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Title:	Improving coarse-textured mineral soils with pulp and paper mill sludges: Functional considerations at laboratory scale
Year:	2023
Version:	Published version
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Please cite the original version:

Räty, M., Termonen, M., Soinne, H., Nikama, J., Rasa, K., Järvinen, M., Lappalainen, R., Auvinen, H., & Keskinen, R. (2023). Improving coarse-textured mineral soils with pulp and paper mill sludges: Functional considerations at laboratory scale. Geoderma, 438, 116617. https://doi.org/10.1016/j.geoderma.2023.116617

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Geoderma



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Improving coarse-textured mineral soils with pulp and paper mill sludges: Functional considerations at laboratory scale

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ARTICLE INFO

Handling Editor: Cornelia Rumpel. Handling Editor: C. Rumpel.

Keywords: Fertility Nitrogen immobilisation Soil respiration Water retention

ABSTRACT

Building up the organic matter content of coarse-textured soils with organic amendments seeks to ameliorate the productivity of these soils, which is limited by plant available water and nutrient supply. Wood fibre-based sludges from the pulp and paper industry have potential for soil conditioning. In this study, the effects of three different pulp and paper mill sludges at application rates of 10 and 20 vol-% on water retention, respiration, and nitrogen (N) dynamics were examined in a series of laboratory studies using coarse field soils. Water retention curves comprising 13 matric potentials revealed that the amendments increased total soil porosity and volumetric water content at matric potentials corresponding to macro- and mesopores size range with pore diameters of $>30 \ \mu m$ and $30-0.2 \ \mu m$, respectively. Volumetric water content at field capacity increased by c. 10-30%, depending on the type (fresh, lime-stabilised and fibre sludge) and application rate of the amendment, with no marked change in the water content at the permanent wilting point. This was reflected as a mean increase of 1.9-3.3 mm in the plant available water content relative to the non-amended soils (17 mm), which corresponds to 19-33 m³ per hectare. At most, an increase of 5.5 mm (55 m³ ha⁻¹) in plant available water was achieved by the fibre sludge amendment at an application rate of 20 vol-%. During a 60-day laboratory incubation, c. 30-40% of the carbon (C) added to soil in the sludge materials was respired as carbon dioxide. Additional N accelerated decomposition without increasing total respired C. Decomposition of the amendments in the soil led to a net N immobilisation of roughly 5–10 mg min-N g⁻¹ added C, which occurred mainly during the first two weeks after soil incorporation. Overall, pulp and paper mill sludge amendments may serve to alleviate water shortages during drought in coarse-textured soils, but may generate a transient plant-microbe competition in N uptake.

1. Introduction

Coarse-textured soils, namely loamy sands and sandy loams dominated by the 0.05–2.0 mm textural fraction, are common in the northern parts of Europe (Ballabio et al., 2016). For example, in Finland nearly 30% of cropland lies on coarse-textured soils (Heikkinen et al., 2013). Agricultural productivity on these soils is often constrained by low water and nutrient holding capacities. According to Salter and Williams (1965b), the mean available water capacity of sandy soils may be only a quarter of that in medium-textured soils and less than half the capacity in clay soils. Räty et al. (2021) recorded an average cation exchange capacity (CEC_{pot}, pH 7.0) of 14 \pm 4.3 cmol(+) kg⁻¹ in coarse soils, while the corresponding value in clays was 25 \pm 5.6 cmol(+) kg⁻¹. Consequently, sandy soils tend to produce lower yields than fine-textured soils (e.g. Nyiraneza et al., 2012; Usowicz and Lipiec, 2017).

Soil organic matter (SOM) can ameliorate the negative properties of coarse soils (Lal, 2020). These soils tend to be inherently relatively low in SOM, as the low production level limits organic inputs, and the low amount of soil fines limits the SOM protection capacity of the soil matrix (Burke et al., 1989; Hassink, 1997; Gross and Harrison, 2019). The

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https://doi.org/10.1016/j.geoderma.2023.116617

Received 10 October 2022; Received in revised form 11 July 2023; Accepted 16 July 2023 Available online 19 July 2023

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application of external organic amendments, e.g. manures and various industrial wastes, provides a means to build up SOM content, increase plant available water storage and enhance nutrient supply (Diacono and Montemurro, 2010; Eden et al., 2017). In a recent *meta*-analysis, Minasny and McBratney (2018) showed the effect of an increase in soil organic carbon (SOC) on the amount of plant available water to be rather small, as a 1% mass increase in SOC increased the average soil available water capacity by 1.16 mm H₂O 100 mm soil⁻¹. However, the effect of SOM on available water capacity is higher in coarse- than in medium- or fine-textured soils, and the lower the initial SOM content of the soil, the more pronounced the effect is (Libohova et al., 2018; Minasny and McBratney, 2018). As an extreme example, Wesseling et al. (2009) found an addition of 10 vol-% of peat in coarse sand mixtures intended for golf greens increased the amount of total available water by c. 140–430%.

The effects of organic amendments on soil properties depend on the quality of the added material, meaning readily decomposable amendments tend to exhibit an intense but short-lived effect, whereas the impact of more resistant materials tends to remain lower but last longer (Eden et al., 2017). Sludges formed in the wastewater treatment processes of the pulp and paper industry reflect the composition of wood fibres and thus constitute a soil amendment material with potentially long-term effectiveness (Camberato et al., 2006). However, the characteristics of these sludges vary greatly, depending on the source and process conditions (e.g. Rasa et al., 2021). Sludges can be categorised into three main types, of which deinking sludges (low nutrient and OM content) are generated during the recycling of used paper. Primary sludges are generated by the initial clarification of pulp and paper mill effluent while secondary sludges are undergone biological wastewater treatment processes (Turner et al., 2022). Nutrient-rich secondary sludges can serve as slow-release sources of nitrogen (N) and other nutrients, whereas the application of nutrient-poor primary sludges can lead to immobilisation of N over extended periods (Camberato et al., 2006). The immobilisation may be biotic (N bound into microbial biomass) or abiotic (e.g. fixation or condensation), depending on the relative availabilities and forms of C and N (Cao et al., 2020). Consequently, to avoid yield losses, fibre sludge-induced transient N limitation needs to be accounted for in the timing of planting and the rate of supplemental N fertilisation.

In this study, the performance of three different pulp and paper mill sludge materials was investigated in coarse mineral soils in a laboratory study to enable the detection of minor effects difficult to discern in varied field conditions. The influences of sludge amendments on soil water retention were explored via water retention curves comprising 13 matric potentials. The short-term stability of the products was assessed by measuring soil carbon dioxide (CO₂) emission rates. In addition, changes in the soil readily available inorganic N pool were followed to evaluate the effects on N dynamics (immobilisation and/or mineralisation). We hypothesised that carbonaceous pulp and paper mill sludges 1) increase plant available water content and 2) induce transient N immobilisation in coarse mineral soils, and that 3) these effects are influenced by the composition of the amendment. The study builds knowledge about the potential to improve the fertility of coarse-textured

soils through wood fibre-based amendments.

2. Materials and methods

2.1. Experimental soils

This study was conducted with two coarse-textured soils collected from the plough layer (0–20 cm) of cultivated fields from eastern central Finland in the province of North Savo. One of the sites was located on the premises of Natural Resources Institute Finland (Luke) in Maaninka, Kuopio, and the other on a private farm in Siilinjärvi, later referred to as Soil 1 and Soil 2, respectively. Soil pH was about 6.0, and total C and N content within the range of 1.59-1.86% and 0.11-0.14% (Table 1), respectively. Soil 1 comprised 12.5% clay, 50.5% silt, and 37.0% sand, whereas the respective values were 10.5%, 31.0%, and 58.5% for Soil 2. Based on the USDA textural triangle, the soils were silt loam and sandy loam, respectively. The soils were classified as Dystric Arenosols according to the World Reference Base for Soil Resources (IUSS Working Group WRB, 2015). The soils were homogenised, passed through a 15 mm sieve and stored field-moist at +5 °C until a series of three controlled laboratory experiments was performed at the Natural Resources Institute Finland, located in Jokioinen in southwestern Finland.

2.2. Pulp and paper mill sludge materials

The experiments included three types of sludge materials originating from the pulp and paper mill industry. These were: 1) lime-stabilised sludge produced by Fortum Waste Solution Oy, Kuopio, Finland (LPMS); 2) fresh and untreated sludge provided by Stora Enso Oyj, Varkaus, Finland (FPMS); and 3) fibre sludge produced by Soilfood Oy, Tampere, Finland, (FS; described in more detail in Rasa et al. (2021) with an identical designation). For the sludge materials, moisture and ash content were analysed gravimetrically at 105 and 550 °C respectively (Table 2). Sludge pH and electrical conductivity (EC) were measured in a sludge-water suspension (1:5 v:v). Laboratory compacted bulk density (g cm⁻³) was determined according to EN 13040. The total content of C was determined by dry combustion (Dumas method, Leco TruMac® CN), and that of N by the Kjeldahl method (Foss, Kjeltec 8400). Water-soluble ammonium-N (NH₄-N) and nitrate-N (NO₃-N) were analysed from 1:60 water extracts with a continuous flow analyser (Skalar San++ System).

2.3. Measurements of soil water holding capacity

The effects of the addition of pulp and paper mill sludges on soil water retention characteristics on the two coarse-textured soils were studied using mixtures packed in metal cylinders with an average volume of 195 cm³ (height c. 4.8 cm, diameter c. 7.2 cm). The bottom part of the cylinder was closed with a thin cloth. Three application levels, 0, 10, and 20 vol-%, were used, and each was conducted in four replicates with both soils. To determine the appropriate soil:sludge ratios for the mixtures prepared by weighing, test cylinders were packed with each pure component in 5–12 replicates. The mass in the cylinder was

Table 1

Particle-size distribution, pH, total carbon (C) and total nitrogen (N) content and carbon-to-nitrogen ratio (C:N) of the experimental soils. All analyses were carried out with air-dried, ground, and sieved (2 mm sieve) soil samples.

	pH ^a	Total C ^b	Total N ^b	C/N	Particle size distribution (µm) ^c						
					< 2.0	2–6	6–20	20-60	60–200	200-600	> 600
		(%)	(%)		Proportion (%)						
Soil 1 Soil 2	5.93 5.99	1.86 1.59	0.14 0.11	13.3 14.5	12.5 10.5	11.0 4.0	13.0 6.5	26.5 20.5	27.0 53.5	7.5 4.0	2.5 1.0

^a Soil pH was measured in a soil-water suspension at a soil:solution ratio of 1:2.5 (v:v).

 $^{\rm b}\,$ Total C and N were determined by the (Dumas) dry combustion method (Leco TruMac® CN).

^c Particle-size distribution was determined by a pipette method as described by Elonen (1971).

Table 2

Selected properties of the pulp and paper mill sludge materials used in the study. The values are means of three replicates \pm standard deviation. Values presented without dispersion statistics have been determined from one bulked sample only. FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge, FS = fibre sludge, DW = dry weight.

	Unit	FPMS	LPMS	FS
Moisture	(%)	$53.8{\pm}0.3$	66.2±0.8	$66.1{\pm}0.3$
Ash	(%, DW)	$17.8{\pm}0.1$	32.5 ± 0.6	$20.1{\pm}0.2$
pH		$6.4{\pm}0.02$	$11.1 {\pm} 0.63$	$7.6{\pm}0.15$
EC	$(mS cm^{-1})$	$1,103{\pm}81$	$2,487{\pm}564$	91±3.4
Volume weight	(g cm ⁻³)	0.36	0.59	0.38
		± 0.004	± 0.002	± 0.003
Total C	$(g kg^{-1})$	432 ± 2	381 ± 7	$386{\pm}2$
	DW)			
Cellulose	$(g kg^{-1})$	471	230	674
	DW)			
Hemicellulose	(g kg ⁻¹	121	122	176
	DW)			
Lignin	$(g kg^{-1})$	284	227	62
	DW)			
Lipophilic extractives	$(g kg^{-1})$	17	64	20
	DW)			
Hydrophilic	$(g kg^{-1})$	29	1.1	12
extractives	DW)			
Total N	$(g kg^{-1})$	$13.4{\pm}0.4$	$14.3 {\pm} 0.2$	$0.4{\pm}0.1$
	DW)			
C:N ratio		$32.4{\pm}1.0$	$26.7 {\pm} 0.1$	926±135
NH4-N	$(g kg^{-1})$	$1.86 {\pm} 0.05$	$0.64{\pm}0.12$	nd
	DW)			
NO3-N	$(g kg^{-1})$	nd	nd	nd
-	DW)			
	-			

nd = not detected.

compacted at a pressure of 3432 Pa with simultaneous vibration produced by dropping a 2.2 kg weight five times from a height of 5 cm at a distance of 10 cm from the cylinder. Finally, the surface of the sample was finished to the upper level of the cylinder. Thereafter, each cylinder was weighed, and the bulk density (BD) was calculated by dividing the dry mass of the sample (the dry matter content of each component was determined gravimetrically by drying the samples in an oven at 105 $^\circ\mathrm{C}$ overnight) by the volume of the cylinder. According to this packing test, separate sample portions for each cylinder were prepared in plastic bags, in which the components were mixed thoroughly, and thereafter poured into the cylinders and packed as described above. The soil-sludge mixture which was surplus relative to the cylinder volume was collected and weighed, to enable estimating the increase in soil volume induced by the amendments. As a quality control, in-house reference soil samples (clay-% 7, silt-% 19, sand-% 74) were included in the determinations (1 reference sample per 6 samples) and they were processed in the same manner as the non-amended samples. Based on the area of the cylinder, the application rates of 10 and 20 vol-% would at field scale correspond to approximately 20-60 fresh-t ha⁻¹, which covers the typical range of incorporation rates used in field experiments and practical cultivation. The rates applied depend on e.g. the type and properties of the sludge materials, soil characteristics and prevailing regulations.

To determine the conventional soil water retention curve, i.e. the pF curve, matric potentials of -0.1, -0.3, -3.2, -6.3, -10, -1.3, -20, -32, -63, -100, -316, -1,585, and -39,811 kPa were applied. After each drying step, changes in the sample height and diameter were measured at six points per sample using a digital vernier calliper and at four points using a feeler gauge, respectively. At the starting point of -0.1 kPa, the samples were saturated with (standing) tap water from the bottom to the top in a box with plastic foam at the bottom by adjusting the water level to the midpoint of the cylinder and equilibrating for 14 days. Thereafter, the free water level in the box was lowered to a level corresponding to a -0.3 kPa matric potential, and the samples were allowed to equilibrate for 3 days. Soil water retention at matric potentials from -3.2 to -316

kPa was determined with an overpressure method using pressure plate extractors (Soilmoisture Equipment Corp., USA), with equilibration times ranging from 3 to 21 days. At the matric potentials of -1,585 and -39,811 kPa, soil water retention was determined with a relative humidity (i.e. vapour pressure) method, using saturated solutions of (NH₄)₂C₂O₄ and NaCl respectively. A 1.0 g air-dried and sieved (a 5-mm sieve) sample of soil or a soil-sludge mixture with four replicates was placed in the desiccator containing the appropriate saturated salt solution at the bottom. The samples were allowed to equilibrate for 21 days and after weighing, the samples were dried overnight in an oven at 105 °C.

Gravimetric water content was determined after each drying step by weighing the cylinder sample and subtracting the mass of the dry soil from the mass of the wet soil, and the mass of the dry soil was derived by drying the sample after the matric potential of -316 kPa in an oven at 105 °C for 3 days. The volumetric water content was obtained as the ratio of water volume to the wet soil volume at the given matric potential value. The water content at two of the most negative matric potential values was converted into volumetric water content using the BD measured at -316 kPa. The BD was calculated by dividing the mass of the oven-dried soil after -316 kPa by the volume of the sample at the given matric potential. The field capacity (FC; at -10 kPa), permanent wilting point (PWP; at -1,585 kPa), total porosity (TP), drainable water (DW), readily plant available water (RPAW) and plant available water (PAW) were derived from the measured water retention values. The TP was estimated as the volumetric water content at -0.1 kPa, i.e. at nearsaturated conditions. According to e.g. Turunen et al. (2021), the DW, RPAW, and PAW was calculated as the difference of the volumetric water content between -0.1 kPa and -10 kPa, between -10 kPa and -316 kPa, and between -10 kPa and -1,585 kPa. The pore size ranges were divided into the macro-, meso- and micropores with equivalent pore diameters of around $>30 \ \mu\text{m}$, $30-0.2 \ \mu\text{m}$, $<0.2 \ \mu\text{m}$, respectively, using the respective matric potential values of > -10 kPa, -10 to -1585 kPa and < -1585 kPa as the drainage boundary (e.g. Wallace et al., 2020).

Soil particle density (PD) was determined with two replicates using a stoppered bottle pycnometer method. Briefly, a 10 g air-dried and sieved (a 5 mm sieve) sample was weighed in the pycnometer, and 20 ml of deionised water was added. The air was removed from the suspension by boiling for 15 min. After cooling to room temperature, the pycnometer was filled with boiled and cooled deionised water and then weighed.

2.4. Measurements of soil respiration

Microbial respiration was measured from Soil 1 amended with the three pulp and paper mill sludges at a 10 vol-% application rate to assess the stability of the materials and their effects on soil microbial activity. To ensure no limitation of the decomposition process due to N deficiency, the FPMS and LPMS materials were also incubated with an additional 100 mg N kg⁻¹ of soil applied as an ammonium nitrate (NH₄NO₃) solution (Table 3). Because the FS had an extremely high C:N ratio, additional N levels of 200 and 400 mg N kg⁻¹ were used. All treatments were carried out in three replicates arranged in a completely randomised design in a constant temperature chamber at 20 °C. Soil moisture was kept between 40 and 60% from FC by adding deionised water according to weight loss.

The experiment was established by weighing 100 g (as dry mass) of the sieved soil (a 15 mm sieve) into 150 ml plastic containers. Pulp and paper mill sludge additions were weighed according to their volume weights (as in the setup of water holding capacity) and mixed thoroughly in the soil. Additional N was applied as NH_4NO_3 solution. Each container was then placed in a gastight 2 l glass vessel with an open bottle containing 10 ml of 3.5 M NaOH to trap the emitted CO₂. The CO₂ traps were renewed after 1, 3, 7, 11, 15, 31, and 63 days from the establishment of the experiment. The amount of entrapped CO₂ was measured via titration of the NaOH solutions with 0.1 M HCl and

Table 3

The pulp and paper mill sludge amendment treatments carried out in laboratory incubations. FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge and FS = fibre sludge, with an additional nitrogen (N) level of 0, 100, 200, and/or 400 mg N kg⁻¹ soil dry weight applied as ammonium nitrate (NH₄NO₃) solution.

Sludge type	Additional N (mg N kg ⁻¹ soil	Treatment abbreviation	Application rate (mg kg ⁻¹ soil dry weight)			
	dry weight)		Total C	Total N	Inorganic N (NH ₄ -N + NO ₃ -N)	
FPMS	0	FPMS-0	7381	228	32	
FPMS	100	FPMS-100	7381	328	132	
LPMS	0	LPMS-0	7818	293	13	
LPMS	100	LPMS-100	7818	393	113	
FS	200	FS-200	5680	206	200	
FS	400	FS-400	5680	406	400	

calculated as the difference between the mean HCl consumption of control traps placed in empty vessels and the traps from the treatment vessels. To discern C emitted due to the amendments, the corresponding background soil respiration measured from the non-amended soils was subtracted from the results.

2.5. Measurements of nitrogen consumption

To assess the consumption of N during decomposition, a set of containers constructed identically to those in the respiration experiment (Table 3) was incubated openly in a constant temperature chamber at 20 °C in a completely randomised design. Soil moisture was kept between 40 and 60% from FC as in the respiration study. Three pots from each treatment were removed and frozen for later analysis after 0, 4, 20, 48, and 60 days from establishing the experiment. Finally, inorganic N (NH₄-N and NO₃-N) was extracted from the entire soil content in each pot (100 g dry weight) with 250 ml of 1 M KCl, and the concentrations in the extracts were analysed with a Skalar San++ auto-analyser. The amounts of inherent NH₄-N and NO₃-N measured from the non-amended soils were subtracted from the results to discern the effects of the pulp and paper mill sludge treatments.

2.6. Statistical analyses

Statistical analyses were performed using ANOVA (the MIXED procedure of the SAS software 9.4.; SAS Institute Inc. Cary, NC, USA). The model for water retention characteristics was constructed using treatment, soil, and treatment \times soil interaction as fixed effects, while a replicate was used as a random effect. Some outliers were removed from the analysis when required. Mean daily CO₂ emission and respiration rates and mineral N immobilisation/mineralisation rates were calculated using a model in which the treatment was considered a fixed, and the replicate a random, effect. The measurement periods were analysed separately. A logarithmic transformation was used if the assumption of the equality of variance was not valid.

A 95% confidence interval, the standard deviation, or the standard error of the mean (SEM) are presented, depending on the situation. The SEM is not presented when the transformation was used. The Residual Maximum Likelihood (REML) estimation method and the Kenward-Roger approximation for degrees of freedom were used in all models. Pairwise comparisons of means were determined using Tukey–Kramer's test with a significance level of 0.05.

3. Results

3.1. Effects of pulp and paper mill sludges on soil water retention characteristics

Fig. 1 illustrates the relationship between volumetric water content and matric potential for the non-amended and sludge-amended coarsetextured soils. The soil water retention curves of the non-amended soils appeared to be rather similar despite the differences in particle-size distribution (Fig. 1). However, the differences in volumetric water content were mainly statistically significant (p < 0.05) over the whole range of the measured matric potentials. Except for the matric potentials from -3.2 to -6.3 kPa, Soil 1 retained more water than Soil 2 at a given matric potential by an average of $0.029 \text{ m}^3 \text{ m}^{-3}$ ($0.006-0.047 \text{ m}^3 \text{ m}^{-3}$). The water retention characteristics of both studied soils were affected by the amendments of different pulp and paper mill sludges over the measured range. The relative impacts of the amendments on volumetric water content substantially diminished at low values of matric potentials from about -1,600 to -40,000 kPa, representing the range of micropores (equivalent pore diameter $<0.2 \mu m$). Sludge applications tended to increase volumetric water content in both soils at matric potentials from -0.1 to -316 kPa, corresponding to macro- and mesopores size range with equivalent pore diameters of around $>30 \,\mu\text{m}$ and $30-0.2 \,\mu\text{m}$, respectively. However, the LPMS treatment at 20 vol-% gave no increase in volumetric water content between -3.2 kPa and -20 kPa in Soil 1. Similarly, between -63 kPa and -316 kPa, neither the FPMS (10 and 20 vol-%) nor the FS treatment at 20 vol-% increased soil water retention in Soil 2.

Initially, the non-amended Soil 1 retained 3 percentage points (7.8% as a relative change) more water at FC than the non-amended Soil 2 (Fig. 1, Table 4). In Soil 2, the volumetric water content at FC was significantly affected by all sludge amendments, increasing proportionately by 9.9–16% and 16–30% with an application rate of 10 and 20 vol-% respectively. Following the application of the FPMS-20%, FS-10%, and FS-20% in Soil 1, water retention at FC showed statistically significant improvements of 9.7, 12, and 16%, respectively in relation to the non-amended soil. In practice, the volumetric water content at the PWP was unaffected by sludge amendments. However, there was a significant negative effect of FS-10% and FS-20% on water retention in Soil 1 and a positive effect of LPMS-20% in Soil 2, attributed to the small variability between replicate samples.

Regarding the non-amended soils, changes in the diameter and height of repacked samples by shrinkage phenomenon between -0.1kPa and -316 kPa resulted in a total volume loss of 15% in Soil 1 and 13% in Soil 2 (data not shown). Amending Soil 1 with pulp and paper mill sludges intensified the total volume loss by an average of 25% relative to the non-amended soil, while amendments had a lesser and partly inconsistent effect on shrinkage behaviour in Soil 2. The most distinct changes in vertical shrinkage occurred above -3.2 kPa, whereas the changes in horizontal shrinkage were more evenly distributed, with decreasing matric potentials. There was a non-significant trend towards decreased PD following the sludge amendments, and a significant decrease was observed only for LPMS in both soils at an application rate of 20 vol-% (Table 4). Compared to the non-amended soils, all sludge amendments significantly decreased BD at a matric potential of -0.1kPa. The TP, defined as volumetric water content in near-saturation condition (-0.1 kPa), obtained for the non-amended Soil 1 was 6.8% higher than that of the non-amended Soil 2. The TP of the amended soils was always higher than that of the non-amended soils, and tended to increase with the sludge amendment, but significant increases were only recorded at the higher application rate (Table 4).

The DW, RPAW, and PAW of the non-amended soils made up 32, 27, and 60% of the TP in Soil 1, and the respective proportions were 33, 32, and 61% for Soil 2 (Table 4). Compared with the non-amended soils, the sludge applications had little detectable positive effect on DW, representing easily drainable macropores with equivalent diameters of c.



Fig. 1. Changes in soil-water characteristics of two cultivated coarse-textured soils as a result of amendments of three different pulp and paper mill sludge types at application rates of a) 10 and b) 20 vol-%. Mean volumetric water content ($m^3 m^{-3}$; n = 4) is presented as a function of matric potential (-kPa). Soil 1 (upper diagrams): 12.5% clay, 50.5% silt, and 37.0% sand. Soil 2 (lower diagrams): 10.5% clay, 31.0% silt, and 58.5% sand. FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge, and FS = fibre sludge. Error bars represent the standard error of the mean (SEM). The value of the SEM is the same for all treatments within matric potentials, and the bars are presented for the non-amended soils. Triangle symbols with the same colours along the x-axis denote that the volumetric water content of the sludge-amended soils differed statistically significantly (p < 0.05) from the non-amended soils in the specific matric potential.

Table 4

Mean particle density (PD; g cm⁻³), bulk density (BD; g cm⁻³) at -0.1 kPa matric potential, total porosity (TP; m³ m⁻³), volumetric water content at field capacity (FC; m³ m⁻³) and permanent wilting point (PWP; m³ m⁻³), drainable water (DW; m³ m⁻³), readily plant available water (RPAW; m³ m⁻³), and plant available water (PAW; m³ m⁻³). Soil 1 = 12.5% clay, 50.5% silt and 37.0% sand, Soil 2 = 10.5% clay, 31.0% silt and 58.5% sand. FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge and FS = fibre sludge, with application rates of 10 and 20 vol-%. SEM = standard error of the mean. Statistically significant differences between treatments within columns are denoted with different letters (p < 0.05).

Soil	Treatment	PD	BD	TP	FC	PWP	DW	RPAW	PAW
		(g cm ⁻³)		$(m^3 m^{-3})$					
1	Non-amended	2.71 ^a	1.14 ^a	0.59 ^a	0.40 ^a	0.05 ^a	0.19 ^a	0.16 ^{ab}	0.35 ^a
	FPMS-10%	2.71^{ab}	1.04^{bc}	0.61 ^{ab}	0.43^{ab}	0.05^{a}	0.18^{a}	0.16^{a}	0.38^{ab}
	FPMS-20%	2.66 ^{ab}	0.95 ^{de}	0.63 ^{bcd}	0.44 ^{bc}	0.05 ^a	0.19 ^a	0.16 ^{ab}	0.39 ^{bc}
	LPMS-10%	2.69 ^{ab}	1.01 ^c	$0.62^{\rm abc}$	0.43 ^{ab}	0.05 ^a	0.19 ^a	0.16 ^{ab}	0.38^{ab}
	LPMS-20%	2.65^{b}	0.92 ^e	0.65 ^d	0.40^{a}	0.05^{a}	0.25^{b}	0.13^{a}	0.35^{a}
	FS-10%	2.70^{ab}	1.06^{b}	0.62^{abcd}	$0.45^{\rm bc}$	0.04^{b}	0.17^{a}	$0.19^{\rm bc}$	0.41^{bc}
	FS-20%	2.68^{ab}	0.99 ^{cd}	0.65 ^{cd}	0.46 ^c	0.04^{b}	0.18^{a}	0.21^{c}	0.42 ^c
	Average	2.68	1.02	0.62	0.43	0.05	0.19	0.17	0.38
2	Non-amended	2.70^{a}	1.24 ^a	0.55^{a}	0.37 ^a	0.03^{ab}	0.18^{a}	0.18^{a}	0.34 ^a
	FPMS-10%	2.69 ^a	1.16^{b}	0.56 ^{ab}	0.41 ^b	0.04 ^a	0.15^{ab}	$0.22^{\rm b}$	0.37^{ab}
	FPMS-20%	2.65^{ab}	1.08 ^{cd}	0.59 ^b	0.43^{b}	0.04 ^a	0.16^{ab}	$0.24^{\rm b}$	0.39^{b}
	LPMS-10%	2.68^{ab}	1.15^{b}	0.58 ^{ab}	0.43^{b}	$0.03^{\rm b}$	0.15^{ab}	$0.22^{\rm b}$	0.40^{b}
	LPMS-20%	$2.62^{\rm b}$	1.06 ^d	0.60^{b}	0.44 ^b	0.04 ^c	0.13^{b}	0.22^{b}	0.40^{b}
	FS-10%	2.68^{ab}	1.15^{b}	0.58^{ab}	0.41^{b}	0.04^{ab}	0.17^{ab}	0.21^{ab}	0.37^{ab}
	FS-20%	2.67 ^{ab}	1.12^{bc}	0.64 ^c	0.49 ^c	0.04 ^a	0.15^{ab}	0.29 ^c	0.45 ^c
	Average	2.67	1.14	0.59	0.43	0.04	0.16	0.23	0.39
	SEM	0.011	0.009	0.007	0.007	0.001	0.010	0.007	0.007
P values	Treatment	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.037	< 0.001	< 0.001
	Soil	0.026	< 0.001	< 0.001	0.28	< 0.001	< 0.001	< 0.001	0.099
	$\text{Treatment} \times \text{Soil}$	0.94	0.046	0.038	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

 $>\!30~\mu\text{m},$ and a significant increase in DW was only seen for LPMS-20% in Soil 1, when the volumetric water content was not affected by the sludge amendment at -10 kPa. Except for the increase in RPAW by 29% achieved following the FS-20% amendment, no other significant

changes in RPAW, i.e. the amount of water held between -10 kPa and -316 kPa matric potentials, were noted for Soil 1. The beneficial effects of sludge amendments on RPAW were more pronounced in Soil 2, with a significant increase from 21 to 64%, and exceptionally, only FS-10% did

not differ from the non-amended soil. Amending experimental soils with different pulp and paper mill sludges increased their water retention relatively more at FC (-10 kPa) than at PWP (-1,585 kPa), tending to lead to a net gain of PAW, i.e. taken as the difference of the amount of water present between the upper (FC) and lower (PWP) limits of PAW. In relation to the average $0.35 \text{ m}^3 \text{ m}^{-3}$ of the non-amended soils, the sludge amendments increased PAW by 7.0 to 20% in Soil 1 (except for LPMS-20%) and by 10 to 33% in Soil 2. However, no significant differences were found in PAW between FPMS-10% and LPMS (10 and 20 vol-%) and the non-amended Soil 1, and FPMS-10% and FS-10% and the non-amended Soil 2. According to the pairwise statistical comparison between treatments, FS-20% led to the greatest increment in RPAW and PAW in both soils, mainly differing significantly from the FPMS and LPMS amendments.

3.2. Respiration patterns and apparent decomposition rates

The temporal pattern and total cumulative amount of CO₂ emissions from pulp and paper mill sludge-amended soils were affected by the type of material (Fig. 2, Fig. 3, Table 5). The FPMS and LPMS materials exhibited the highest mean daily respiration rates during the first week of incubation, whereas in the FS treatments, the CO₂ emissions peaked later, in the second week. In the first week of incubation, FPMS-0 and LPMS-0 amended soils respired 5.5 and 13% of their added C. The addition of N enhanced respiration, and after a week, FPMS-100 and LPMS-100 had lost 9.1 and 16% of the amount of added C respectively. The C respired from FS-200 and FS-400 during the first week was significantly less, corresponding to 2-3% of the added C. Though additional N increased mean daily respiration at the beginning of incubation for FPMS and LPMS, it had no influence on the total cumulative CO₂ emissions. Ultimately, the total amount of C emitted corresponded to nearly 30% of the C introduced within the FPMS and FS materials. In the LPMS, this value was significantly higher, nearly 40%. CO₂ production from the non-amended control soil remained constant throughout the incubation period (Fig. 2).

3.3. Consumption of soil N reserves

In all the pulp and paper mill sludge treatments, decomposition bound readily available inorganic N (negative net change in NH_4 -N + NO_3 -N) in soil (Fig. 4a, Table 5). The concentrations of NH_4 -N decreased throughout the study in all treatments (Fig. 4b). For NO_3 -N, the concentrations decreased during the first third of the incubation in all but the FS-400, but thereafter turned to a slight increase, except for the FS-400, again showing an opposite trend to the other treatments (Fig. 4c). In FPMS and LPMS, additional N significantly boosted decomposition during the first third of the study and doubled the mean daily immobilisation rates (Table 5). In the FS, this effect was not significant. During the latter period of the study, the biological activity slowed in all the treatments, and except for the FS-400, this was accompanied by a major decrease in N consumption. In FPMS-100, some mineralisation of N was observed during the last 40 days of the study. Overall, the total amount of N immobilised during the 60-day study period tended to be higher when a higher amount of N was available, even though a significant difference between the N levels was recorded only with FS.

4. Discussion

4.1. Pulp and paper mill sludge impacts on soil water holding capacity

One of the most pronounced positive effects of pulp and paper mill sludges on soil physical properties is the decline in the BD of the sludgeamended soils (Zibilske et al., 2000; Foley and Cooperband, 2002; Price and Voroney, 2007). Also in the present study, the BD of the repacked coarse-textured soils was systematically reduced with an increased amendment rate. This was attributable to the lower BD of sludge materials in relation to the initial BD of mineral soils and interactions between soil and the sludges creating new inter-particle pores (Wallace et al., 2020). In general, decreased BD following sludge amendments has been linked to increased TP and further to improved water retention properties (Foley and Cooperband, 2002). In the current study, the TP values of the studied non-amended soils were relatively high, but the results were in line with earlier published values by Turunen et al. (2021) for similarly packed coarse sand and fine clay loam soils. Despite the increasing trend in TP with the application rate, the response of amendments on water retention properties at the measured matric potentials partly differed between the sludge and soil type and the amendment rate. As for the DW, RPAW and PAW, the extent of the sludges' effectiveness was not significantly rate-related (except for FS in RPAW and PAW in Soil 2). Rasa et al. (2018) noted that biochar amendment modified soil moisture characteristics via direct (biochar internal pore system) and indirect (inc. soil structural changes, e.g. aggregate formation) mechanisms. Wallace et al. (2020) found that greater mesoporosity was produced with decreasing particle size of municipal compost. Also in the current study, the differences in the physical and chemical characteristics of sludges, including the differences in fiber length and/or particle size distribution, might have affected the soil-sludge interaction, and therefore the arrangement of particles and pore size distribution. Furthermore, the different responses to the amendments may be attributed to their decomposability and the



Fig 2. Cumulative CO₂-C (mg g⁻¹ soil) emissions during a 63-day incubation from non-amended soil and from soils amended with three different pulp and paper mill sludges with or without additional nitrogen (N). Error bars represent the standard deviations (n = 3). Non-amended and amended soil = Soil 1 (12.5% clay, 50.5% silt, and 37.0% sand), FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge, and FS = fibre sludge, with an additional N level of 0, 100, 200, and/or 400 mg N kg⁻¹ applied as ammonium nitrate (NH₄NO₃) solution.



Daily respiration and mineral N (NH₄-N + NO₃-N) immobilisation/mineralisation rates calculated per added carbon (C) during the early and latter periods of laboratory incubation of three pulp and paper mill sludge materials in soil at different nitrogen (N) application levels, and the proportions of added C respired and amounts of mineral N immobilised throughout the study period (60–63 days). Statistically significant differences between treatments within columns are denoted with different letters (p < 0.05). FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge, and FS = fibre sludge, with an additional N level of 0, 100, 200, and/or 400 mg N kg⁻¹ applied as ammonium nitrate (NH₄NO₃) solution.

	Mean respiration rate (mg $CO_2 g^{-1}$ added C/d)		Proportion of added C emitted as CO ₂ (%)	Mean net immobilis (-)/miner (+) rate (g ⁻¹ addee	Total change in soil min-N over the study (mg	
	Early period ¹	Latter period ¹		Early period ¹	Latter period ¹	min-N g ⁻¹ added C)
FPMS-	28 ^a	12 ^{ab}	27 ^a	-0.32^{a}	-0.02^{ab}	-7.1 ^{ab}
0						
FPMS-	42 ^b	7.1 ^c	27 ^a	-0.62^{b}	0.03 ^a	-11^{a}
100						
LPMS-	53 ^c	12^{ab}	37 ^b	-0.14^{c}	-0.02^{ab}	-3.7 ^b
0	,		,		,	
LPMS-	64 ^a	10 ^{ac}	39 ^b	-0.31^{a}	0.00 ^{ab}	-6.2 ^b
100	-6	L	_	_		_
FS-	21 ^{ci}	15"	28ª	-0.32^{a}	-0.10°	-11^{a}
200	o=af	b	2.03	o o=3	o. o.=C	246
FS-	25 ^{°°}	15"	30°	-0.37^{a}	-0.35°	-21
400						

 $^1\,$ Early period = 0–15 days for respiration and 0–20 days for immobilisation; latter period = 15–63 days for respiration and 20–60 days for immobilisation/mineralisation.

partial decomposition of sludge material during the relatively long determination period of the soil water retention curve (about 100 days), which was also demonstrated as respiration losses from added C in the present study.

In general, the soil water retention curves (i.e. pF curve) for the studied silt loam and sandy loam soils followed the course of the curves commonly described for medium- to coarse-textured soils. The amount

Fig 3. Mean daily CO₂ emission rates (mg ¹ added C/day) from soils (Soil 1) amended with three different pulp and paper mill sludges with or without additional nitrogen (N). The mean daily values are calculated from total emissions collected during 7 consecutive measuring periods, varying from 1 to 32 days in length. The total duration of the study was 63 days. The values are mean estimates (n = 3) with 95% confidence intervals. Statistically significant differences between treatments within measuring periods are denoted with different letters (p < 0.05). Soil 1 = 12.5% clay, 50.5% silt, and 37.0% sand. FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge, and FS = fibre sludge, with an additional N level of 0, 100, 200, and/or 400 mg N kg⁻¹ applied as ammonium nitrate (NH₄NO₃) solution.

of soil water retained at a given matric potential depends on pore size distribution and pore geometry and volume, and soil textural and structural (e.g. BD) properties are therefore the key factors determining soil water retention characteristics. Sandy soils retain less water than clayey soils at the same matric potential, and correspondingly, the binding strength increases with increasing clay content at the same water content. Compared to fine-textured soils, coarse-textured soils release more (relatively weakly bound) water in the less negative matric potential range up to about -6.3 kPa (<pF 1.8) and retain less water in the more negative range (>pF 4.2; -1,500 kPa) unavailable for water uptake by roots. Consequently, the curves for coarse-textured soils are typically characterised by an initially steep slope, which levels sharply close to the x-axis (Ehlers and Goss, 2003; Hillel, 2003; Blume et al., 2016). Amending the studied coarse-textured agricultural soils with different pulp and paper mill sludges tended to increase their water retention over the measured matric potentials, especially increasing the amount of water hold over a wide pore size range from macro- to mesopores. In some treatments, more water was also retained at the PWP, which may be explained by the minor variability between replicate measurements, as well as by the development of fine pores (Foley and Cooperband, 2002) and increases in a soil's surface area (Minasny and McBratney, 2018), along with additions of OM to coarse-textured soils. However, water retention improved significantly more at FC than at PWP, leading to a net gain of PAW, representing water which can be extracted by plant roots.

The amount of PAW for the studied packed soils was close to the mean value of $0.33 \text{ m}^3 \text{ m}^{-3}$ (range $0.18-0.55 \text{ m}^3 \text{ m}^{-3}$) reported for the 5–10 cm uppermost soil layer of afforested arable land sites in western Finland (Wall and Heiskanen, 2003). In the study by Wall and Heiskanen (2003), PAW increased significantly with an increasing OM content and decreasing BD, being higher in mull soils (OM 15–40%) than in mineral soils (OM <15%) in the soil profile to a depth of 40 cm. In their review of the soil survey database, Rawls et al. (2003) concluded that at low initial SOC content, an increase in SOM content resulted in an increase in water retention in sandy soils, whereas an opposite trend was seen in fine-textured soils. At high initial SOC values, all soil types showed an improvement in water retention, although the greatest benefit was attained in sandy and silty soils. However, for noncalcareous soils, Bagnall et al.'s (2022) pedotransfer functions showed that the absolute



Fig 4. Concentrations (mg kg⁻¹) of soil a) total mineral nitrogen (ammonium-N; NH₄-N + nitrate-N; NO₃-N), b) NH₄-N, and c) NO₃-N in non-amended soils and soils amended with three different pulp and paper mill sludges with or without additional N. The values are averages of three replicates with the standard deviations. Non-amended and amended soil = Soil 1 (12.5% clay, 50.5% silt, and 37.0% sand), FPMS = fresh pulp and paper mill sludge, LPMS = lime-stabilised pulp and paper mill sludge, and FS = fibre sludge, with an additional N level of 0, 100, 200, and/or 400 mg N kg⁻¹ applied as ammonium nitrate (NH₄NO₃) solution.

effect of increasing SOC on PAW was greatest for fine-textured soils. As a source of SOC, for comparison, the effects of biochar amendments on soil water retention properties have been contradictory in recent Finnish laboratory and field plot scale experiments, depending on biochar and soil properties. Only minor or no clear impacts were detected in studies carried out using wastewater sludge chars and packed fine- and coarsetextured mineral soil samples (Turunen et al., 2021) or using the undisturbed samples collected from the control and forest residue biochartreated plots on a clay soil field (Soinne et al., 2020), while Rasa et al. (2018) reported that willow biochar increased PAW by 17-32% in packed heavy clay soil samples in a laboratory study. With respect to paper mill sludge amendments, Foley and Cooperband (2002) investigated the effects of repeated annual applications of different paper mill sludges (at a rate of about 22-78 dry Mg ha⁻¹) on water retention properties in sandy soils, finding a 15-45% increase in PAW following the second application. Due to the relatively fast microbial

decomposition of pulp and paper mill sludges (Zibilske et al., 2000; Foley and Cooperband, 2002; Rasa et al., 2021; Heikkinen et al., 2021), as was confirmed by the present findings, annual or biennial reapplications may be required for the maintenance or increase of soil C and therefore to maintain the achieved favourable soil physical properties (Zibilske et al., 2000; Foley and Cooperband, 2002). Water retention characteristics obtained from undisturbed samples may substantially differ from that for disturbed samples, especially at low tensions (Salter and Williams, 1965a). In addition, the effects will likely change and diminish gradually along with the decomposition of sludge materials in the field conditions. However, the present results demonstrated that the sludge materials have potential to improve the water retention characteristics of the coarse-textured soils and consequently, emphasizing need for further research and plot-scale field experiments.

As a consequence of climate change, the tendency towards declining soil moisture conditions in most parts of Europe is expected. In northern Europe, the strongest decline in near-surface soil moisture content is projected to occur in the spring, whereas it is anticipated that drying is fairly modest in the summer (Ruosteenoja et al., 2018). Despite yield losses of spring cereals caused by early summer droughts, the high regional and interannual variation in growing conditions and the uncertainties of projections have slowed the transition of Finnish farmers from rainfed to irrigated agriculture (Peltonen-Sainio et al., 2021). In practice, the results derived from this empirical approach suggest that sludge-amended coarse-textured soils at application rates of 10 and 20 vol-% may retain an average of 1.9 and 3.3 mm more PAW (except for LPMS at a 20 vol-% rate in Soil 1), in relation to about 17 mm in the nonamended soils, equating to 19-33 m³, which is stored in the 5 cm soil layer (cylinder height) on a hectare basis. These were associated with an average increase of 0.6 and 1.3 percentage points in SOC relative to the non-amended soils, respectively. In Soil 2, the FS amendment at an application rate of 20 vol-% produced the greatest improvement in PAW of 5.5 mm, equating to 55 m^3 ha⁻¹. However, these estimates did not take into account the changes in soil volume due to the amendment-soil interaction (Wallace et al., 2020). In the present study, the increase in the amended soil volume averaged around 6 and 14% at the respective application rates of 10 and 20 vol-%, also leading to extended soil depth available for water retention. Based on the monthly average crop evapotranspiration (ET₀) values of around 60-100 mm in east-central Finland during the summer months 1981–2020 (Pirinen et al. 2022), the sludge-induced increases in PAW correspond to an average ET₀ occurring over 1-2 days. These improvements in PAW with an addition of $1\% (10 \text{ g kg}^{-1})$ SOC were greater than the average increases of 1.4 and 1.9 mm 100 mm⁻¹ reported by Minasny and McBratney (2018) for fineand coarse-textured soils respectively. More recently, the pedotransfer functions (for FC, the water content was measured at -33 kPa) developed by Bagnall et al. (2022) predicted larger average increases of 3.8 and 2.7 mm 100 mm⁻¹ in PAW for fine- and coarse-textured soils, respectively, as a result of a management-induced increase of 1% in SOC in noncalcareous soils. A sludge-induced improvement in PAW might alleviate water shortages and help secure adequate water for root uptake during short-term droughts in spring and summer in boreal conditions. In an irrigated crop production system, paper mill sludges may reduce irrigation requirements (Foley and Cooperband, 2002) and also provide flexibility to irrigation schedule.

4.2. Pulp and paper mill sludge decomposition and related N dynamics in soil

The C released in respiration during the 2-month incubation of sludge-amended soils suggested generally similar decomposition patterns for pulp and paper mill sludges as reported by Fierro et al. (2000) and Chantigny et al. (2000) for de-inking paper sludge. During the first week of incubation, the respiration rate was highest in soil amended with LPMS, in which the greatest amount of lipophilic and hydrophilic extractives was added to the soil (Table 2). Soluble and readily available C is mineralised at the first stage of added organic matter decomposition, whereas lignin, as the generally most recalcitrant component, is attacked at a later stage (Berg and Matzner, 1997). The results clearly showed that the initial relatively fast C mineralisation phase was followed by slower mineralisation.

An increase of labile organic matter, an easy source of energy and nutrients, stimulates the rapid growth of microorganisms (Fontaine et al., 2006) and results in the immobilisation of N within new cellular components (Cao et al., 2020). Additional N especially enhanced the fast mineralisation in LPMS and FPMS during the first two weeks, while N immobilisation also increased. Such an effect of N on decomposition and immobilisation is typical with easily degradable materials, whereas a significant amount of readily available N may inhibit enzyme production, hampering the mineralisation of complex organic molecules like lignin (Berg and Matzner, 1997; Carreiro et al., 2000; Fog, 1988). In a field trial on a clay-textured soil, fungal indicator species associated with decomposition of lignin and cellulose were more abundant in the plots that received pulp and paper mill sludges three years before, indicating that part of the added OM decomposed slowly (Rasa et al., 2021). Furthermore, the slower mineralisation of the more recalcitrant C pools is C-limited and thus does not favour N immobilisation (Cao et al., 2021). Consequently, in the FPMS- and LPMS-amended soils, the respiration and immobilisation rates decreased once the labile C was exhausted. Immobilised N can be released from decaying microbial biomass in the short term (Kumar and Goh, 2003), of which some indication was seen during the study's latter period. Overall, additional N accelerated the decomposition of FPMS and LPMS materials but had no net effect on the end result, which is a quite typical finding (Fog, 1988). Overall, the net N immobilisation amounted to roughly 5–10 mg min-N g⁻¹ added C and mainly occurred during the first two weeks after soil incorporation. To avoid plant-microbe competition in N uptake, supplemental N fertilisation or a sufficient safety period between sludge application and sowing can be recommended.

According to Chantigny et al. (2000), the decomposition of de-inking paper sludge is slower because of the shortage of other nutrients when added in large amounts, but when the de-inking paper sludge is halved, the decomposition rate is enhanced. In the current study, the very low N content of the FS (Table 2) was compensated for with high additional N to avoid N limitation in decomposition. However, the results demonstrated that the selected addition levels were significantly in excess, which, besides compositional differences, may explain the more even respiration rate pattern and somewhat higher total immobilisation in FS-400 than in the FPMS and LPMS treatments. As discussed earlier, excess N may depress microbial activity (e.g. Corre et al., 2007). As for immobilisation, abiotic processes favoured under N-enriched conditions (Cao et al., 2020) and microbial luxury uptake of N, meaning immobilisation without a metabolic requirement, may have contributed to the high immobilisation values in the FS-400 (Fog, 1988; Yevdokimov et al., 2005). In treatments that received additional N, decreases in soil inorganic N occurred mainly in the NH₄-N fraction, reflecting nitrification (i. e. oxidation of $\rm NH_4^+$ to $\rm NO_3^-)$ and possibly the preference of soil microbes for NH_4^+ over NO_3^- (Geisseler et al., 2010).

5. Conclusions

Amending coarse mineral soils with pulp and paper mill sludges decreased soil bulk density, increased total porosity, and improved soil water retention properties. The amendments increased soil volumetric water content mainly in the range of macro- and mesopores (-0.1 to -316 kPa). The increase at field capacity (-10 kPa) varied between 10 and 30% compared to non-amended soil, depending on the application rate and characteristics of the sludge and receiving soil, while the effect on the volumetric water content at permanent wilting point (-1585 kPa) was marginal. Consequently, the sludge applications increased plant available water content by 7–33% compared to non-amended soils.

In the decomposition of the sludges in soil, a fast and short C mineralisation phase was followed by slower mineralisation. The fast decomposition of labile organic matter was accompanied by net N immobilisation. During the 2-month study, the total C respired corresponded to 30–40% of the amount of C added to the sludges, while the net N immobilisation amounted to roughly 5–10 mg min-N g⁻¹ of added C. Additional N accelerated decomposition but had no effect on the total cumulative CO₂ emissions and significantly increased the net N immobilisation rate only at an extreme N input.

Pulp and paper mill sludge amendments improved the water retention capacity of coarse-textured soils and may be useful in overcoming water shortages during drought events. The short-term peak in net N immobilisation following sludge application needs to be considered to avoid adverse effects on plant growth due to N availability limitation.

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.

Data availability

Data will be made available on request.

Acknowledgements

This study was conducted in the "Biosphere North Savo: Utilisation of biomass and biorefining techniques for novel industrial products (grant number A75980)" project, which has received its main funding from the European Regional Development Fund (ERDF) through the Regional Council of Pohjois-Savo. The main funder, the municipal and private funders, and the corporate enterprises are acknowledged. The authors are grateful to Research Engineer Taina Koivuniemi for undertaking physical analysis, Laboratory Engineer Outi Haapala and the entire staff for undertaking chemical analysis at Luke's Jokioinen laboratory, and Senior Scientist Petri Kilpeläinen for enabling and organising the determination of lignocellulosic fractions at Luke's Otaniemi laboratory.

References

- Bagnall, D.K., Morgan, C.L.S., Cope, M., Bean, G.M., Cappellazzi, S., Greub, K., Liptzin, D., Norris, C.L., Rieke, E., Tracy, P., Aberle, E., Ashworth, A., Bañuelos Tavarez, O., Bary, A., Baumhardt, R.L., Borbón Gracia, A., Brainard, D., Brennan, J., Briones Reyes, D., Honeycutt, C.W., 2022. Carbon-sensitive pedotransfer functions for plant available water. Soil Sci. Soc. Am. J. 1-18 https://doi.org/10.1002
- Ballabio, C., Panagos, P., Monatanarella, L., 2016. Mapping topsoil physical properties at European scale using the LUCAS database. Geoderma 261, 110-123. https://doi. . 10.1016/i oderma.2015.07.006.
- Berg, B., Matzner, E., 1997. Effect of N deposition on decomposition of plant litter and soil organic matter in forest systems. Environ. Rev. 5, 1-25. https://doi.org/ 10.1139/a96-017
- Blume H.-P., Brümmer G.W., Fleige H., Horn R., Kandeler E., Kögel-Knabner I., Kretzschmar R., Stahr K., Wilke B.-M., 2016. Scheffer/Schachtschabel Soil Science. Springer - Verlag GmbH Berlin, Heidelberg. 618. 10.1007/978-3-642-30942-7.
- Burke, I.C., Yonker, C.M., Parton, W.J., Cole, C.V., Flach, K., Schimel, D.S., 1989. Texture, climate, and cultivation effects on soil organic matter content in U.S. grassland soils. Soil Sci. Soc. Am. J. 53, 800-805. https://doi.org/10.2136/ ssaj1989.03615995005300030029x.
- Camberato, J.J., Gagnon, B., Angers, D.A., Chantigny, M.H., Pan, W.L., 2006. Pulp and paper mill by-products as soil amendments and plant nutrient sources. Can. J. Soil Sci. 86, 641–653. https://doi.org/10.4141/S05-120.
- Cao, Y., Zhao, F., Zhang, Z., Zhu, T., Xiao, H., 2020. Biotic and abiotic nitrogen immobilization in soil incorporated with crop residue. Soil Tillage Res. 202, 104664 https://doi.org/10.1016/j.still.2020.104664.
- Cao, Y., He, Z., Zhu, T., Zhao, F., 2021. Organic-C quality as a key driver of microbial nitrogen immobilization in soil: A meta-analysis. Geoderma 383, 114784. https:// doi.org/10.1016/i.geoderma.2020.114784.
- Carreiro, M.M., Sinsabaugh, R.L., Repert, D.A., Parkhurst, D.F., 2000. Microbial enzyme shifts explain litter decay responses to simulated nitrogen deposition. Ecology 81, 2359-2365, https://doi.org/10.2307/177459
- Chantigny, M.H., Angers, D.A., Beauchamp, C.J., 2000. Decomposition of de-inking paper sludge in agricultural soils as characterized by carbohydrate analysis. Soil Biol. Biochem. 32, 1561-1570. https://doi.org/10.1016/S0038-0717(00)00069-9.
- Corre, M.D., Brumme, R., Veldkamp, E., Beese, F.O., 2007. Changes in nitrogen cycling and retention processes in soils under spruce forests along a nitrogen enrichment gradient in Germany. Glob. Chang. Biol. 13, 1509-1527. https://doi.org/10.1111/ i.1365-2486.2007.01371.x.
- Diacono, M., Montemurro, F., 2010. Long-term effects of organic amendments on soil fertility. A review. Agron. Sustain. Dev. 30, 401-422. https://doi.org/10.1051/agro/ 2009040
- Eden, M., Gerke, H.H., Houot, S., 2017. Organic waste recycling in agriculture and related effects on soil water retention and plant available water: a review. Agron. Sustain. Dev. 37, 11. https://doi.org/10.1007/s13593-017-0419-9.
- Ehlers, W., Goss, M., 2003. Water Dynamics in Plant Production. CABI Publishing. 273, p.

Elonen, P., 1971. Particle-size analysis of soil. Acta Agral. Fenn. 122, 1–122. Fierro, A., Angers, D.A., Beauchamp, C.J., 2000. Decomposition of paper de-inking sludge in a sandpit minesoil during its revegetation. Soil Biol. Biochem. 32, 143-150. https://doi.org/10.1016/S0038-0717(99)00123-6.

- Fog, K., 1988. The effect of added nitrogen on the rate of decomposition of organic matter. Biol. Rev. 63, 433-462. https://doi.org/10.1111/j.1469-185X.1988
- Foley, B.J., Cooperband, L.R., 2002. Paper mill residuals and compost effects on soil carbon and physical properties. J. Environ. Qual. 31, 2086-2095. https://doi.org/ 10.2134/jeq2002.2086
- Fontaine, S., Mariotti, A., Abbadie, L., 2006. The priming effect of organic matter: a question of microbial competition? Soil Biol. Biochem. 35, 837-843. https://doi. org/10.1016/S0038-0717(03)00123-8
- Geisseler, D., Horwath, W.R., Joergensen, R., Ludwig, B., 2010. Pathways of nitrogen utilization by soil microorganisms - A review. Soil Biol. Biochem. 42, 2058-2067. https://doi.org/10.1016/j.soilbio.2010.08.021.
- Gross, C.D., Harrison, R.B., 2019. The case for digging deeper: Soil organic carbon storage, dynamics, and controls in our changing world. Soil Syst. 3, 28. https://doi. 90/soilsystems3020028

Hassink, J., 1997. The capacity of soils to preserve organic C and N by their association with clay and silt particles. Plant Soil 191, 77-87. https://doi.org/10.1023/A: 1004213929699.

- Heikkinen, J., Ketoja, E., Nuutinen, V., Regina, K., 2013. Declining trend of carbon in Finnish cropland soils in 1974–2009. Glob. Chang. Biol. 19, 1456–1469. https://doi. org/10 1111/gch 12137
- Heikkinen, J., Ketoja, E., Seppänen, L., Luostarinen, S., Fritze, H., Pennanen, T., Peltoniemi, K., Velmala, S., Hanajik, P., Regina, K., 2021. Chemical composition controls the decomposition of organic amendments and influences the microbial community structure in agricultural soils. Carbon Manag. 12, 359-376. https://doi. org/10.1080/17583004.2021.1947386.
- Hillel D., 2003. Introduction to Environmental Soil Physics. Elsevier Science (USA). Academic Press. 494. 10.1016/B978-0-12-348655-4.X5000-X.
- IUSS Working Group WRB. 2015. World Reference Base for Soil Resources 2014, update 2015. International soil classification system for naming soils and creating legends for soil maps. World Soil Resources Reports No. 106. FAO: Rome. 192 p.
- Kumar, K., Goh, K.M., 2003. Nitrogen release from crop residues and organic amendments as affected by biochemical composition. Commun. Soil Sci. Plant Anal. 34, 2441-2460. https://doi.org/10.1081/CSS-120024778.
- Lal, R., 2020. Soil organic matter and water retention. Agron. J. 112, 3265-3277. https://doi.org/10.1002/agi2.20282
- Libohova, Z., Seybold, C., Wysocki, D., Wills, S., Schoeneberger, P., Williams, C., Lindbo, D., Stott, D., Owens, P.R., 2018. Reevaluating the effects of soil organic matter and other properties on available water-holding capacity using the National Cooperative Soil Survey Characterization Database. J. Soil Water Conserv. 73, 411-421. https://doi.org/10.2489/iswc.73.4.411.
- Minasny, B., McBratney, A.B., 2018. Limited effect of organic matter on soil available water capacity, Eur. J. Soil Sci. 69, 39–47, https://doi.org/10.1111/
- Nyiraneza, J., Cambouris, A.N., Ziadi, N., Tremblay, N., Nolin, M.C., 2012. Spring wheat yield and quality related to soil texture and nitrogen fertilization. Agron J. 104, 589-599. https://doi.org/10.2134/agronj2011.0342
- Peltonen-Sainio, P., Juvonen, J., Korhonen, N., Parkkila, P., Sorvali, J., Gregow, H., 2021. Climate change, precipitation shifts and early summer drought: An irrigation tipping point for Finnish farmers? Clim. Risk Manag. 33, 100334 https://doi.org/ 10 1016/i crm 2021 100334
- Pirinen, P., Lehtonen, I., Heikkinen, R.K., Aapala, K., Aalto, J., 2022. Daily gridded evapotranspiration data for Finland for 1981-2020. FMIs Climate Bulletin Research Letters 4, 35-37. https://doi.org/10.35614/ISSN-2341-6408-IK-2022-11-RL
- Price, G.W., Voroney, R.P., 2007. Papermill biosolids effect on soil physical and chemical properties. J. Environ. Qual. 36, 1704-1714. https://doi.org/10.2134/ 2007 0043
- Rasa, K., Heikkinen, J., Hannula, M., Arstila, K., Kulju, S., Hyväluoma, J., 2018. How and why does willow biochar increase a clay soil water retention capacity? Biomass Bioenergy 119, 346-353. https://doi.org/10.1016/j.biombioe.2018.10.004.
- Rasa, K., Pennanen, T., Peltoniemi, K., Velmala, S., Fritze, H., Kaseva, J., Joona, J., Uusitalo, R., 2021. Pulp and paper mill sludges decrease soil erodibility. J. Environ. Oual, 50, 172-184, https://doi.org/10.1002/jeg2.20170.
- Räty, M., Keskinen, R., Yli-Halla, M., Hyvönen, J., Soinne, H., 2021. Estimating cation exchange capacity and clay content from agricultural soil testing data. Agric. Food Sci. 30, 131-145. https://doi.org/10.23986/afsci.112
- Rawls, W.J., Pachepsky, Y.A., Ritchie, J.C., Sobecki, T.M., Bloodworth, H., 2003. Effect of soil organic carbon on soil water retention. Geoderma 116, 61-76. https://doi. org/10.1016/S0016-7061(03)00094-6.
- Ruosteenoja, K., Markkanen, T., Venäläinen, A., Räisänen, P., Peltola, H., 2018. Seasonal soil moisture and drought occurrence in Europe CMIP5 projections for the 21st century. Clim. Dyn. 50, 1177-1192. https://doi.org/10.1007/s00382-017-3671-4.
- Salter, P.J., Williams, J.B., 1965a. The influence of texture on the moisture characteristics of soils. I. A critical comparison of techniques for determining the available-water capacity and moisture characteristic curve of a soil. J. Soil Sci. 16, 1-15. https://doi.org/10.1111/j.1365-2389.1965.tb01416.x.
- Salter, P.J., Williams, J.B., 1965b. The influence of texture on the moisture characteristics of soils II. Available-water capacity and moisture release characteristics. J. Soil Sci. 16, 310-317. https://doi.org/10.1111/j.1365-2389.1965. tb01442.x
- Soinne, H., Keskinen, R., Heikkinen, J., Hyväluoma, J., Uusitalo, R., Peltoniemi, K., Velmala, S., Pennanen, T., Fritze, H., Kaseva, J., Hannula, M., Rasa, K., 2020. Are there environmental or agricultural benefits in using forest residue biochar in boreal agricultural clay soil? Sci. Total Environ. 731, 138955 https://doi.org/10.1016/j. itotenv.2020.138955

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- Turner, T., Wheeler, R., Oliver, I.W., 2022. Evaluating land application of pulp and paper mill sludge: A review. J. Environ. Manage. 317, 115439 https://doi.org/10.1016/j. jenvman.2022.115439.
- Turunen, M., Hyväluoma, J., Keskinen, R., Kaseva, J., Nikama, J., Reunamo, A., Rasa, K., 2021. Pore structure of wastewater sludge chars and their water retention impacts in different soils. Biosyst. Eng. 206, 6–18. https://doi.org/10.1016/j. biosystemseng.2021.03.010.
- Usowicz, B., Lipiec, J., 2017. Spatial variability of soil properties and cereal yield in a cultivated field on sandy soil. Soil Tillage Res. 174, 241–250. https://doi.org/ 10.1016/j.still.2017.07.015.
- Wall, A., Heiskanen, J., 2003. Water-retention characteristics and related physical properties of soil on afforested agricultural land in Finland. For. Ecol. Manag. 186, 21–32. https://doi.org/10.1016/S0378-1127(03)00239-1.
- Wallace, D., Almond, P., Carrick, S., Thomas, S., 2020. Targeting changes in soil porosity through modification of compost size and application rate. Soil Res. 58, 268–276. https://doi.org/10.1071/SR19170.
- Wesseling, J.G., Stoof, C.R., Ritsema, C.J., Oostindie, K., Dekker, L.W., 2009. The effect of soil texture and organic amendment on the hydrological behaviour of coarsetextured soils. Soil Use Manag. 25, 274–283. https://doi.org/10.1111/j.1475-2743.2009.00224.x.
- Yevdokimov, I.V., Saha, S., Blagodatsky, S.A., Kudeyarov, V.N., 2005. Nitrogen immobilization by soil microorganisms depending on nitrogen application rates. Eurasian Soil Sci. 38, 516–523.
- Zibilske, L.M., Clapham, W.M., Rourke, R.V., 2000. Multiple applications of paper mill sludge in an agricultural system: soil effects. J. Environ. Qual. 29, 1975–1981. https://doi.org/10.2134/jeq2000.00472425002900060034x.