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### Soil organic carbon and clay content as deciding factors for net nitrogen mineralization and cereal yields in boreal mineral soils

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### Abstract

To achieve appropriate yield levels, inherent nitrogen (N) supply and biological N fixation are often complemented by fertilization. To avoid economic losses and negative environmental impacts due to over-application of N fertilizer, estimation of the inherent N supply is critical. We aimed to identify the roles of soil texture and organic matter in N mineralization and yield levels attained in cereal cultivation with or without N fertilization in boreal mineral soils. First, the net N mineralization and soil respiration were measured by laboratory incubation with soil samples varying in clay and organic carbon (C) contents. Secondly, to estimate the inherent soil N supply under field conditions, both unfertilized and fertilized cereal yields were measured in fields on clay soils (clay 30-78%) and coarse-textured soils (clay 0-28%). In clay soils (C 2.5-9.0%), both the net N mineralization and the cereal yields (without and with fertilization) decreased with increasing clay/C ratio. Moreover, in soils with high clay/C ratio, the agronomic N use efficiency (additional yield per kg of fertilizer N) varied considerably, indicating the presence of growth limitations other than N. In coarse-textured soils, the yield increase attained by fertilization increased with increasing organic C. Our results indicate that for clay soils in a cool and humid climate, the higher the clay content, the more organic C is needed to produce reasonable yields and to ensure efficient use of added nutrients without high N losses to the environment. For coarse soils having a rather high mean organic C of 2.3%, the organic C appeared to improve agronomic N use efficiency. For farmers, simple indicators such as the clay/C ratio or the use of non-N-fertilized control plots may be useful for site-specific adjustment of the rates of N fertilization.

### Highlights

• We aimed to identify simple indicators of inherent soil N supply applicable at the farm level.

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- In clay soils, the net N mineralization was found to correlate negatively with the clay/C ratio.
- In coarse-textured soils, agronomic N use efficiency improved with increasing soil organic C.
- Clay soils with high clay/C ratio are at risk of low yield levels.

#### **KEYWORDS**

agricultural soil, clay, N mineralization, soil carbon mineralization, soil fertility, yield response

### **1** | INTRODUCTION

Synthetic nitrogen (N) fertilizers produced via the Haber-Bosch process from atmospheric N<sub>2</sub> have greatly contributed to the substantial increases in crop yields during the past decades (Sinclair & Rufty, 2012; Smil, 1999; Tilman, Cassman, Matson, Naylor, & Polasky, 2002). However, currently only half or less of the global fertilizer N input is converted into harvested crop products (Lassaletta, Billen, Grizzetti, Anglade, & Garnier, 2014; Tilman et al., 2002), indicating major waste of fossil energy and losses of reactive N to the environment (Fields, 2004; Gruber & Galloway, 2008; Mosier, 2002). This excess N creates many negative impacts, including eutrophication and acidification of terrestrial and aquatic systems, a decrease in biodiversity and accelerated global warming (e.g., Fields, 2004). Although synthetic N fertilization increases biomass production, and thus the potential input of crop residues into soil and accumulation of soil organic carbon (OC), it may also increase microbial oxidation of the residue and native soil OC, resulting in loss of soil organic matter (OM) and total soil N (Khan, Mulvaney, Ellsworth, & Boast, 2007; Mulvaney, Khan, & Ellsworth, 2009). According to Singh (2018), however, N fertilizer-induced acceleration of soil OC mineralization takes place only when fertilizer N is applied at excessive rates. whereas Ladha, Reddy, Padre, and van Kessel (2011) concluded that in agricultural soils, inorganic fertilizer N significantly reduces the rate at which soil OM declines. Thus, considering all these aspects and the cost of the fertilization, site-specific adjustment for an optimal rate of synthetic N input is essential for sustainable N management.

The bulk of soil N reserves is organic, and therefore, the cycles of N and C are tightly coupled (Castellano, Kaye, Lin, & Schmidt, 2012; Gruber & Galloway, 2008; Schulten & Schnitzer, 1998; Stevenson, 1982). Microbial decomposition of soil OM continuously releases ammonium ions, most of which are assimilated by microbial biomass (Jarvis, Stockdale, Shepherd, & Powlson, 1996). Net N mineralization, which is the difference between gross N mineralization and N immobilization in the microbial biomass, determines the amount of labile inorganic N available for plant uptake. In soils with inherently high N availability, the response to added fertilizer N is low and the fertilizer N use efficiency remains weak (Schindler & Knighton, 1999). Meta-analyses of Finnish N fertilization trials of spring cereals and perennial grasses showed that optimum fertilizer rates were clearly related to the yields achieved without added N fertilizer in a given soil, and these yields, in turn, depended on soil-OM content (Valkama et al., 2016; Valkama, Salo, Esala, & Turtola, 2013). To avoid unprofitable and unsustainable over-application of fertilizer N, the inherent N supply in soil should thus be considered in fertilizer recommendations. In field conditions, the difference between yield at optimum N application rate and yield with no fertilizer N added can serve as an indirect measure to determine fertilizer N need (Lory & Scharf, 2003).

The capacity of soil to supply N depends largely on the content and quality of its OM (Berendse, 1990; Matus & Rodríguez, 1994). Ros, Hanegraaf, Hoffland, and van Riemsdijk (2011a), for instance, found that total and extractable OM explained 78% of the variation of mineralizable N in soil. As arable soils in boreal regions tend to be rather rich in OC (Heikkinen, Ketoja, Nuutinen, & Regina, 2013), their indigenous soil N availability may be higher than in warmer climates. The rate of N mineralization is related to the activity of the microbial decomposer community (Bengtsson, Bengtson, & Månsson, 2003), as influenced by factors such as temperature and moisture (Guntiñas, Leirós, Trasar-Cepeda, & Gil-Sotres, 2012; Paul et al., 2003), cultivation practices (Jarvis et al., 1996; Silgram & Shepherd, 1999), and pH and soil chemical fertility (Ros, Temminghoff, & Hoffland, 2011b). In addition, mineralization rates tend to be lower in finetextured than in coarse-textured soils due to the ability of clay to protect OM against decay (Castellano et al., 2012; Hassink, 1997; van Veen, Ladd, & Amato, 1985). Instead of the amount of soil OC per se, the ratio of OC to clay or the fine textural fraction can be a more useful tool in the

prediction of grain yields (Quiroga, Funaro, Noellemeyer, & Peinemann, 2006; Schjønning et al., 2018).

By the law of diminishing returns, the agronomic N use efficiency should be higher in soils that have low inherent N supply than in soils with properties indicating high inherent N supply. Because of the stabilizing effect of clay on soil OC, we hypothesize that to reach similar N supply originating from OM mineralization, soils with high clay content should have higher OC content than soils with lower clay content. With this background, the aim of this study was to build knowledge for developing science-based practical tools for farmers to adjust N fertilization for more sustainable N management. The specific objectives were (a) to identify the roles of soil OC and clay content in the net N mineralization and inherent N supply and (b) to assess the effects of varying soil texture and OC contents on cereal yields in boreal mineral soils.

### 2 | MATERIAL AND METHODS

### 2.1 | Soils and climatic conditions

Eighteen fields from two research stations (Jokioinen, south-western Finland, and Maaninka, east-central Finland) of the Natural Resources Institute Finland (Luke) and 16 fields of private farmers located similarly in southwestern Finland and in east-central Finland (Table 1, Figure 1) were selected for this study. All fields were in soil regions with a predominantly boreal continental to temperate continental climate (BGR 2005). The annual precipitation in southern and central Finland varies typically between 600 and 700 mm and mean yearly temperature is c. 5°C in southern Finland and declines towards the north. The formation of soils in Finland has been influenced by the last Weichsel glacial period, ending about 11,500 years ago, and by evolutionary stages of the Baltic Sea. Owing to the young pedogenetic age, most of the soils are relatively weakly developed. Cultivated clay soils of Finland are typically classified as (Vertic Luvic) Stagnosols or as (Luvic) Gleysols in wet depressions. Silt soils are classified as (Stagnic) Regosols, very fine sand or fine-sandy moraine soils are commonly classified as (Endogleyic) Cambisols and podzolized coarse mineral soils as (Glevic) Podzols (Lilja et al., 2017). The majority of the fields are artificially drained due to the excessive moisture (Puustinen, Merilä, Palko, & Seuna, 1994).

Only fields growing spring cereals in the study years 2016 and 2017 were chosen from the selected locations. Farmers were advised to leave an unfertilized area of at least one seed drill width and of 20 m in length at a representative site at an appropriate distance from the field edge. The observed part within each field (approximately

 $20 \text{ m} \times 20 \text{ m}$ ) thus included adjacent fertilized and unfertilized areas that were otherwise similar with regard to soil type and productivity according to the farmer's judgement.

### 2.2 | Incubation study for soil respiration and potential net nitrogen mineralization

To determine the potential net N mineralization, samples from six fields (included also in the field study, Table 1) were taken in autumn 2016 after the harvest of a spring cereal crop. The fields were selected based on records of their yields over the past 5 years and their clay contents to include both fields of high and low productivity and variation in texture. Each field had a sampling location placed on both fertilized and unfertilized areas of the field. On three of the six fields, an additional third sampling location was included to account for unexpected visually observed variability in the crop productivity. The final set of soil samples used for respiration and N mineralization studies in the laboratory thus represented 15 locations (Table 2). At each sampling location, two to four replicate soil samples were taken c. 5 m apart with an auger from the 0-20-cm soil layer. In the laboratory, the samples were passed through a 5-mm sieve and then stored field moist at +5°C for 2 weeks prior to establishing the incubation experiment. The sieved samples were analysed for organic carbon (C%) and total nitrogen (N%), as well as for pH (1:2 H<sub>2</sub>O) and electrical conductivity (EC) (1:2.5 H<sub>2</sub>O) (Table 2). Soil texture was measured from one sample per location.

For the incubation, 10 g (dry matter, DM) of each soil was weighed in a 120-ml infusion bottle, after which the soil moisture was adjusted to 60% of the water-holding capacity (WHC) of the particular soil. The WHC was measured separately for each of the sampling locations with a funnel method (20 g soil (DM) weighed into a funnel lined with filter paper, saturated with water for 1 hr and left to drain freely for 24 hr). Each replicate soil sample was used to fill four infusion bottles. Two of the bottles were assigned for N measurements carried out on days 0 and 14, the third one was frozen and reserved for determination of water-extractable organic carbon (WEOC) on day 0 and the last one used for measuring respiration throughout the study period and N at the end of the incubation (day 30). The infusion bottles were covered loosely with aluminium foil and incubated at 20°C in the dark for a maximum of 30 days. At the beginning of the incubation and on days 14 and 30, a set of sample bottles was frozen for later extraction of inorganic and total dissolved N.

		Variable 10–30%		Variable 10-30%	Variable 10–30%			Variable >30%	Variable 10–30%			Variable >30%			Variable 10–30%				74		Variable >30%	Variable 10–30%	Variable >30%		Variable 10–30%	Variable >30%	Variable 10–30%	Variable >30%	Variable >30%	Variable 10 2000
5 years	Crons <sup>b</sup>	Variab	Cereals	Variab	Variab	Cereals	Cereals	Variab	Variab	Cereals	Cereals	Variab	Cereals	Cereals	Variab	Cereals	Cereals	Cereals	Cereals	Cereals	Variab	Variab	Variab	Cereals	Variab	Variab	Variab	Variab	Variab	Variah
Management, 5 years	Oroanic fert <sup>a</sup>	<b>B</b>	++++	+++	++++	I	Ι	Ι	++++	++++	I	I	I	I	++++	I	++++	+++++	I	I	Ι	I	I	++++	I	+	+	Ι	+	-
g ha <sup>-1</sup>	N 2017	66	107	102	100	100	100	120	94	107	114	180	108	101	100	101	110	110	86	86	83	120	108	78	120	84	110	81	108	100
N fert. kg $ha^{-1}$	N 2016	92			100				93				81		66						107		81	109		110	82	87	81	50
стр	mg l <sup>-1</sup> snil	14	17	5	7	5	9	27	15	12	9	26	10	6	11	7	4	5	13	5	13	4	7	5	1	9	4	10	8	
nu.	рпн20	6.4	5.8	5.9	6.2	5.1	5.8	6.3	6.8	6.6	5.7	7.0	5.7	5.7	6.5	6.3	5.9	6.1	6.8	5.5	6.1	6.7	6.4	6.3	6.7	6.7	6.0	5.1	6.3	
UEU	ko⁻¹		4	4	6	2	4	8	12	7	7	12	6	11	12	16	13	13	12	13	20	19	24	20	22	23	24	17	26	
المولي		0	3	3	2	2	4	5	5	12	7	10	6	6	11	8	5	7	15	5	13	19	6	17	17	20	17	6	23	0
NJ		13	16	14	14	13	13	14	14	12	14	14	14	14	14	15	16	15	14	16	12	12	14	13	11	12	13	14	12	ç
Z	2	0.2	0.1	0.1	0.2	0.3	0.2	0.1	0.1	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.4	0.3	0.2	0.6	0.3	0.2	0.4	0.2	0.3	0.2	0.3	0.6	0.3	0
ت	د	2.1	1.8	1.9	2.2	3.4	2.4	1.8	2.0	1.3	2.7	2.1	2.4	2.7	2.4	3.7	6.2	4.8	2.5	9.0	3.6	2.6	5.4	3.1	3.2	2.8	4.1	8.0	3.1	1 7
Clav	LIAY	0	5	ŝ	S	9	6	10	10	15	19	21	22	24	25	28	30	32	37	42	47	48	50	51	54	54	68	69	71	5
Silt	IIIC	13	8	~	31	11	17	17	14	21	30	18	10	37	31	43	36	37	26	45	22	34	37	12	36	11	13	18	18	0
buo	Sanu %	87	87	87	64	83	74	73	76	64	51	61	68	39	4	29	34	31	37	13	31	18	13	37	10	35	19	13	11	10
	2017	Barley	Wheat	Oats	Barley	Oats	Oats	Oats	Barley	Wheat	Wheat	Wheat	Wheat	Barley	Barley	Barley	Barley	Barley	Barley	Barley	Oats	Barley	Wheat	Oats	Barley	Wheat	Oats/wheat	Oats	Wheat	Whent
Crop	2016	Barley			Barley				Barley						Barley						Wheat					Barley		Oats	Barley	F
	Field	1	2	ю	4	ŝ	9	7	8	6	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	00

Properties of the field soils in ascending order of clay% (silt, particle size 0.002-0.02 mm; CEC, cation exchange capacity; STP, soil test phosphorus), the applied N fertilization **TABLE 1** 

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	Crop		Sand	Silt	Sand Silt Clav C	U	z	C/N	N C/N Clav/C CEC	CEC	DHu30 STP	STP	N fert. k	N fert. kg ha <sup>-1</sup>	Management, 5 years	years
Field	2016	2017	%							cmol kg <sup>-1</sup>	1	mg l <sup>-1</sup> soil N 2016 N 2017	N 2016	N 2017	Organic fert. <sup>a</sup> Crops <sup>b</sup>	Crops <sup>b</sup>
30	Barley	Barley Barley	16	13	13 71	3.5	0.3	0.3 14 20	20	21	5.9	2	81	83	++++	Variable 10–30%
31	Barley		18	11	71	3.8	0.3	0.3 13	19	29	6.2	4	80		Ι	Variable >30%
32		Barley	17	6	74	4.0	0.3	0.3 13 18	18	31	6.2	3		80	I	Variable >30%
33	Wheat Oats	Oats	11	12	77	4.2	0.3	0.3 13	18	34	6.2	3	95	81	Ι	Variable 10–30%
34	Wheat Oats	Oats	6	13 78	78	4.1	0.3	0.3 13	19	26	6.1	5	109	75	++++	Variable 10-30%
<sup>a</sup> Use of o <sup>b</sup> Crops gi	rganic fert ·own in fiv	Use of organic fertilization (animal manure) in five previous years: – only mineral fertilization; + sometimes; ++ regularly. Crops grown in five previous years: cereals, only cereals in rotation; variable 10–30%, 10–30% crops in rotation other than ce	al manur s: cereals	e) in fiv , only c	e previou ereals in	is year rotatio	s: – on n; vari:	ly mine. able 10-	ral fertiliza -30%, 10–30	tion; + somet. 0% crops in ro	imes; ++ re tation othe	egularly. r than cereals; v	variable >3	30%, more t	han 30% of the cro	<sup>a</sup> Use of organic fertilization (animal manure) in five previous years: – only mineral fertilization; + sometimes; ++ regularly. <sup>b</sup> Crops grown in five previous years: cereals, only cereals in rotation; variable 10–30%, 10–30%, rotation other than cereals; variable >30%, more than 30% of the crops in rotation other

than cereals.

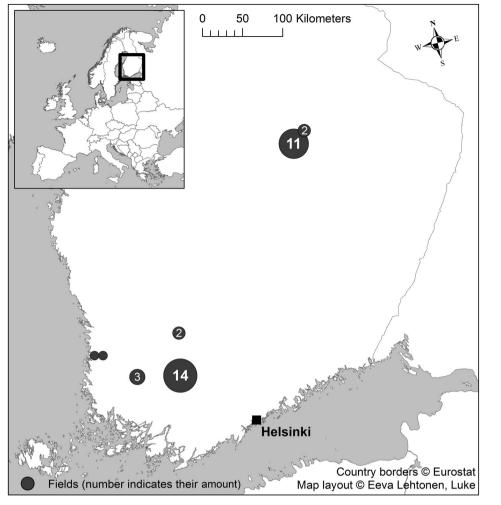
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The WEOC was extracted with deionized water (1:10) at room temperature for 1 hr, filtered (0.2-µm syringe filters) and analysed with high-temperature catalytic oxidation (HTCO) using a Shimadzu TOC-V CPH analyser (Shimadzu Scientific Instruments, Kyoto, Japan). Soil mineral N was extracted with 1 M KCl (1 hr agitation at soil-to-solution ratio 1:5), and after filtration (0.7 µm glass fibre filter) the NO<sub>3</sub>-N and NH<sub>4</sub>-N concentrations of the extracts were analysed with a continuous flow analyser (Skalar San++ System, Skalar Analytical BV, Breda, The Netherlands). The total dissolved N (TDN) content of the KCl extracts was analysed after oxidative digestion with potassium persulphate (K<sub>2</sub>O<sub>8</sub>S<sub>2</sub>, Skalar San++ system). Finally, net nitrogen mineralization was calculated as the difference between soil mineral N contents (sum of NO<sub>3</sub>-N and NH<sub>4</sub>-N) at the end and at the beginning of the incubation period (0-14 days and 14-30 days).

For the soil respiration measurements conducted on days 1, 3, 5, 10, 15, 21 and 30 from the start of incubation, the incubation bottles were first aerated with pressurized air and then closed with a chlorobutyl septum for 24 hr before sampling of gas from the headspace. The sample for background concentration of CO<sub>2</sub> was taken from an infusion bottle aerated and incubated without soil. Gas samples of 8 ml were taken with an injection needle pushed through the septum and introduced to He-flushed and evacuated 3-ml Exetainer® vials. The CO2 concentrations of gas samples were analysed with a gas chromatograph (Agilent 7890B GC system, customized, equipped with thermal conductivity detector (TCD), flame ionization detector (FID) and electron capture detector (ECD) and an autosampler; Agilent Technologies, Santa Clara, CA, USA). After subtracting the background concentration from the concentrations in the bottles incubated with soil, the respiration rate at each sampling time was calculated as mg of CO<sub>2</sub>-C per kg of soil dry matter (DM, oven drying at 105°C) per day and as mg of CO<sub>2</sub>-C per kg of soil OC per day. Respiration between sampling times was estimated with linear interpolation.

# 2.3 | Nitrogen fertilization and yield responses

For estimating the inherent soil N supply under field conditions, unfertilized areas within fertilized cereal fields were established at 13 fields in 2016 and at 33 fields in 2017 (Table 1). In 2016, fields of the research stations of the Natural Resources Institute Finland (Luke) in Jokioinen in south-western Finland and Maaninka in east-central Finland were used. In 2017, in turn, 17 fields of the experimental stations, including 12 sampled already in 2016 (Table 1), were complemented with 16



**FIGURE 1** Locations of fields where soil and yield samples were collected. Number  $(\geq 2)$  indicates the number of fields in the location shown by the circle

fields of private farmers. In each unfertilized area and adjacent fertilized area, a representative central sampling point was chosen, and three surrounding sampling points were placed on a circumference at an approximate distance of 5 m from the central point. Yield samples were collected from unfertilized and fertilized areas, but soil samples for agronomic soil testing and textural analysis were only taken from one point per field and were expected to represent the texture of the uniform sampling area.

Plant samples for determining cereal yields were collected from the same four sampling points before harvest in late summer 2016 and/or 2017. The yield samples were harvested after ripening by cutting down the entire plant stand within a 75 cm  $\times$  75 cm frame. The plant material was separated into straw and spikes, dried at 60°C and weighed. The spikes harvested from the central sampling point were further threshed to separate grains from the chaff, after which the share of grains in the spikes was calculated. This share was used in converting the spike yields to grain yields also in the three surrounding sampling points. Finally, the grain yield in each site was calculated as a mean of the four sampling points. For the crop N uptake, the content of total N (TN) in the grains and straw from the central sampling point were analysed by the Kjeldahl method (Foss Kjeltec<sup>™</sup> 8,400; Hilleroed, Denmark). Crop N uptake per hectare was calculated as a sum of N in the grains and straw. This crop N uptake was compared with grain yield and with the clay soil N mineralization potential estimated on the basis of the linear regression between the clay/C ratio and N mineralization in the laboratory incubation. Furthermore, apparent N recoveries were calculated as percentages of added N measured in the grains and straw at harvest. For this aim, the difference in crop N uptake between fertilized and unfertilized areas was divided by the amount of applied N. The agronomic N use efficiency was calculated by dividing the difference in crop yield between fertilized and unfertilized areas by the amount of applied fertilizer N.

Soil samples for the agronomic soil test (STP) (0.5 M ammonium acetate-acetic acid extraction (AAAc,

 TABLE 2
 Properties of the soils used in the laboratory incubation study in ascending order of clay %

				%				mg kg <sup>-1</sup>			
Field	Location	n	pН	Clay	С	Ν	Clay/C	WEOC <sup>a</sup>	NH <sub>4</sub> -N	NO <sub>3</sub> -N	TDN <sup>b</sup>
4	Α	4	6.4	5	2.1	0.21	2	83	0.29	14.0	21.4
4	b	2	6.2	13	2.3	0.22	6	76	0.33	17.7	26.5
8	С	4	6.8	14	2.0	0.20	7	80	0.37	10.5	15.4
8	d	4	6.6	15	2.1	0.19	7	76	0.33	9.7	14.7
4	e	2	5.8	30	9.7	0.63	3	170	0.77	19.9	30.8
20	f	4	6.0	44	3.3	0.33	13	160	0.81	15.5	23.3
20	G	4	5.9	47	3.5	0.34	13	157	0.86	21.4	28.2
27	h	4	5.0	68	8.5	0.69	8	157	0.73	23.3	40.1
27	Ι	4	5.0	69	7.2	0.60	10	148	0.63	21.6	35.8
28	J	4	6.3	71	3.2	0.33	22	213	0.75	12.9	19.9
29	K	4	6.0	71	3.6	0.35	20	196	0.94	14.0	22.3
29	1	4	6.0	73	3.5	0.35	21	240	0.70	10.1	17.6
29	m	4	6.0	73	3.8	0.35	19	252	0.67	17.2	25.6
28	n	4	6.3	74	3.2	0.31	23	230	0.70	11.1	17.3
28	0	4	6.5	75	3.0	0.30	25	165	0.80	11.3	17.8

The field numbering corresponds to that used in Table 1. Locations marked in capital and small letters refer to samples taken from fertilized and unfertilized areas, respectively. Values are means of 2-4 replicate samples (*n*) taken from each sampling location. Soil texture (clay%) was measured from one sample per location.

<sup>a</sup>Water extractable organic carbon.

<sup>b</sup>Total dissolved nitrogen.

pH 4.65), Vuorinen & Mäkitie, 1955), textural analysis, pH and potential cation exchange capacity (CEC) were collected from the 0–20-cm surface layer at each central sampling point. For analyses of OC and total N concentrations, a 4.5-cm-diameter soil core (0–20 cm) was augered, dried at 40°C, weighed and milled through a 2mm sieve. The C and N were analysed by dry combustion (Leco TruMac CN, LECO Corporation, St. Joseph, Michigan, USA), assuming that in these acidic soils very low in carbonates, the amount of total C is practically equal to the amount of OC. The C and N concentrations were calculated per mass of oven dry (105°C) soil.

### 2.4 | Statistical analyses

The relationship between soil C and mineralized N (mg N kg<sup>-1</sup> day<sup>-1</sup>), and the clay/C ratio and mineralized N in clay soils during the incubation study was assessed with linear regressions. A broken-stick regression model was fitted to C% or clay/C and CO<sub>2</sub> production (mg C kg<sup>-1</sup> day<sup>-1</sup>) in clay soils and the change points were modelled with a nonlinear model using least squares through an NLIN procedure in SAS.

The linear relationship between clay/C ratios and N mineralization in the incubation study was applied in

estimating the net N mineralization in clay soils of the field experiment. To characterize the relationship between the calculated estimates of the N mineralization and actual crop N uptake in the field, a linear regression line was fitted to the results.

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The relationships between selected soil properties (C %, N%, Clay%, Clay/C and CEC) and grain yields were investigated with Spearman correlations. Based on the preliminary screenings the fields were divided into two soil types; coarse (clay <30%, in 2016 n = 4 and in 2017 n = 16) and clay (clay  $\geq 30\%$ , in 2016 n = 9 and in 2017 n = 17) soils. A linear regression line was fitted to the grain yield (unfertilized or fertilized) and soil C% or Clay/C of clay soil fields. Similarly, a linear regression line was fitted to the grain yield (unfertilized or fertilized) and CEC of coarse-textured fields. To analyse whether the relationship between the soil properties (C%, Clay/C in clay soils and CEC in coarse-textured soils) and grain yields differed between fertilized and unfertilized areas, linear mixed models with C% (or Clay/C or CEC in other models) and the soil type and their interaction as fixed effects, were used. Correlated years from the same sites were taken into account with a G-side random effect, and unfertilized and comparable fertilized areas with an Rside random effect having a compound symmetry covariance structure. Comparisons of the slopes were conducted with two-tailed *t*-tests. All models were created using the GLIMMIX procedure of the SAS Enterprise Guide 7.1 (SAS Institute Inc., Cary, NC, USA).

### 3 | RESULTS

## 3.1 | Potential net N mineralization and soil respiration

In laboratory incubation, net N mineralization generally increased with increasing soil C%, but in clay soils with similar C% the amount of N mineralized varied largely (Figure 2a). The mean daily CO<sub>2</sub>-C production over the 30-day incubation increased with soil C% up to the change point of C% 3.4, after which the broken stick regression model showed a change in the relation and the increase in respiration rate with soil C% slowed down (Figure 2b). In coarse-textured soils (clay <30%), the C% was lower than in clay soils, and consequently, the respiration remained lower. When expressed as CO<sub>2</sub>-C produced per kg of soil C, in contrast, respiration rates were lowest in soils with C > 7% (Supporting Information 1).

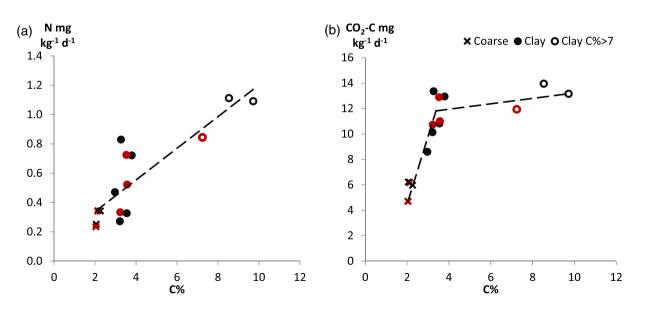
In clay soils, there was a clear negative linear relationship between net N mineralization and the soil clay/ C ratio (Figure 3a). The net mineralization was low in coarse-textured soils, and similar rates of net N mineralization were measured in coarse-textured soils with C% of 2.3 and in clay soils with C% of 3.2. Clay soils also exhibited a clear negative relationship between the clay/ C ratio and respiration rate but only in soils with clay/C ratios higher than 18 (Figure 3b). In clay soils with a clay/C ratio below 18, the respiration was not related to the clay/C ratio. In coarse-textured soils, respiration was low despite the low clay/C ratio (Figure 3b).

## 3.2 | Soil net N mineralization and crop N uptake

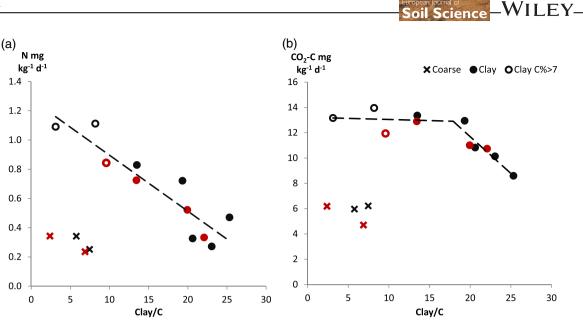
potential The estimated net Ν mineralization (mg N kg<sup>-1</sup> day<sup>-1</sup>) in the clay soils, acquired with the regression equation accounting for the soil clay/C ratio based on the incubation experiment, correlated fairly well with the crop N uptake at the unfertilized sites in the field (Figure 4). As expected, there was a significant positive correlation between the measured crop N uptake and grain yield despite the different crops, fertilization levels and variation in the weather conditions between sites and years (Figure 5). In the current grain yield range of up to 7,000 kg  $ha^{-1}$ , the correlation between crop N uptake and grain yield appeared linear. Thus, the relationships between soil properties and grain yield are similar, as the relationships between soil properties and crop N uptake would be.

## 3.3 | Yields in unfertilized and fertilized areas in the field

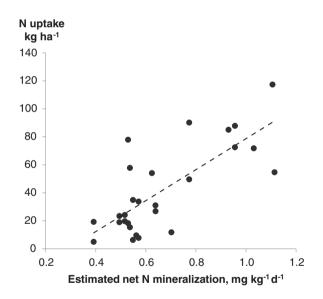
In the unfertilized areas of the coarse-textured soils, no significant correlation was found between soil C% or N%



**FIGURE 2** Net nitrogen mineralization (a) and respiration rate (b) over the 30-day laboratory incubation in coarse-textured (clay% <30) and clay (clay% >30) soils and in clay soils with C% higher than 7 plotted against soil C%. Red colour indicates that the soil samples were taken from the fertilized area. In (a) the linear regression line was fitted to the clay soil data points (p < .0001). In (b), the broken-stick regression model showed a significant positive relationship below the change point C 3.4% (p < .0001) and after the change point (p < .0001) [Color figure can be viewed at wileyonlinelibrary.com]

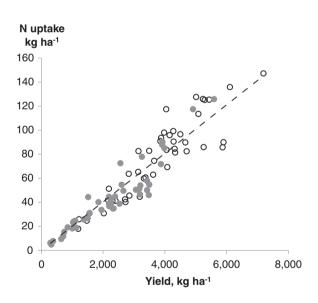


**FIGURE 3** Net nitrogen mineralization (a) and respiration rate (b) over the 30-day laboratory incubation in coarse-textured (clay% <30) and clay (clay% >30) soils and in clay soils with C% higher than 7 plotted against the soil clay/C ratio. Red colour indicates that the soil samples were taken from the fertilized area. In (a), the linear regression line was fitted to the clay soil data points (p < .0001). In (b), the broken-stick regression model showed a negative relationship between respiration rate and clay/C ratio in clay soils having a clay/C ratio higher than 18 (p = .0135), whereas in clay soils with clay/C ratio smaller than 18, the respiration rate was not related to clay/C ratio (p = .831) [Color figure can be viewed at wileyonlinelibrary.com]



**FIGURE 4** Net N mineralization rate in the clay soils as estimated from the relationship between mineralization and soil clay/C ratio in the incubation study plotted against crop N uptake in unfertilized areas of the fields. Linear regression line was fitted to the results ( $r^2 = 0.5667$ , p < .0001)

and crop yield (Table 3), but, however, the fertilized yield and the agronomic N use efficiency increased with increasing C% and N%. Because there was a significant positive relationship between soil C% and N% (Supporting Information 2), further results are presented only in relation to soil C%. In clay soils, a positive



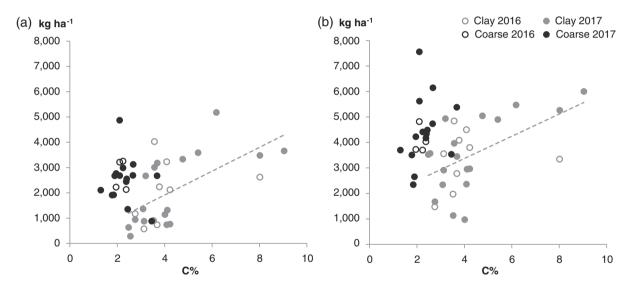
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**FIGURE 5** Grain yields (kg ha<sup>-1</sup>) in fertilized (open black circles) and unfertilized (grey) areas plotted against N uptake (kg ha<sup>-1</sup>) in the central sampling points of the fields. Linear regression line fitted to the results including both fertilized and unfertilized areas ( $r^2 = 0.8787$ , p < .0001)

correlation was found between soil C% and both the fertilized and unfertilized yields, but there was a large variation in yields especially among the soils with C%, ranging between 2 and 4 (Figure 6). Clay and the clay/C ratio correlated negatively with yields in both unfertilized and fertilized areas (Figure 7). The slope of the regression **TABLE 3** Spearman correlations between soil parameters (organic carbon%, C%; total nitrogen, N%; clay content, clay%; clay/C ratio; cation exchange capacity, CEC) and cereal yields (unfertilized yield, yield 0; fertilized yield, yield N; yield increase obtained with 1 kg of fertilizer N, agronomic NUE [N use efficiency]) of (a) coarse-textured soils and (b) clay soils

	(a)									
Coarse-textured soils	<b>C%</b>		N%		Clay%		Clay%/C	9%	CEC	
N = 19	R	р	R	р	R	р	R	р	R	р
Unfertilized yield	0.075	.7612	0.031	.9005	-0.064	.7931	-0.110	.6544	0.493	.0320
Fertilized yield	0.470	.0422	0.486	.0349	0.236	.3310	0.096	.6965	0.453	.0517
Agronomic NUE	0.729	.0004	0.805	<.0001	0.283	.2409	0.025	.9204	0.097	.6938
	(b)									
Clay soils	<b>C%</b>		N%		Clay%		Clay%/C%		CEC	
N = 27	R	р	R	р	R	р	R	p	R	р
Unfertilized yield	0.607	.0008	0.571	.0019	-0.395	.0417	-0.6815	<.0001	-0.315	.1090
Fertilized yield	0.498	.0082	0.500	.0079	-0.457	.0165	-0.6809	<.0001	-0.367	.0599
Agronomic NUE	-0.074	.7139	-0.056	.7829	0.032	.8739	0.0092	.9638	-0.006	.9783

Statistically significant (P < .05) correlations indicated in bold.



**FIGURE 6** Grain yields (kg ha<sup>-1</sup>) in 2016 and 2017 from (a) unfertilized and (b) fertilized soils plotted against soil C%. Regression lines were fitted to the clay soil data points with a significant linear relationship between C% and (a) unfertilized yield ( $r^2 = 0.3552$ , p = .001) and (b) fertilized yield ( $r^2 = 0.2925$ , p = .0037)

equation of yield over clay/C ratio did not differ between unfertilized and fertilized soils (p = .722). In coarse soils, the clay/C ratio was not related to the yields, but instead, the yields in unfertilized and fertilized areas were positively related to CEC (Figure 8). However, fertilization did not induce larger yield increases at lower CEC than at higher CEC, as indicated by broadly parallel slopes of the regression equations in unfertilized areas with no fertilization for two sequential years did not produce lower yields in the second year than in the first year (Supporting Information 3). The yield differences between adjacent fertilized and unfertilized areas within the same field corresponded to on average 16 kg additional grain yield per 1 kg of applied fertilizer N. However, the variation was large, and in some areas, yield increase was not achieved at all with N fertilization. Consequently, the apparent N recovery ranged between 0 and 79%, being on average 35%. As the yield gained without fertilization increased, the maximum additional yield per kg of fertilizer N decreased (Figure 9). At sites with low inherent productivity (low yields without fertilization), the effectiveness of N fertilization varied considerably.

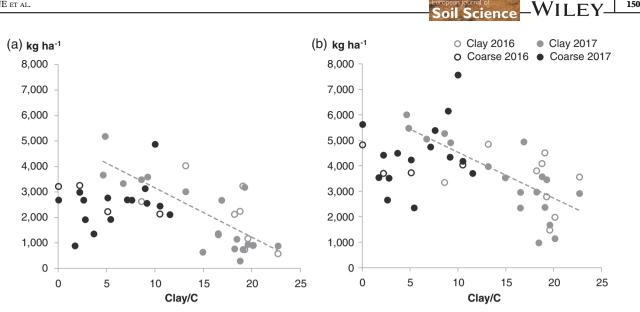


FIGURE 7 Grain yields (kg ha<sup>-1</sup>) in 2016 and 2017 from (a) unfertilized and (b) fertilized soils plotted against the soil clay/C ratio. Regression lines were fitted to the clay soil data points with a significant negative linear relationship between the clay/C ratio and (a) unfertilized yield ( $r^2 = 0.5866$ , p < .0001) and (b) fertilized yield ( $r^2 = 0.4873$ , p < .0001)

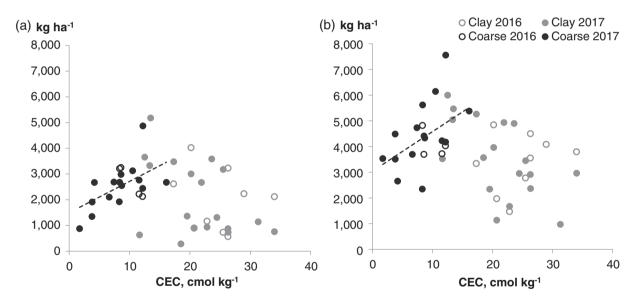


FIGURE 8 Grain yields (kg ha<sup>-1</sup>) in 2016 and 2017 from (a) unfertilized and (b) fertilized soils plotted against soil cation exchange capacity (CEC). Regression lines were fitted to the coarse soil data points with a significant linear relationship between CEC and (a) unfertilized yield ( $r^2 = 0.2800$ , p = .0198) and (b) fertilized yield (coarse soils:  $r^2 = 0.2117$ , p = .0475)

#### 4 DISCUSSION

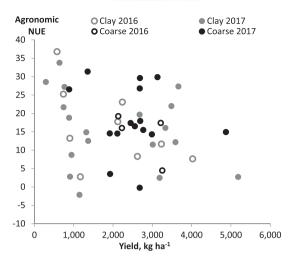
#### Organic matter mineralization and 4.1 soil N supply affected by varying clay contents

In favourable temperature and moisture conditions, the soil respiration rate is generally limited by the supply of substrate (Wang, Dalal, Moody, & Smith, 2003). As expected, because soil CO<sub>2</sub> production is largely dependent on soil OC (Wang et al., 2003), we measured the

lowest respiration rates in our laboratory incubation in soils with the lowest C%. The respiration rate increased linearly up to C% 3.4, but the soils with C% higher than 7 produced less CO<sub>2</sub>-C than expected based on their C%. In these high-C% soils, the WEOC did not clearly differ from the other clay soils with much lower C% (coarse soils were clearly lower in WEOC), indicating solubilityrelated differences in OM quality. The dissolved OC is probably the most bioavailable fraction of soil OM (Marschner & Kalbitzb, 2003), and thus, in addition to the total amount of OC and soil water content (Schjønning,

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**FIGURE 9** Relationship between unfertilized grain yields (kg ha<sup>-1</sup>) and agronomic N use efficiency (agronomic NUE, additional grain yield kg ha<sup>-1</sup> per kg of fertilizer N) in clay and coarse-textured soils in 2016 and 2017

Thomsen, Moldrup, & Christensen, 2003), the properties of OM that determine the solubility of soil OC regulate the soil OC mineralization.

The sorption of organic molecules onto mineral particles affects soil OM turnover through reduced microbial access to the substrate (Dungait, Hopkins, Gregory, & Whitmore, 2012; Schmidt et al., 2011). Lower soil OM mineralization with increasing soil clay content has been reported (Cote, Brown, Pare, Fyles, & Bauhus, 2000; Hassink, 1997; McLauchlan, 2006; Wang et al., 2003), and in accordance with this, our results showed that among the soils with similar C%, the lowest respiration was observed in soil with the highest clay content (Supporting Information 1). In soils with a clay/C ratio above 18, the respiration rate clearly decreased with increasing clay/C ratio, suggesting that in soils with a clay/C ratio below 18 the accessibility of substrate does not significantly reduce respiration.

Because of the tightly coupled cycles of C and N, higher OC mineralization rates may indicate also higher potential of soils to supply N. We measured clearly higher respiration rates for clay soil with C% of 3.2 than for coarse soil with C% of 2.1; however, the net N mineralization rate did not differ between these two soils. This suggests that the factors regulating mineralization of organic N partly differ from those of organic C. The incubation experiment showed a linear decrease in the net N mineralization with an increase in the clay/C ratio and may indicate a more important role of clay in controlling the availability and mineralization of organic N than in regulating the respiration. Thus, our results support the finding that N-rich organic molecules that sorb directly onto mineral surfaces (Dümig, Häusler, Steffens, & KögelKnabner, 2012; Kopittke et al., 2020) are better protected by clay particles than molecules with a higher C/N ratio. This is in agreement with the previous research reporting higher C/N ratios in unprotected OM than in organic molecules adsorbed onto clay particles (Guggenberger, Christensen, & Zech, 1994; Kleber, Sollins, & Sutton, 2007).

Soil aggregation and pore structure are linked intimately to the processes that stabilize OM and protect it from microbial degradation. The incubation experiment was performed at room temperature with sieved (<5 mm) soil samples, and the soil pore structure was changed by loosening in pretreatments. Thus, the determined respiration and net N mineralization reflect the potential for mineralization, and the results are not directly applicable to field conditions (e.g., Moinet et al., 2016). Hassink (1992) observed that sieving caused a temporary increase in the mineralization of C and N, which interacted with soil texture such that the relative increase in C and N mineralization due to sieving was larger for loamy and clayey soils than for sandy soils. On average, the daily net N mineralization in our incubation experiment was  $0.57 \text{ mg kg}^{-1}$ . By using typical bulk densities and accounting for the textural differences, this mineralization rate can be calculated to equal  $1.23 \text{ kg N ha}^{-1} \text{ day}^{-1}$  (0.7–2.0 kg N ha<sup>-1</sup> day<sup>-1</sup>). This value is somewhat higher than available earlier estimates from field experiments in comparable climatic conditions. Lindén, Lyngstad, Sippola, Søegaard, and Kjellerup (1992) reported an average N mineralization rate of 0.40 kg ha<sup>-1</sup> day<sup>-1</sup> in field experiments conducted in Denmark, Finland, Norway and Sweden in different soils. Delin and Lindén (2002) reported that daily net N mineralization within one field in south-western Sweden varied between 0.10 and 0.93 kg N ha<sup>-1</sup> day<sup>-1</sup>. Compared with the reported field observations, the results of the laboratory incubation agree quite well with the observation by Stenger, Priesack, and Beese (1995) that mineralization rates of disturbed samples were approximately twice as high as those of undisturbed samples.

## 4.2 | Yield levels as affected by soil organic carbon and texture

According to Ros (2012), irrespective of geographical location and land use, soils with higher OC content have higher N mineralization rates, and thus, higher inherent N supply. Likewise, Hijbeek et al. (2017) and Schjønning et al. (2018) indicated a reduction in the need for mineral N with an increase in soil OM content. In previous studies, both positive or conditionally positive effects (e.g. on a limited range of C%) (Majumder et al., 2008; Oldfield,

Bradford, & Wood, 2019; Oldfield, Wood, & Bradford, 2018) and a lack of a relationship or even negative influences in experiments where N supply had been secured with fertilization (Hijbeek et al., 2017; Schjønning et al., 2018) of soil OM on yields have been reported. In the results of Oldfield et al. (2018) showing on average higher maize and wheat yields with higher soil OC, the yield increases levelled off at c. 2% OC. In our field measurements, a positive relation was found between soil C% and grain yields in fertilized and unfertilized clay fields, although in all studied clay soils C% was higher than 2. In the coarser unfertilized soils with C% varying between 1.3 and 3.7, the relation between the yields and soil C% was insignificant but the yields increased with increasing CEC, suggesting that the availability of nutrient cations was more important in regulating the yields in these fields. Further in coarse soils, the agronomic N use efficiency was higher in soils with higher C%, also indicating that the OC enables efficient use of added fertilizer N. This further highlights the importance of soil OC for other soil productivity factors (water retention, structure, macro- and micronutrients) besides N supply.

In agreement with the results of laboratory incubation of clay soils, unfertilized areas in fields with a higher clay/C ratio resulted in lower grain yields, suggesting lower mineralization rates due to the N-rich molecules being protected by clay mineral particles. However, a similar trend of decreasing yields with increasing clay/C ratio was detected in the areas receiving mineral N fertilization. This conflicts with the general assumption that in soils with properties indicating low inherent N supply the yields would be increased more with proper N fertilization. Especially in soils with a clay/C ratio higher than 15, the variation in vields in fertilized areas was large; this implies that some of the areas suffered from yield restrictions that prevented the utilization of fertilizer N. Yield restrictions non-compensable by N fertilization may derive from, for instance, shortages of other nutrients or defects in soil structure affecting root growth, water infiltration and storage, and air-soil gas exchange (Olfs et al., 2005). In poor structured soils with restricted aeration, N limitation due to gaseous losses via denitrification is also possible. In fact, high clay/C ratios have been found to indicate poor structure in clay soils (Johannes et al., 2017; Soinne, Hyväluoma, Ketoja, & Turtola, 2016). Thus, for clay soils in a cool and humid climate, the higher the clay content, the more OC is needed to compensate for it and to maintain both soil structural stability and efficient use of applied N.

In Denmark, Schjønning et al. (2018) found the yields of non-N-fertilized winter wheat to range from less than 1,000 kg ha<sup>-1</sup> to more than 9,000 kg ha<sup>-1</sup>. In the data set

of Valkama et al. (2013), which included 61 relevant N fertilizer trials conducted with spring cereals in Finland since the 1940s, the yields of non-N-fertilized control areas ranged similarly, although with a lower upper range, from 1,000 to 4,000 kg  $ha^{-1}$ . In the present study, the wide range of yields achieved without N fertilization (from 400 to 6,000 kg  $ha^{-1}$ ) is in agreement with previous findings and reflects substantial variation in the inherent productivity of the studied soils. However, in fertilized areas, the variation in yields was roughly similar to that in the unfertilized areas. The very low apparent N recoveries occasionally recorded in the current study (<10% or even zero) indicate a serious lack of synchrony between the supply and demand of N. This will result in a negative economic impact and unnecessary environmental hazards (Cassman, Dobermann, & Walters, 2002). Together, the large differences in the yield response to added N between fields (additional yield per kg of fertilizer N varying from -2 to 37 kg) and the high variation in yields in the fertilized areas indicate a coinciding influence of growth factors other than N supply.

The observation that the incremental agronomic N use efficiency decreased with increasing non-N-fertilized yield level is in agreement with the previous findings of Valkama et al. (2013). It is also compatible with Mitscherlich's law of diminishing returns, which assumes that the fraction of fertilizer addition used by the crop becomes smaller and smaller as the production approaches its maximum level (Ferreira, Zocchi, & Baron, 2017). Overall, the results of the present and earlier studies (Kachanoski, O'Halloran, Aspinall, & Von Bertoldi, 1996; Lory & Scharf, 2003; Valkama et al., 2013) demonstrate that the yields achieved with (near-) optimum N fertilization are poor predictors of responsiveness of a crop to added N at a given site. Therefore, instead of estimating the fertilizer N requirement based on yield expectancy, determination of the ability of added N to increase the yield site-specifically by comparison of fertilized and non-N-fertilized areas appears recommendable. The efficiency of fertilizer N use could be greatly enhanced by the allocation of higher rates of N to responsive sites and reducing inputs to non-responsive sites, which may, according to the current data and Kachanoski et al. (1996), be the highest and lowest-yielding sites, respectively.

### 5 | CONCLUSIONS

In boreal clay soils with varying cultivation and fertilization history, both the net N mineralization and soil productivity decreased with increasing clay/C ratio. Our results suggest that for soils with high clay content, the -WILEY-Soil Science

protective effect of clay on soil organic N should be considered as one factor when estimating the inherent N supply of the soil. In soils with a high clay/C ratio, the agronomic N use efficiency varied considerably, suggesting that in these soils the growth is often limited by poor soil structure. In a cool and humid climate, the higher the clay content, the more OC is needed to ensure production of high yields in an environmentally sustainable way. In studied coarse-textured soils with a narrower range of OC, the yields increased with increasing CEC while OC enabled efficient use of added fertilizer N.

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Study concept and design:T. Salo, E. Turtola, H. Soinne, R. Keskinen, S. Kanerva, A. Simojoki and V. Nuutinen. Acquisition, analysis and interpretation of data: H. Soinne, R. Keskinen, M. Räty, S. Kanerva, A. Simojoki, J. Kaseva and T. Salo. Drafting the manuscript: H. Soinne, R. Keskinen, M. Räty, S. Kanerva, A. Simojoki and T. Salo. Critical revision of the manuscript for important intellectual content: E. Turtola and V. Nuutinen. Statistical analysis: J. Kaseva and H. Soinne. Obtained funding: T. Salo.

### **CONFLICTS OF INTEREST**

The authors have no conflicts of interest to declare.

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### SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

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