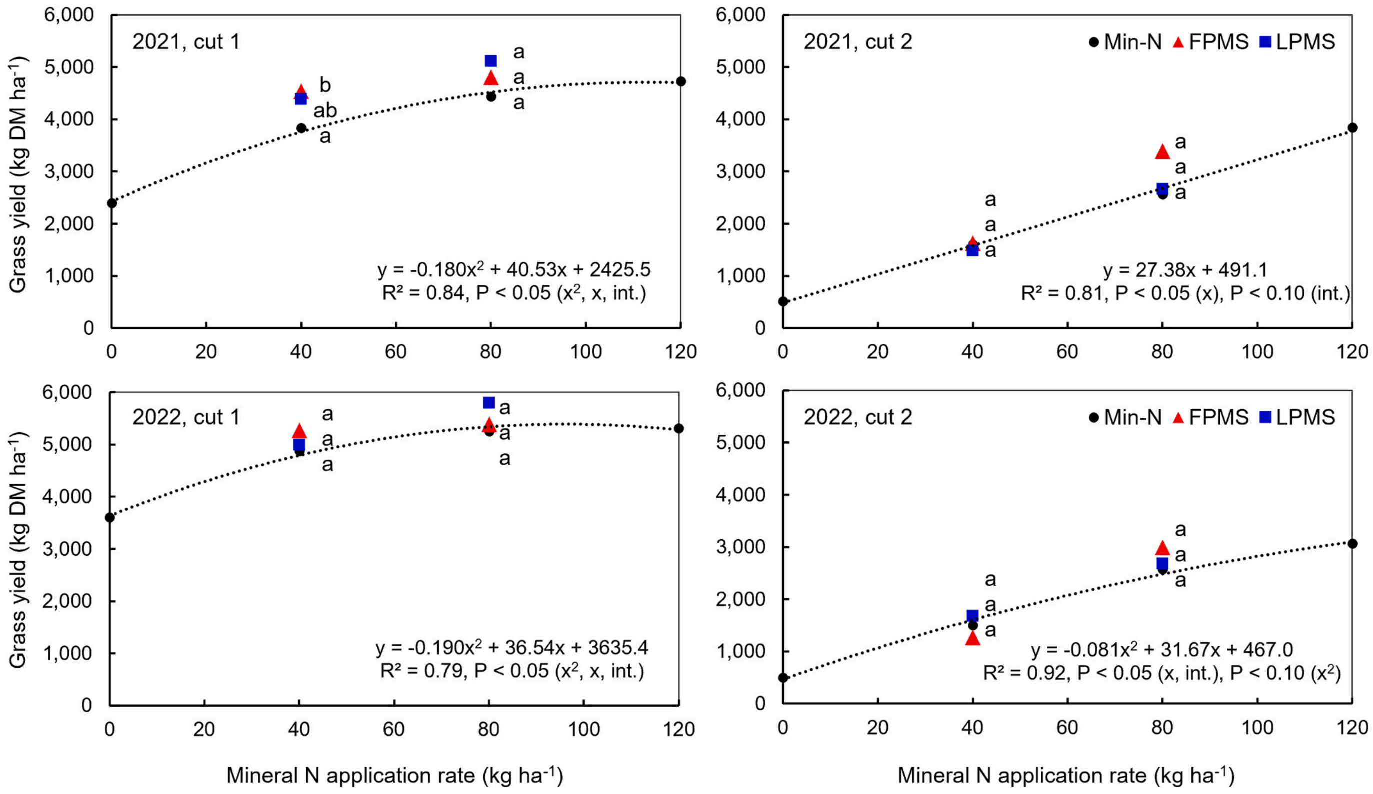

Fig. 2. The grass yield response curve (dry matter yield, kg DM ha^{-1}) of increasing mineral nitrogen (Min-N) application rates of 0, 40, 80, and 120 kg N ha^{-1} and DM yields of LPMS and FPMS in the first and second cut in 2021 (upper diagrams) and 2022 (lower diagrams). FMPS = fresh pulp and paper mill sludge, and LPMS = lime-stabilized pulp and paper mill sludge, which were applied as a single application in June 2020. Different letters indicate statistically significant differences ($P \leq 0.05$) separately at N-levels Min-40 N and Min-80 N. For Min-0 N and Min-120 N, the means of four replicates were used, not including the statistical model.

Table 2
Test weight (TW, kg hL^{-1}), thousand grain weight (TGW, g), nitrogen (N) uptake, and grain content of N, phosphorus (P), potassium (K), sulfur (S), calcium (Ca), and magnesium (Mg) for cover crop barley in 2020. Min-40 N and Min-80 N = application levels of mineral N fertilizer of 40 and 80 kg N ha^{-1}. FPMS = fresh pulp and paper mill sludge, and LPMS = lime-stabilized pulp and paper mill sludge, with an additional mineral N fertilizer level of 40 and 80 kg N ha^{-1}. Presented values are model estimated means with their standard errors (SE). Means ($n = 4$) marked with different letters indicate statistically significant differences ($P \leq 0.05$) within columns separately at N-levels Min-40 N and Min-80 N.

Treatment	TW	TGW	N uptake	N	P	K	S	Ca	Mg
	(kg hL^{-1})	(g)	(kg ha^{-1})	(g kg^{-1} DM)					
Min-40 N	60.6^{a}	38.5^{a}	44.1^{a}	15.4^{a}	4.58^{a}	4.88^{a}	1.33^{a}	0.45^{a}	1.33^{a}
FPMS-40 N	61.2^{a}	36.1^{b}	37.9^{ab}	14.7^{a}	4.58^{a}	4.75^{a}	1.33^{a}	0.45^{a}	1.33^{a}
LPMS-40 N	59.1^{a}	35.6^{b}	33.4^{b}	16.0^{a}	4.70^{a}	4.80^{a}	1.38^{a}	0.45^{a}	1.38^{a}
SE	0.89	0.76	4.65	0.37	0.12	0.09	0.04	0.03	0.05
Min-80 N	61.4^{a}	37.2^{a}	56.0^{a}	16.3^{a}	4.45^{a}	4.85^{a}	1.35^{a}	0.50^{a}	1.30^{a}
FPMS-80 N	61.7^{a}	35.0^{a}	49.8^{b}	15.1^{b}	4.45^{a}	4.85^{a}	1.38^{a}	0.45^{a}	1.25^{a}
LPMS-80 N	61.1^{a}	35.6^{a}	51.2^{ab}	15.8^{ab}	4.75^{a}	5.03^{a}	1.38^{a}	0.50^{a}	1.38^{a}
SE	0.48	1.12	4.14	0.48	0.08	0.07	0.03	0.02	0.05

was lower in FPMS-80 N than in Min-80 N control. The PPMS had no significant effects on other nutrient content in grain. The concentrations of Cd were analyzed from the grain samples of the Min-80 N control and FPMS-80 N and LPMS-80 N treatments, and all the measured concentrations were below the detection limits of <0.03 mg kg^{-1} DM (data not shown).

The use of PPMS did not affect or affected inconsistently the D value, NDF, sugars, or nutrient contents of grass yield (Supplementary Table 4 and 5). In 2021, LPMS-80 N increased the content of P, K, Ca, and Mg in the second cut, while FPMS-80 N increased the content of K and Mg. In general, the content of S was low compared with the average S content of grass (2.0 g kg^{-1} DM; Luke, 2023), with a tendency for a higher S content in response to the LPMS and FPMS amendments.

3.3. Soil chemical properties

As a response to LPMS amendments in the upper 20 cm soil layer, significant increases in Ca concentration and pH were observed (Table 3). In the LPMS-amended soils relative to the Min-N controls, the magnitude of the increase in the mean Ca concentration and pH was c. 240–660 mg l^{-1} and pH 0.5–0.7 units respectively over the three-year period. Due to both PPMS amendments, the mean S concentration of the plow layer increased by an average of 25 and 33 % at the 40 and 80 Min-N levels respectively, and a significant average increase of 58 % was also detected for the FPMS in the 20–40 cm layer. At the 20 cm depth, a significant increase of 29 % with FPMS-40 N was still observed in 2021. Compared with the Min-N controls, the PPMS treatments had no significant effects on the concentrations of P, K, and Mg at the end of the

Table 3

Mean value of soil pH (H_2O) and content of total carbon (C, g kg^{-1}) and nitrogen (N, g kg^{-1}) and acid ammonium acetate extractable phosphorus (P, mg L^{-1}), potassium (K, mg L^{-1}), sulfur (S, mg L^{-1}), calcium (Ca, mg L^{-1}), and magnesium (Mg, mg L^{-1}) at depths of 0–20 and 20–40 cm at the end of the 2020 growing season. Min-40 N and Min-80 N = application levels of mineral N fertilizer of 40 and 80 kg N ha^{-1}. FPMS = fresh pulp and paper mill sludge, and LPMS = lime-stabilized pulp and paper mill sludge, with an additional mineral N fertilizer level of 40 and 80 kg N ha^{-1}. Presented values are model estimated means with their standard errors (SE). Means (n = 4) marked with different letters indicate statistically significant differences ($p \leq 0.05$) within columns separately at N-levels Min-40 N and Min-80 N.

	pH (H_2O)		C		N		P		K		S		Ca		Mg	
			(g kg^{-1})		(g kg^{-1})		(mg L^{-1} soil)		(mg L^{-1} soil)		(mg L^{-1} soil)		(mg L^{-1} soil)		(mg L^{-1} soil)	
Treatment	Depth (cm)		Depth (cm)		Depth (cm)		Depth (cm)		Depth (cm)		Depth (cm)		Depth (cm)		Depth (cm)	
	0–20	20–40	0–20	20–40	0–20	20–40	0–20	20–40	0–20	20–40	0–20	20–40	0–20	20–40	0–20	20–40
Min-40 N	6.3^{a}	6.5^{a}	15.3^{a}	11.8^{a}	1.23^{a}	0.90^{a}	9.9^{a}	7.0^{a}	85^{a}	74^{a}	6.4^{a}	6.0^{a}	1128^{a}	1085^{a}	95^{a}	102^{a}
FPMS-40 N	6.4^{a}	6.5^{a}	17.7^{a}	9.4^{a}	1.38^{a}	0.73^{a}	9.9^{a}	7.0^{a}	79^{a}	79^{a}	8.1^{b}	9.0^{b}	1180^{a}	944^{a}	97^{a}	85^{ab}
LPMS-40 N	6.8^{b}	6.5^{a}	18.0^{a}	9.0^{a}	1.43^{a}	0.68^{a}	11.8^{a}	7.5^{a}	86^{a}	82^{a}	7.9^{b}	6.5^{a}	1504^{b}	898^{a}	94^{a}	77^{b}
SE	0.2	0.2	1.41	1.41	0.12	0.12	1.2	1.2	5.2	5.2	0.3	0.6	85	85	5.9	5.9
Min-80 N	6.4^{a}	6.7^{a}	16.3^{a}	8.7^{a}	1.25^{a}	0.63^{a}	9.2^{a}	5.2^{a}	78^{a}	73^{a}	6.5^{a}	6.3^{a}	1117^{a}	1021^{a}	94^{a}	99^{a}
FPMS-80 N	6.4^{a}	6.4^{a}	18.2^{b}	8.7^{a}	1.43^{b}	0.63^{a}	9.8^{a}	6.6^{a}	84^{a}	83^{a}	8.7^{b}	10.4^{b}	1114^{a}	922^{a}	91^{a}	84^{a}
LPMS-80 N	6.9^{b}	6.6^{a}	18.5^{b}	11.0^{a}	1.45^{b}	0.78^{a}	12.0^{a}	8.2^{a}	85^{a}	69^{a}	8.6^{b}	6.2^{a}	1778^{b}	1133^{a}	103^{a}	107^{a}
SE	0.2	0.2	1.40	1.40	0.12	0.12	1.3	1.3	5.9	5.9	0.7	0.7	88	88	7.9	7.9

first growing season (except lower Mg for the LPMS-40 N at the 20–40 cm depth) (Table 3) or significant residual effects at the 0–20 depth after two and three growing seasons in 2021–2022 (Supplementary Table 6). In the 0–20 cm soil layer, however, the use of LPMS tended to lead to an average increase of 25 % in soil P concentration in the first study year.

At the end of the first growing season after applications, PPMS increased the soil total C concentrations at the 0–20 cm depth compared with the Min-N controls by an average of 13 % at the Min-80 N level. Similarly, the measured total C tended to be higher in PPMS-amended soil at the Min-40 N level, but the difference was not statistically significant (Table 3). Further examination among the three selected treatments at the Min-80 N level revealed that the relative increase in the C concentrations occurred in the uppermost 10 cm layer, in which the PPMS were incorporated by harrowing, with a significant increase from 17.4 g kg^{-1} in the Min-80 N treatment to 19.5 g kg^{-1} in the PPMS treatments in 2020 (data not shown). In 2021–2022, FPMS-amended soils tended to have only slightly higher C content than non-amended soils, but no significant changes were observed except for FPMS-80 N at the 0–10 cm depth in 2022. As a response to the PPMS applications, trends in the soil total N concentrations followed a similar pattern to the C concentrations. Except for FPMS-80 N and LPMS-80 N at the 0–20 cm depth (Table 3) and LPMS-80 N at the 0–10 cm depth in 2020 (1.53 g kg^{-1} vs. 1.35 g kg^{-1} in the Min-80 N; data not shown), no other significant improvements in total N concentrations were detected.

For soil inorganic N concentrations in 2020, the concentrations of NH_4-N (0.90–1.64 mg kg^{-1}) and NO_3-N (0.33–0.56 mg kg^{-1}) exhibited an increasing tendency following the PPMS amendments, especially in the 0–20 cm layer (data not shown). However, the difference proved significant only to NO_3-N in LPMS-80 N at the 20–40 cm depth (0.64 mg kg^{-1} vs. 0.23 mg kg^{-1} in the Min-80 N). Overall, the sum of inorganic N (NH_4-N + NO_3-N) concentrations was generally small in all treatments, corresponding to an average of around 4 kg ha^{-1} in the plow layer in 2020, and about 85 % of the acquired soluble total N (9.1–13.8 mg kg^{-1}) occurred in organic form (SON; 7.7–11.7 mg kg^{-1}).

3.4. *Leachate samples*

Table 4 summarizes the results of the leaching experiment using undisturbed soil columns taken one growing season after the PPMS were applied. The overall level of total N leaching in both the Min-40 N and Min-80 N levels was 11.4 mg L^{-1}, of which 92 % was in the form of NO_3-N. When calculated to correspond to 150 mm drainage, the leached amount of total N over the Min-N levels was 17.3 kg ha^{-1}. There were no statistical differences in leachate parameters between treatments at the Min-40 N level except for drainage S concentration, which was more than 3.5 times higher in the FPMS-40 N treatment than in the Min-40 N and LPMS-40 N treatments.

At the Min-80 N level, the total N concentration was 143 % higher in the LPMS-80 N treatment than in the Min-80 N (control) treatment and 77 % higher than in the FPMS-80 N treatment. The drainage S concentration was more than 4.5 times higher in FPMS-80 N than in the other treatments, reflecting the high S input into the soil in the FPMS amendment. The drainage Mg concentration was statistically higher in both PPMS treatments than in the Min-80 N control, and the same tendency was observed in the Ca concentration, although this was not statistically significant. There were no differences in total P, dissolved P, TSS, or DOC concentrations.

Table 4

Mean concentration (mg L^{-1}) of leachate total nitrogen (TN), nitrate nitrogen (NO_3-N), calcium (Ca), magnesium (Mg), sulfur (S), total phosphorus (TP) dissolved phosphorus (DP), total suspended solids (TSS), and dissolved organic carbon (DOC) from soil monoliths. Min-40 N and Min-80 N = application levels of mineral N fertilizer of 40 and 80 kg N ha^{-1}. FPMS = fresh pulp and paper mill sludge, and LPMS = lime-stabilized pulp and paper mill sludge, with an additional mineral N fertilizer level of 40 and 80 kg N ha^{-1}. Means (n = 3) marked with different letters indicate statistically significant differences ($P \leq 0.05$) within columns separately at N-levels Min-40 N and Min-80 N.

Treatment	TN	NO_3-N	Ca	Mg	S	TP	DP	TSS	DOC
	(mg L^{-1})								
Min-40 N	11.5^{a}	10.6^{a}	21.7^{a}	4.89^{a}	2.90^{a}	0.07^{a}	0.02^{a}	0.016^{a}	8.5^{a}
FPMS-40 N	12.9^{a}	12.0^{a}	26.2^{a}	6.03^{a}	11.29^{b}	0.08^{a}	0.03^{a}	0.040^{a}	7.8^{a}
LPMS-40 N	13.3^{a}	12.2^{a}	20.9^{a}	4.79^{a}	3.42^{a}	0.07^{a}	0.02^{a}	0.003^{a}	8.7^{a}
SE	2.49	2.24	4.74	0.72	2.64	0.02	0.008	0.012	1.06
Min-80 N	11.3^{a}	10.4^{a}	20.8^{a}	4.46^{a}	2.81^{a}	0.08^{a}	0.02^{a}	0.025^{a}	8.2^{a}
FPMS-80 N	15.5^{a}	14.1^{a}	36.5^{a}	7.29^{b}	16.49^{b}	0.05^{a}	0.02^{a}	0.012^{a}	8.5^{a}
LPMS-80 N	27.5^{b}	25.0^{b}	34.2^{a}	8.49^{b}	4.33^{a}	0.08^{a}	0.04^{a}	0.013^{a}	9.2^{a}
SE	3.21	2.74	5.87	0.89	3.41	0.02	0.01	0.014	1.21

3.5. Soil hydraulic properties

The soil pore-size distribution presented in Fig. 3 revealed that the volume of the fine pores and wide coarse pores, with respective equivalent pore diameters of <0.2 μm and > 46 μm, were unaffected by the additions of PPMS compared with the Min-80 N control. However, the incorporation of PPMS significantly decreased the volume of the pores in the size range of fine coarse pores with an equivalent pore diameter of 46–9.1 μm, which was accompanied by an increase in the volume of medium-sized pores with an equivalent diameter of 9.1–0.2 μm, but a significant increase was only recorded for LMPS-80 N. Of the total porosity, an average of about 26, 15, 53, and 5 % was present in the pore size classes of wide coarse, fine coarse, medium-sized, and fine respectively.

The soil-water characteristic curves of the FPMS- and LPMS-amended soils appeared very similar (Fig. 4). Compared with the Min-80 N control, both PPMS decreased soil water retention by an average of 4.4 % in the macropore size range at matric potentials from −0.3 to −3.2 kPa. However, FPMS and LPMS produced 2.0–10 % and 3.5–15 % increases in volumetric water content respectively at a given matric potential between −10 and − 316 kPa, and the relative increment increased with decreasing matric potentials. A statistically significant increase in volumetric water content under the FPMS-80 N and LPMS-80 N treatments was obtained at matric potentials from −13 to −316 kPa, and exceptionally, only FPMS at −13 kPa did not differ from the Min-80 N control. Amending soils with PPMS increased their water retention relatively more at −316 kPa than at −10 kPa, leading to a statistically significant decrease by an average of 16 % in RPAW (0.166 cm^3 cm^{-3} in the Min-80 N; Supplementary Table 7). In terms of other common water retention parameters, the PPMS amendments had no effect on the total porosity (an average over treatments 0.517 cm^3 cm^{-3}) estimated in near-saturated conditions (at −0.1 kPa) and volumetric water content held at the FC (0.363 cm^3 cm^{-3}) and PWP (0.039 cm^3 cm^{-3}), and no significant change in DW (0.154 cm^3 cm^{-3}) and PAW (0.324 cm^3 cm^{-3}) was therefore observed.

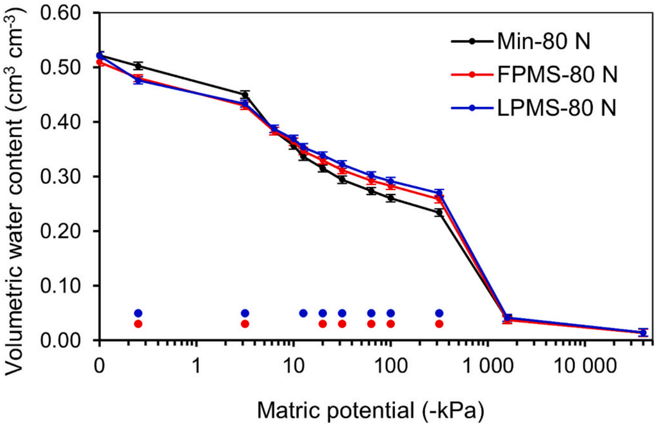

Fig. 4. Soil-water characteristic curves of the cultivated silt loam soil samples without (Min-80 N) and with two different pulp and paper mill sludge materials (FPMS-80 N and LPMS-80 N) in the soil 2–7 cm layer. The mean volumetric water contents (cm^3 cm^{-3}; n = 6) are presented at 13 matric potentials (-kPa). Min-80 N = application level of mineral nitrogen (N) fertilizer of 80 kg N ha^{-1} (non-amended control treatment), FPMS-80 N = fresh pulp and paper mill sludge, and LPMS-80 N = lime-stabilized pulp and paper mill sludge, with an additional mineral N fertilizer level of 80 kg N ha^{-1}. Error bars represent the standard error (SE). Red circle (FPMS-80 N) and blue circle (LPMS-80 N) symbols along the x-axis denote that the volumetric water content of the sludge-amended samples differed statistically significantly ($P < 0.05$) from the Min-80 N, i.e. non-amended samples, in the specific matric potential.

The mean values of soil K_{sat} tended to be greater in the FPMS- and LPMS-amended soils than in the Min-80 N control treatment. The K_{sat} values showed an ascending order: 0.196 cm min^{-1} (0.056, 0.689) for Min-80 N, < 0.271 cm min^{-1} (0.077, 0.952) for FPMS-80 N, < 0.441 cm min^{-1} (0.126, 1.548) for LPMS-80 N, with the respective mean and the lower and upper bound of 95 % confidence intervals in parentheses. However, the K_{sat} values did not differ statistically between treatments. In the same treatment, variations in the replicate samples were partly large within and between plots, generating substantially wide confidence intervals.

4. Discussion

The three-year field experiment showed mainly minor or nonsignificant effects of a single PPMS amendment on productivity, yield quality, and the risk of nutrient losses. The immediate effect of the PPMS

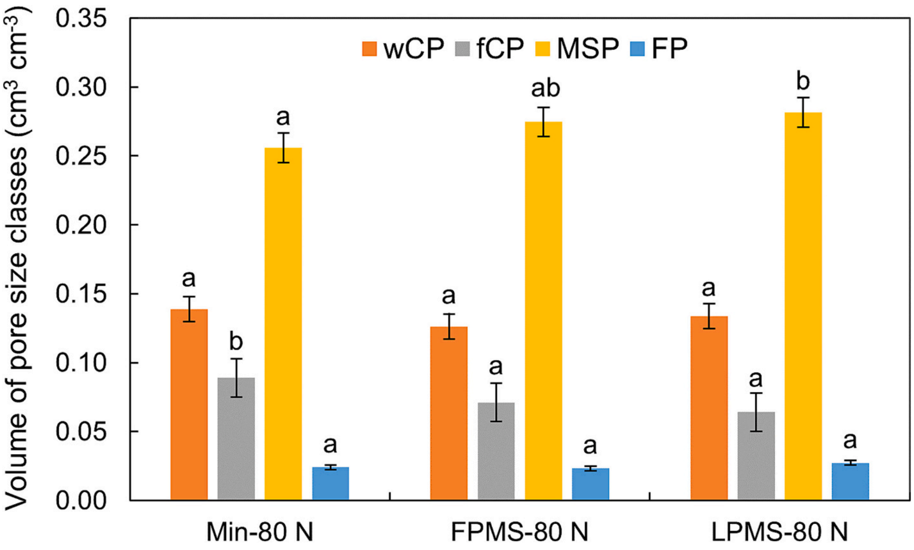

Fig. 3. Volume fractions of pore size classes (cm^3 cm^{-3}) for wide coarse pores (wCP), fine coarse pores (fCP), medium-sized pores (MSP), and fine pores (FP) of the cultivated silt loam soil samples without and with two different pulp and paper mill sludge types in the soil 2–7 cm layer. The respective equivalent pore diameters were about >46 μm, 46–9.1 μm, 9.1–0.2 μm, and < 0.2 μm. Min-80 N = application level of mineral nitrogen (N) fertilizer of 80 kg ha^{-1} (control treatment), FPMS-80 N = fresh pulp and paper mill sludge, and LPMS-80 N = lime-stabilized pulp and paper mill sludge, with an additional mineral N fertilizer level of 80 kg N ha^{-1}. Error bars denote standard error (SE). Means ($n = 6$) with the same letter are not significantly different at $P \leq 0.05$.

application on grain yield tended to be negative, probably due to transient N immobilization. However, the standard N application rate was adequate to avoid significant yield losses. During the two following years, the yield responses of grass ley to the PPMS treatments were rather positive because of the residual N fertilizer effect, the short-term increase in soil C, the liming effect of the LPMS, or additional nutrient inputs, namely Ca, P, and S. The PPMS treatments induced no marked changes in yield quality at any point of the study. In the soil pore system, the PPMS additions induced a small increase in the volume of medium-sized pores and a decrease in fine coarse pores, which caused a slight decrease in readily plant-available water. Soil saturated hydraulic conductivity tended to improve following the PPMS amendments, reducing the risk of surface runoff, while no gains were obtained in the nutrient losses via leachates.

4.1. Amendment effects on crop yields and yield quality

The PPMS amendments may contain a large amount of C relative to N (high C:N ratio), which can result in immobilization of N within the microbial biomass, decreasing the amount of plant-available N (Cao et al., 2020). Thus, the spreading of PPMS has also been observed to have negative effects on the growth of annual crops (Camberato et al., 2006; Rasa et al., 2021). This study demonstrated that applying Min-N fertilizer in an amount of 80 kg ha^{-1} with PPMS prevented the decrease in yield that could have been expected based on previous research (Räty et al. 2023), particularly as sowing was done without the recommended two to four weeks' delay after PPMS application (Simpson et al., 1983 in Camberato et al., 2006). In addition to mineral N fertilization, the lack of a noticeable effect on yield could be explained by the moderate C:N ratio of used PPMS compared with the ratios of 70:1–323:1 and 698:1 used in the studies of Camberato et al. (2006) and Rasa et al. (2021) respectively. Although the C:N ratios of FPMS and LPMS (27:1 and 24:1 respectively) only just exceeded the critical C:N ratio of 20–30, which may cause immobilization of N in soil (Stevenson and Cole, 1999), the decrease in barley grain yield and N uptake in the treatments with lower N fertilization (40 kg Min-N ha^{-1}) can be regarded as a result of PPMS-induced N immobilization. According to the Min-N yield response curve (Fig. 1), the applied N was not used by the plant in the LPMS-40 N treatment. The maximum N fertilization level of 80–100 kg N ha^{-1} is allowed for barley in mineral soils (VnA 235/2015, 2015), and according to our results, yield losses caused by PPMS at this N level can be prevented. However, in this study, N immobilization was still observed in a lower N content of grain and lower N uptake in FPMS-80 N, but not in LPMS-80 N. Depending on the type of PPMS, a slight decrease in yield quality is therefore still possible, although the yield level is maintained with a sufficient N fertilizer application.

In contrast with the initial short-term N immobilization, a positive residual effect of slow mineralization of the previously immobilized N and non-soluble N occurs as a response to PPMS amendments (Räty et al. 2023). In this study, the 2021 and 2022 grass yields were used to observe this residual effect. In the first cut of grass in 2021, the PPMS treatments tended to produce 8–18 % higher DM yields than the corresponding Min-N control plots, which can be explained by the residual effect. In the second cut of 2021 and in 2022, some tendency to yield responses (from −16 to +32 %, average + 6 %) in PPMS treatments was observed, but the phenomenon was not systematic. Clear residual effects have been observed, for example, in the study of Simard (2001), in which combined primary/secondary papermill sludge with moderate C:N ratios (28:1 to 42:1) was an effective source of N for horticultural crops and had significant residual N effects for the following crops. A tendency for a residual N fertilizer effect was also observed by Rasa et al. (2021; LPMS +500 kg ha^{-1} grain yield of oat, C:N 36:1, $P = 0.2$). Overall, small increases in N availability are challenging to detect in field conditions against the large background variations in weather, amendment composition, and inherent soil fertility (Webb et al., 2010).

In this experiment, where the average status of topsoil S was fair, PPMS materials with more abundant S supply did not affect the S content of the grain, which were above the average S content of barley grain (1.2 g kg^{-1}DM; Luke, 2023), indicating that barley did not suffer from S deficiency in the current study. In contrast, the S content of grass in both the first and second cuts in 2021 was low, with a nonsignificant increase in response to PPMS amendments. Hahtonen and Saarela (1995) found only a small, inconsistent, and statistically nonsignificant growth response of grass silage to supplementary S fertilization on six sites in central and northern Finland with a soil S concentration of 9–30 mg L^{-1}. Meanwhile, in an Irish study on a sandy loam soil, Aspel et al. (2022) reported a yield increase of perennial ryegrass, with a simultaneous increase in apparent fertilizer N recovery and decrease in nitrate leaching loss, in response to S addition in conjunction with N fertilization. Especially in S-deficient cultivated soils, PPMS materials may therefore act as a substantial source of S.

The application rate of FPMS exceeded the national regulatory limits for Cd, with an unexpectedly high Cd content, while the application rate for LPMS could have been adjusted slightly higher. The Cd concentrations measured here for the barley grain (< 0.03 mg kg^{-1} DM) were lower than the mean values of 0.03–0.09 mg kg^{-1} for the Finnish cereal grain reported by Louekari et al. (2000) and Soinne et al. (2022), lying within a range of 0.01–0.06 mg kg^{-1} for oat and wheat grain reported by Rasa et al. (2021) over the four-year study period, with no significant PPMS treatment effects.

4.2. Amendment effects on soil carbon

The C concentration in Finnish mineral topsoil is decreasing at an average rate of 0.38 % yr^{-1} (Heikkinen et al., 2013; Heikkinen et al., 2022), emphasizing the need to cherish the soil C reserves. A relatively high single application rate of PPMS-C (approx. 3100 kg C ha^{-1}), roughly corresponding to about 7 % of the C stock in the plow layer on a hectare basis, led to a measurable rise in topsoil C concentrations in the establishment year, but the relative differences in SOC concentrations between treatments diminished over the three-year study period. Similarly, four years after a single application of a large dose of PPMS-C (approx. 8400 kg C ha^{-1}), Rasa et al. (2021) recorded minor detectable effects on C concentration in a clay soil. Using the same PPMS as in the present field experiment, Räty et al. (2023) showed that nearly 30 and 40 % of the amount of C added to FPMS and LPMS materials respectively, was respired during a 60-day laboratory incubation. Also, Chantigny et al. (1999) and Fierro et al. (2000) reported about one third of de-inking sludge-C decomposed during an initial rapid decay phase, and the remaining material during a slow decay phase, associated with high lignin content. By contrast, a more constant improvement in topsoil C concentrations in different soil textures has been obtained especially as a result of annual or biennial PPMS applications (Zibilske et al., 2000; Foley and Cooperband, 2002; Price and Voroney, 2007). Consequently, repeated applications of PPMS are probably required for the maintenance of the beneficial direct and indirect effects of PPMS on crop yields and soil physico-chemical properties.

4.3. Amendment effects on soil chemical properties and nutrient leaching

As one of the most pronounced beneficial impacts on soil chemical properties, PPMS materials may have a substantial liming capability (Camberato et al., 2006; Muukkonen et al., 2009; Turner et al., 2022). In the present study, LPMS served as a liming agent, and the effect was also reflected as a slight increase in the content of Ca in the second cut of grass ley. Although pH in Finnish cultivated mineral soils averages around 6.0 (Keskinen et al., 2016), pH values (as measured in water) may range from very acidic to neutral (4.0–7.2) in the plow layer (Räty et al., 2021). For the relative availability of the most essential plant nutrients, the recommended soil pH value is around 6.0–6.5 (5.3–5.8 in peaty soils) in several countries (Goulding, 2016), many agricultural soils being below this favorable pH range. In the LPMS treated plots, the substantial but nonsignificant rise in the soil AAAc-extractable P, being also reflected in the plant P contents, was probably accounted for by a combination of the amount of applied P and pH-induced changes in the solubility of soil P. As the sorption tendency of phosphate onto soil components is known to decrease with increasing soil pH (Ryden and Syers, 1975), liming may result in an increase in the extractability and availability of applied P (Haynes, 1982; Holland et al., 2018). Also, the availability of P in organic forms may improve through the stimulation of microbial activity contributing to P mineralization (Haynes, 1982; Holland et al., 2018).

In the leaching test carried out after the first growing season, the percolation water N concentrations of all the treatments were significantly higher than the N concentrations reported in Rasa et al. (2021) from PPMS-amended clay soil with crop cultivation. However, the results were slightly lower but comparable to the N concentrations from pasture grass during the renovation year detected earlier near the location of the present PPMS field experiment (Saarijärvi et al., 2007). The study area also has a grass ley rotation background from previous years, followed by plowing and crop cultivation, which may increase the levels of leachate N concentration. It is also well known that fine-textured soils retain N more efficiently than coarse-textured soils (Gaines and Gaines, 1994). Interestingly, at the Min-80 N fertilization level, total N and NO_3-N concentrations were markedly higher in LPMS-80 N than in other treatments. This could be due to the different decomposition rate of LPMS compared to FPMS, which is supported by a significantly lower N uptake of the LPMS crop at the Min-40 N level than with FPMS and mineral N control. Possibly, at the Min-80 N level, mineral N was sufficient to cover the demand of the crop and the decomposition needs of LPMS, which was reflected in higher N leaching in the monolith experiment.

For the studied silt loam soil, the PPMS amendments did not prove to function as an agricultural water protection measure as in the earlier Finnish study on clay soil by Rasa et al. (2021), in which the reduction in soil particle mobilization and associated total P loss were attributed to improved soil structural stability with the addition of PPMS. In the study region, soils are generally considered to be less susceptible to erosion (Lilja et al., 2017), and a low mean erosion rate (115 ± 103 kg ha^{-1} yr^{-1}) was also recorded in a five-year monitoring of an agricultural subcatchment with short-term leys (Räty et al., 2020). In particular, a higher proportion of total P is typically transported in dissolved than in particulate form from grasslands (e.g., Heathwaite and Dils, 2000; Järvenranta et al., 2014). Furthermore, soil profiles are relatively rich in hydrous oxides of Al and Fe (Yli-Halla and Räty, 2024), restraining P losses in matrix flow through mineral soil profiles due to P retention by subsoil with a high P sorption capacity (e.g., Heathwaite and Dils, 2000; Järvenranta et al., 2014).

Both PPMS materials enriched S in the plow layer, and in the case of FPMS with a markedly higher S application rate, a part of S was already leached downward in the soil after the first growing season during the fall soil sampling. Following the application of sulfuric acid-treated cattle slurry, Keskinen et al. (2022) detected a similar S accumulation in the soil layers, and Loide et al. (2020) an increase of S leaching through repacked topsoil columns. Like nitrate, sulfate is known to be highly vulnerable to leaching, especially in coarse-textured soils. As expected, an intensified S leaching was also evident in the present study in response to the application of S-rich FPMS-80 N (an average of 42 mg l^{-1} as sulfate). For comparison, the mean sulfate concentration varies between 8.4 and 45 mg L^{-1} in runoff from small agricultural catchments (excl. Acid sulfate soils), with the national mean sulfate concentration of 15 mg L^{-1} (median 3.8 mg L^{-1}) for freshwater in Finland (Ekholm et al., 2020).

4.4. Amendment effects on soil hydraulic properties

The average saturated hydraulic conductivity obtained for silt loam topsoil in the current study fell within a range of 0.02–0.55 cm min^{-1} reported by Yost and Hartemink (2018) for loamy sand in the top 50 cm. Keskinen et al. (2019) reported near-saturated hydraulic conductivities of 0.004–0.12 cm min^{-1}, measured with a tension infiltrometer at a supply pressure head of −1 cm, for coarse-textured soils, representing similar surface soil types to the experimental area. The 6–14-fold higher hydraulic conductivity obtained in the present study was principally associated with the soil saturated state, in which all pores are supposed to be filled with water and conductive, and large pores participate in water transportation to a greater extent (Angulo-Jaramillo et al., 2016; Blume et al., 2016). In the study of Keskinen et al. (2019), pores with an equivalent diameter of >2.9 mm were excluded during the infiltration process.

The present trend in increased K_{sat} following PPMS applications may partly be accounted for by the improved structural stability during water entry into the soil and enhanced aggregation and macroaggregate formation (Nemati et al., 2000; Chow et al., 2003). Chow et al. (2003) used undisturbed samples taken one year after PPMS applications and found that the K_{sat} of a gravelly loam soil tended to increase from 0.14 to 0.41 cm min^{-1} with increasing rates of PPMS additions (20–160 Mg DM ha^{-1}). Nemati et al. (2000) measured field-saturated hydraulic conductivity (K_{fs}) with a constant head pressure infiltrometer one year after the first application of PPMS (8, 16, and 24 Mg DM ha^{-1}) and detected a trend toward increasing K_{fs} for loamy and silty clay soils with an increasing rate of PPMS applications. For the sandy loam, however, they found the reverse trend, which was associated with clogging of pore spaces by sludge materials and the reduction in aggregate stability due to the increased decomposition of OM. The collapse of aggregates may also block up macropores upon wetting in structurally unstable coarse-textured soils during measurements (Keskinen et al., 2019) and may explain part of the observed variation in the K_{sat} of the current study. As a dynamic soil property, relatively high spatial and temporal variation is generally characteristic of hydraulic conductivity (e.g., Zhou et al., 2008; Keskinen et al., 2019).

Concerning the impact of PPMS on the volume of large pores, an apparent discrepancy between data derived from the measured water retention values and K_{sat} measurement may be associated with the methods. There were few measuring points between −0.1 and − 3.2 kPa matric potentials, combined with uncertainty related to the draining of large pores by gravity during weighing. In the present study, an increased volume of pore space and hence water retention in the range of medium-sized pores resulting from PPMS applications was observed, representing pores which contribute to water storage in soil (Dexter, 1988). Excluding RPAW, the effects of PPMS amendments on the other common water retention parameters were revealed to be nonsignificant. As the water content at FC was not affected by PPMS treatments, an estimated net gain of PAW was not obtained. Along with the higher rates of repeatedly applied PPMS and peat materials (c. 22–78 Mg ha^{-1} DM), Foley and Cooperband (2002) obtained an improvement of 15–45 % in PAW following the second amendment to a loamy sand soil with an inherently low total C content (5 g kg^{-1}). Zibilske et al. (2000) tested different application schedules of PPMS at rates ranging from 0 to 225 fresh-Mg ha^{-1} over a five-year experiment and reported that PAW was significantly related to cumulative sludge-C added to a fine sandy loam soil. In recent laboratory experiments with the same PPMS to that used in the present field experiment, Räty et al. (2023) demonstrated the potential of PPMS to enhance the water retention properties of repacked coarse-textured soils. In that case, water retention at FC increased proportionately by 10–30 %, leading to a relative increase in PAW by 7.0–33 %, following PPMS applications at a rate of 10 and 20 vol-%. Generally, water retention tends to be greater in disturbed than in undisturbed samples, especially at lower suctions, attributed to the lower BD values of the repacked samples (Shaykewich, 1970; Tuli et al., 2005). Arguably, the treatment-induced changes in soil hydraulic properties may also be easily masked by inherent soil variability. In changing climate conditions, soil amendments inducing the modification of soil pores toward an increased volume of medium-sized pores and an increase in the K_{sat} may help alleviate water shortages during short-term droughts, reducing the probability of ponding after heavy rainfall, with the concomitant risk of surface runoff and P mobilization from the uppermost soil layers.

5. Conclusions

In this three-year field experiment, FPMS and LPMS amendments were included in part of rotational leys by applying them as a single dose prior to the sowing of grass ley under a barley cover crop in the spring. Although PPMS application occurred simultaneously with the sowing, a relatively large amount of PPMS could be spread without a loss in grain yield, in conjunction with adequate supplemental N to overcome an anticipated N immobilization. In the second and third year following the applications, the PPMS treatments tended to show positive residual effects on the total DM yield of grass ley, associated with sludge-N mineralization. In addition to N, nutrient-rich PPMS may act as a source of plant nutrients, showing a tendency to increase AAAc-extractable P, S, and Ca in the surface soil layer. LPMS had a liming capacity, and the increment in soil pH lasted over the three-year study period. Based on the leaching experiment, PPMS amendments did not prove to be an agricultural water protection measure in the less erosion-prone silt loam soil, but the leaching of P, TSS, or DOC through the soil profile did not increase due to PPMS amendment either, as occurred for S in response to S-rich FPMS application.

In the establishment year, PPMS increased the soil total C concentrations in the uppermost soil layer, but this was no longer detectable in subsequent years due to the decomposition of PPMS materials. Although no improvement was achieved in PAW, the amendments increased volumetric water content at matric potentials corresponding to the medium-sized pore range. The tendency for improvement in the soil's hydraulic conductivity was found following the PPMS treatment. The PPMS-induced modification in the pore size distribution indicated that PPMS materials might have potential to improve soil water storage capacity and drainage properties in coarse-textured soils. Further studies on coarse-textured soils in boreal conditions should focus on the long-term effects of repeated applications of different kinds of PPMS on crop yields and soil biological, chemical, and physical properties. In short-term ley rotations, more information is also needed about practices in which organic fertilizers such as cattle slurry are used during growing years, and PPMS are applied during grass establishment as an additional input of exogenous organic matter.

CRediT authorship contribution statement

Mari Räty: Conceptualization, Methodology, Investigation, Resources, Writing – original draft, Writing – review & editing, Visualization, Project administration. **Maarit Termonen:** Conceptualization, Methodology, Investigation, Resources, Writing – original draft, Writing – review & editing, Visualization. **Juha Hyvönen:** Formal analysis, Writing – review & editing, Visualization. **Jaana Uusi-Kämppä:** Conceptualization, Writing – original draft, Writing – review & editing. **Kirsi Järvenranta:** Conceptualization, Methodology, Investigation, Resources, Writing – review & editing. **Helena Soinne:** Conceptualization, Methodology, Writing – review & editing. **Johanna Nikama:** Conceptualization, Methodology, Investigation, Writing – review & editing. **Kimmo Rasa:** Conceptualization, Writing – review & editing. **Mikko Järvinen:** Funding acquisition, Resources, Writing – review & editing. **Riikka Keskinen:** Conceptualization, Methodology, Investigation, Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.geodrs.2024.e00894.

Data availability

Data will be made available on request.

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