AGRICULTURAL AND FOOD SCIENCE

Agricultural and Food Science (2023) 32: 195–206

https://doi.org/10.23986/afsci.131550

Gypsum amendment influences soil and plant chemical composition temporarily

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Field application of gypsum ($CaSO_4 \times 2H_2O$) is a tool to decrease phosphorus (P) load to watercourses. Effects of gypsum application (4 tons ha^{-1}) on soil and crop composition were monitored up to four years after the treatment in the Savijoki catchment, southwestern Finland. Eleven amended fields and 17 control fields were analyzed for easily soluble P, Ca, Mg, K, S, pH(H_2O), electrical conductivity (EC) and loss-on-ignition. Plant samples were analyzed for most nutrients and Se. Downward S transport was monitored in two amended fields. In the first and second spring after gypsum application, S concentrations and EC were substantially but not harmfully elevated in the plough layer, with no other significant effects on soil composition. Sulfur leached out slightly less rapidly from clay than from the coarse mineral soil. Plant S concentration increased for the first and the second year. In conclusion, gypsum application did not have adverse effects on soil or crop chemical composition in this survey of predominantly clay soils.

Key words: electrical conductivity, leaching, magnesium, phosphorus, sulfur

Introduction

Anthropogenic P loading into the Baltic Sea is a major environmental problem (Andersen et al. 2017). The Archipelago Sea on the southwestern coast of Finland is particularly loaded, with severe signs of eutrophication. This sea area is impacted not only by the generally weak state of the Baltic Sea but especially in the coastal areas also by continuous nutrient losses from the clayey cultivated land.

Application of gypsum to agricultural fields has been identified as a cost-effective tool to decrease P losses into watercourses (Iho and Laukkanen 2012, Ollikainen et al. 2020). Gypsum has been studied, and used, mainly in the USA (e.g., Shainberg et al. 1989, DeSutter and Cihacek 2009, Kost et al. 2018) and Finland (Ollikainen et al. 2020) and found to decrease the P load up to 50% (Ekholm et al. 2012, Uusitalo et al. 2012). In Finland, a large-scale pilot was organized in the Savijoki catchment (Ollikainen et al. 2020) where gypsum generated as a by-product of phosphoric acid production in Siilinjärvi, eastern Finland was applied.

Application of gypsum gives rise to several reactions in soil because the prevailing semi-equilibrium is disturbed upon addition of a relatively easily soluble component into the system. Solubility of gypsum in water is about 3.15 g l^{-1} while the solubility of calcium carbonate is only 0.013 g l^{-1} (Appelo and Postma 2005). The elements S and Ca are released into the soil solution as sulfate (SO_4^{2-}) and Ca^{2+} ions upon gypsum dissolution. In the slightly or moderately acidic soils of Finland, practically no adsorption of sulfate takes place (Yli-Halla 1987), so this ion remains in the pore water. Gypsum is an old commercial fertilizer used as a source for both S and Ca (Batte and Forster 2015). It is largely acknowledged that S fertilization is needed in Finland (Yli-Halla et al. 2011). Particularly, horticultural crops and potato receive their fertilizer K in the form of K_2SO_4, being supplied with up to 50 kg of S per hectare while cereals and grass receive fertilizers that contain KCl but, owing to added S compounds in many fertilizers, also those crops get a S dose of 10–20 kg ha^{-1}. Moreover, gypsum application has been proposed to increase Ca supply to potato without increasing the pH (Sipilä and Virtanen 2010) as a contrast to agricultural lime. Since sulfate is not retained by the soil matrix, the EC is increased in the soil solution, which may at high values hamper plant growth. On the other hand, lack of sulfate adsorption results in ultimate leaching of this ion out of the soil, similar to nitrate and chloride, accompanied by cations.

Cation exchange reactions take place after Ca is dissolved from gypsum. Calcium is the dominant exchangeable cation in soils of Finland (Räty et al. 2021) and compared to the original stock, the amount of Ca addition is relatively small. However, Ca originating from gypsum likely displaces some Mg and K from the exchange sites, as found in a lysimeter experiment (Uusitalo et al. 2012). There is also competition between Ca and Mg in plant uptake (e.g., Mengel and Kirkby 1979 p. 117–118 and p. 420). Both leaching and cationic competition can in principle result in decreased crop Mg uptake by excess Ca originating from the added gypsum. Certain proportions of Ca,

Received 14 July 2023 / Accepted 21 October 2023

The Scientific Agricultural Society of Finland

Mg and K (Ca 60–85%, Mg 6–20%, K 2–5% of cation exchange sites) have been proposed optimal for an "ideal" soil (Graham 1959, Albrecht 1975, Baker and Amacher 1981). However, this basic cation saturation ratio theory has lately been criticized and declared that "the scientific community disregards this theory" (Chaganti and Culman 2017). Experiments in soils which have deviated from these proportions have not produced adverse effects on crop production provided the amounts of available cations are at a sufficient level (e.g., Koppitke and Menzies 2007, Chaganti and Culman 2017, Leiva Soto et al. 2023). Occurrences of Mg deficiency in crop production in Finland have been difficult to find (Jokinen 1982). Among exchangeable cations, the amount of K retained in the cation exchange capacity (CEC) sites is very small (Räty et al. 2021) while much of the K supply to the crop comes from other K pools (Joy et al. 1973), most notably from the non-exchangeable K reserves, particularly abundant in clay soils (Kaila 1967). Sites of non-exchangeable K are not accessible to Ca and it is thus likely that K supply to plants can be affected by gypsum application only to a limited extent.

Phosphorus is the major element targeted by gypsum application. Losses of P from soil can take place 1) in the dissolved form both in surface runoff and in the discharge through the subsurface drainage pipes and 2) in a particulate form attached to or incorporated in the eroded soil material. Increased EC promotes flocculation of soil and decreases the tendency of the detachment of soil particles into the surface runoff, thus mechanically decreasing the flux of P out of the soil. Increased EC also increases sorption of dissolved P and lowers the equilibrium P concentration (Yli-Halla and Hartikainen 1996). Whether the decline of dissolved P concentration caused by the elevated EC upon gypsum addition decreases P supply to plants is a topic worth considering.

Micronutrient availability is largely controlled by soil pH. By temporarily increasing EC of soil solution, addition of gypsum may decrease the pH, just like the difference of about 0.5 pH units between pH(H_2O) and pH($CaCl_2$) in non-saline soils (Minasny et al. 2011). In principle, there is thus a short-lived possibility of a slight increase of micronutrient availability in soil after gypsum application. An additional and a more likely interaction is competition between S and Se in plant uptake (e.g., Yläranta 1990b). Very low level of Se in crops in Finland without Se fertilization has been identified (Sippola 1979) and common application of Se fertilization since 1985 has increased the crop Se concentration to the optimal level as for consumers (Yläranta 1990a). It needs to be seriously considered whether gypsum application gives rise to a need of elevated Se fertilization.

The primary purpose of the entire pilot was to examine whether extensive application of gypsum within a watershed has an influence on the nutrient losses from fields to surface waters. The application has impacts on soil and potential effects on the composition of plants growing in the respective fields as well. The extent and duration of these effects are reported in this paper.

Materials and methods

The pilot area is located in southwestern Finland in the catchment of the Savijoki river, which is a tributary of the Aurajoki river in the catchment flowing into the Archipelago Sea. The Savijoki catchment (130 km^2), mostly in the Lieto municipality, consists of cultivated river valleys and forested upland areas. The activities of this project were carried out on cultivated private fields within the upper and middle reaches (81.3 km^2) of the catchment. The fields cover 43% of the area and are dominated by clay soils (Vertic Stagnosols; IUSS working group WRB 2014). The fields commonly slope towards creeks and the river which has carved itself down to the ground moraine through the glacial clay deposits often more than 20 metres thick. Some fields, usually bordering the upland forested areas, are formed of glacial till and have loamy and fine sand textures in the plough layer. The forested areas (51% of the area), located in the periphery of the catchment, are covered with glacial till (Gleyic Cambisols and Podzols), occasionally with very shallow and stony soil (Leptosols) and sparse vegetation, and the highest areas are characterized by exposed bedrock. Built areas cover 5% while there are few peatlands (1%) and no lakes in the catchment. There were 123 farms in the pilot area, and most of them grow spring-sown crops (82% of field area), mostly cereals with some oilseed crops. There were only 12 farms with animal husbandry, and grass covered only 17% of the field area in 2015. Other crops (mainly beets) accounted for less than 1%.

In August–November 2016, gypsum was applied onto 1494 ha of field with machinery used for spreading solid manure or agricultural lime (Ollikainen et al. 2020). The gypsum originated from the Yara Siilinjärvi phosphoric acid factory. This gypsum does not contain heavy metals or radioactive elements (Karhunen and Vermeulen 2000). The application rate was 4000 kg ha^{-1}, containing about 800 kg ha^{-1} of calcium and 640 kg ha^{-1} of S. If evenly distributed into the 20-cm plough layer (2×10^6 litres of soil), the increases of Ca and S correspond to 400 mg l^{-1} and 320 mg l^{-1}, respectively. The moisture content of gypsum was about 15%. Because of the origin, gypsum contains about 2 g kg^{-1} P in water-soluble form (Yara 2020), the P application in gypsum being 8 kg ha^{-1}. Gypsum also contains some F, originating from the apatite used as the raw material of phosphoric acid.

Gypsum was applied to the fields of farmers agreeing to participate in the project. Gypsum was applied onto 52% of the field area in the middle section (66.8 km^2) of the catchment. Besides gypsum application, the farmers managed their fields in their own individual way growing the crops of own selection and fertilizing them mostly with chemical fertilizers.

In August 2016 before gypsum application, plough layer soils of 28 fields were sampled at 0–20 cm. Two replicate 0.5-dm^3 samples were taken with an auger, each consisting of at least seven borings, and analyzed separately. The exact locations of the sampling sites were determined with GPS. The same sites were resampled after one, two and four winters after gypsum application in May 2017, May 2018 and April 2020. Eleven of the sites were in fields to be amended with gypsum and 17 in the control fields. The sites were chosen so that all areas and soil types (clay/ medium and coarse textured mineral soils) were covered both for gypsum-treated and untreated plots. Three fields receiving gypsum and two control fields were also sampled without replicates at the depth of 20–40 cm and 40–60 cm (the latter not in 2016) in order to monitor the downward transport of substances. Due to experimental errors, the results of one amended field had to be discarded. The soil samples were analyzed for pH(H_2O) and EC of the suspension at the soil-to-water ratio of 1:2.5, and easily soluble P, K, Ca, Mg and S were extracted with an acid (pH 4.65) ammonium acetate solution (0.5 M CH_4COOH, 0.5 M CH_4COONH_4), which is the soil testing method used in Finland (Vuorinen and Mäkitie 1955). The concentrations of the elements were measured with an ICP-OES device and results expressed on a volumetric basis, i.e., milligrams per litre of soil (mg l^{-1}). In cases where the concentration was below the detection limit of the analysis, the concentration was assumed to be 0.5 times the detection limit. As for a rough estimate of soil organic matter, loss-on-ignition (LOI) was determined by heating the soil samples at 550 °C for 4 h. The LOI results were not corrected on the basis of clay content. Soil texture was determined by finger assessment.

Composite samples of above-ground vegetation were collected with scissors at the flag leaf stage from the same sites sampled for soil analyses. Two samples were taken from each field on 26–30 June 2016, 2017 and 2020 and on 9–11 July 2018. In most fields, spring-sown cereals were growing, but some grass, pea and rapeseed stands were also present. The occasional grass, pea and rapeseed stands were sampled at the same time. Complete sets of samples were received from 25 fields (10 amended with gypsum and 15 control) because in some cases the field was bare or the grass had just been harvested, with no plants to be sampled. The samples were analyzed for N using the Kjeldahl method and for P, K, Ca, Mg, S, B, Cu, Zn, Mn and Fe by ICP-OES after dry ashing at 550 °C and dissolution of the ash with HCl. For the Se determination, the plant material was digested with HNO_3 and $Mg(NO_3)_2$ and Se was measured with the AAS using the hydride method.

The meteorological conditions may affect the fate of gypsum. The mean long-term temperature in a nearby meteorological site was 5.5 °C (Turku airport, 1971–2020), while the study period was warmer (6.4 °C, 2016–2020). The mean long-term precipitation in the Savijoki was 655 mm which is almost the same (642 mm) as in the study period, 19% of precipitation coming as snow. A total of 47% of precipitation formed runoff. Meteorological data was taken from the Watershed Simulation and Forecasting System (WSFS) (Vehviläinen et al. 2001) and runoff data from a hydrological database, both maintained by Finnish Environment Institute (Syke 2023).

The spreading period of gypsum (August–October 2016) was exceptionally dry, forming ideal conditions for gypsum spreading on the clay soils, easily disturbed by heavy machinery when wet. The late autumn of 2016 was also dry and the winter 2017 was relatively mild with little snow, topsoil being frozen only occasionally. The runoff (73 mm from November 2016 to April 2017) was only half of the usual amount, suggesting that gypsum leaching may not have been very effective during the first winter period (Table S1). The winters 2018 and 2019 were colder, but winter 2020 was exceptionally wet and warm. In January–May 2020, runoff in the Savijoki was 229 mm, whereas in other years it was in the corresponding period 65–191 mm.

As for the growing conditions of the crop, the reference year 2016 was rather average (accumulated effective temperature above 5 °C until sampling in late June 563 °C, Table S2) while the spring and early summer of 2017, i.e., for the first crop after gypsum application, were cool (399 °C), and spring cereals could be sown about one week later than usually. Instead, the spring and early summer of 2018 were warm (625 °C) and dry while the year 2020 was average in temperature (542 °C) and rather dry. This variation can be reflected in the physiological age of plants at sampling in different years and complicate the comparison of nutrient concentrations between the years.

Statistical modelling

The concentrations in soil and plants were modelled with a linear mixed-effects model with repeated measures so that the fixed effect variables were gypsum amendment (yes, no), sampling year (2016, 2017, 2018, 2020) and soil texture (clay, coarser mineral soil) plus their interaction when significant. Additionally, in case of soil analysis, continuous variables pH and LOI were used as covariates. The within group-errors were allowed to be correlated and have unequal variances. Several correlation structures for errors were tested: compound symmetry, unstructured, autoregressive covariances, autoregressive covariances with heterogeneous variances and autoregressive moving average covariances. The correlation structure was chosen based on the Akaike Information Criteria (AIC) and inspection of the data, and usually a first-order autoregressive structure with heterogenous variance-covariance structure or an unstructured covariance matrix produced the lowest AIC value. Field plot comprised the random effect variable, that is the inherent differences in the sites were taken into account but were not focal in our analysis. A mixed-effects model suits to our data, which is not balanced, i.e. the number of observations in each group differs. The analysis was made on log-transformed (except for pH) variables using lme function in R package nlme (Pinheiro et al. 2022) using R version 4.2.2 (R Core Team 2022). Log-transformation improved the scatter of standardized residuals, although in some cases did not eliminate the tendency of residuals to form separate groups, when gypsum had a high but temporary effect on the concentrations. Post hoc analysis comparing the contrast ratios of the estimated marginal means was performed by the emmeans package (Lenth 2023).

Results and discussion

Concentrations of elements in the plough layer soil

Clay soils, particularly clay loam, dominated (n = 20, 69%) among the samples while coarser loamy soils (n = 9, 31%) occupied the rest of the sampled fields. Fine sand and coarse silt were the principal textural classes within the coarse mineral soil samples. This distribution coincided with soil test results of the Lieto municipality in 2016–2020, where 77% of all tested samples (n = 1227) represented clay soils and 22% coarse mineral soils (Eurofins Viljavuuspalvelu Oy 2022). The absence of heavy clays, silt soils and organic soils in our material was in agreement with their very low occurrence in the fields of this municipality. LOI averaged at 6.9% (range 4.3–9.9%) and 6.3% (4.3–11.1%) in clay and coarser mineral soils, respectively.

Application of gypsum strongly increased the S concentration of the soil (Table 1) from the average of 8.1 mg l^{-1} (range 5.7–16.7 mg l^{-1}) in autumn 2016 before gypsum application to above 100 mg l^{-1} in spring 2017, i.e., 16- and 12-fold in clay soils and coarse mineral soils, respectively. A wide range of concentrations (8.3–371 mg l^{-1}) was probably caused by uneven distribution of gypsum. According to the results of the mixed-effects model, the S concentrations were higher ($p < 0.001$) in the amended than in the unamended plots after the first and the second winter, but after the fourth winter there was no more a significant difference between these two groups (Table S3). Soil texture did not explain S concentrations either alone or in interaction with other variables (Table S3).

Compared to fields not amended with gypsum, the soil S concentration after the first winter was 118 and 95 mg l^{-1} higher in clay and coarse mineral soils amended with gypsum (Table 1). This is less than the added amount 320 mg l^{-1}, if gypsum was evenly distributed in a 20 cm-deep plough layer. The difference can be caused by 1) less than complete dissolution of sulfate derived from gypsum during the 1-hr extraction, 2) leaching of S out of the plough layer already during the first winter and 3) relocation of the surface-applied gypsum by ploughing to depths slightly below the 20-cm sampling. The deficit can also be partly explained by the lower density of the pulverized soil measured for the analysis by the volume, compared to the undisturbed soil in the field.

After two winters, the average S concentration (24.9 mg l^{-1}) had substantially decreased. The concentrations were not monitored after the third winter but, after the fourth one and a total of 1110 mm of runoff, the average S concentration (11.4 mg l^{-1}) had decreased to the original level. Even at the end, there was a wide variation between the fields (3.3–87 mg l^{-1}). Similar large spatial variation was also observed in earlier studies (Yli-Renko and Rasa 2011, Ekholm et al. 2012, Suojala-Ahlfors and Laamanen 2014), probably resulting from uneven application of gypsum with robust machinery followed by conventional tillage, not actually homogenizing the soil. Spatial variability can be caused also by the preferential flow paths in soil, leaching S out of the soil around these cracks and macropores while S diffused into the aggregates may remain largely intact.

If 50% of the typical precipitation in the area (600 mm) is drained from the plough layer, and this water was continuously saturated with gypsum, as much as 9450 kg ha^{-1} of gypsum could be dissolved in one year, which is

2.8 times the applied amount. Even though the runoff water were not saturated with gypsum all the time, this simple calculation supports our finding that the applied amount of sulfate can be dissolved and removed from the plough layer within less than four years.

Soil amendment with gypsum was associated with elevation of EC. There was a strong correlation (Pearson correlation coefficient r = 0.82, $p < 0.001$) between the EC and the S concentration. The average EC in the 1:2.5 soil-to-water suspension before gypsum application was 0.09 dS m^{-1} and after one winter the EC in spring 2017 was 0.35 dS m^{-1} and 0.29 dS m^{-1} in clay soils and coarser mineral soils, respectively (Table 1). A large range of EC values (0.1–0.7 dS m^{-1}), even within a given field, also suggests an uneven distribution of gypsum in soil. The EC was higher in the amended than in the non-amended plots after the first ($p < 0.001$) and the second ($p = 0.004$) winter, but like S, not anymore after the fourth winter (Table S3). However, the reducing effect of gypsum on P losses lasted longer (Ekholm et al. 2022), which suggests that elevated salt concentration may not be the only decisive factor reducing P losses upon gypsum application.

Table 1. Mean and range of soil analyses before gypsum application (August 2016) and up to the fourth spring after the application in clay soils and coarser mineral soils. On clay soils, there were 9 fields amended with gypsum and 11 control fields. On coarser mineral soils, 3 fields received gypsum and 7 fields served as controls. G = Gypsum application

Variable	G	Soil type	August 2016	May 2017	May 2018	April 2020	Lieto[1]
EC dS m^{-1} 1:2.5	No	Clay	0.092 (0.03–0.22)	0.15 (0.04–0.47)	0.12 (0.04–0.33)	0.14 (0.05–0.36)	–
	Yes	Clay	0.098 (0.05–0.16)	0.35 (0.10–0.70)	0.14 (0.09–0.26)	0.15 (0.08–0.28)	–
	No	Coarse	0.058 (0.03–0.10)	0.10 (0.04–0.21)	0.071 (0.04–0.12)	0.076 (0.03–0.14)	–
	Yes	Coarse	0.078 (0.06–0.12)	0.29 (0.02–0.48)	0.14 (0.08–0.25)	0.096 (0.08–0.13)	–
S mg l^{-1}	No	Clay	8.5 (5.1–18.3)	15.1 (6.0–33)	8.5 (4.3–14.0)	7.8 (4.1–18.0)	12.4
	Yes	Clay	8.4 (5.7–16.7)	133 (8.3–371)	23.3 (5.3–65)	12.9 (3.3–87.0)	–
	No	Coarse	6.6 (4.3–8.7)	19.0 (4.5–70.8)	7.5 (4.0–12.0)	6.8 (3.7–16.0)	8.8
	Yes	Coarse	9.9 (6.0–15.7)	114 (53.8–215)	29.6 (11–74)	6.9 (5.7–8.8)	–
Ca mg l^{-1}	No	Clay	3290 (1400–5300)	3890 (1500–9400)	3210 (1200–7400)	3520 (1300–13000)	2710
	Yes	Clay	3060 (2200–5200)	3310 (2100–4500)	2850 (1800–4300)	3530 (1300–13000)	–
	No	Coarse	1890 (730–2500)	2120 (730–3100)	1710 (610–2200)	1910 (810–2500)	1840
	Yes	Coarse	2060 (1700–3000)	2560 (2400–2700)	2060 (1800–2500)	2220 (1900–3000)	–
K mg l^{-1}	No	Clay	226 (140–500)	244 (140–480)	214 (130–470)	226 (120–420)	224
	Yes	Clay	215 (110–320)	260 (130–640)	236 (110–510)	229 (120–380)	–
	No	Coarse	199 (120–290)	191 (120–310)	164 (100–250)	181 (98–250)	184
	Yes	Coarse	192 (140–250)	218 (180–270)	168 (150–190)	184 (160–210)	–
Mg mg l^{-1}	No	Clay	497 (230–1200)	548 (240–1200)	452 (220–1000)	480 (220–1100)	501
	Yes	Clay	352 (170–530)	365 (250–610)	312 (160–430)	334 (210–560)	–
	No	Coarse	249 (93–430)	277 (110–490)	231 (80–440)	263 (100–440)	255
	Yes	Coarse	154 (78–210)	166 (85–230)	129 (60–190)	146 (74–230)	–
P mg l^{-1}	No	Clay	18.1 (3.2–45.0)	17.5 (3.1–41.0)	17.8 (3.6–44.0)	16.9 (2.9–42.0)	16.8
	Yes	Clay	18.6 (4.9–39.0)	17.4 (5.0–35.0)	17.4 (4.9–34.0)	16.1 (5.0–40.0)	–
	No	Coarse	12.1 (6.0–37.0)	10.9 (5.0–28.0)	10.8 (4.7–30.0)	9.5 (4.2–24.0)	10.5
	Yes	Coarse	12.7 (9.4–15.0)	14.4 (9.2–18.0)	13.6 (9.1–17.0)	12.6 (9.2–14.0)	–
pH	No	Clay	6.6 (5.5–7.2)	6.6 (5.6–7.3)	6.6 (5.5–7.3)	6.5 (5.5–7.3)	6.2
	Yes	Clay	6.7 (6.3–7.2)	6.3 (5.6–6.9)	6.5 (5.9–7.1)	6.6 (5.5–7.5)	–
	No	Coarse	6.1 (5.5–6.6)	6.0 (5.2–6.8)	6.1 (5.5–6.9)	6.1 (5.4–6.8)	6.3
	Yes	Coarse	6.3 (5.8–6.9)	6.1 (5.6–6.5)	6.3 (5.7–6.8)	6.4 (5.8–6.9)	–

[1] Average of soil test results in Lieto in 2016–2020 (Eurofins Viljavuuspalvelu Oy 2022)

A soil is considered saline if the EC is greater than 4 dS m^{-1} in a saturated paste extract (e.g., James et al. 1982). The measured EC decreases upon increasing the amount of water that is used in the determination (i.e., widening the solution-to-soil). Equations have been developed to convert EC measured at different soil-to-solution ratios to the EC in the saturated paste extract (Richards 1954, He et al. 2012). In this study, the EC values reported in

Table 1 were measured at the soil-to-solution ratio 1:2.5. When applying the factor 0.167 presented by Richards (1954) for conversion of the EC in the saturation paste extract to values at soil-to-solution ratio 1:2, the average EC measured one winter after gypsum amendment (0.34 dS m^{-1}) corresponded to 2.0 dS m^{-1}, being below the threshold for saline soils. However, the highest value (0.67 dS m^{-1}) , measured in one subsample of a clay loam soil, corresponded to 4.2 dS m^{-1}, and the second highest value (0.52 dS m^{-1}) was equivalent to 3.1 dS m^{-1} in the saturated paste extract. The result suggest that it may be advisable not to grow crops sensitive to salinity, most of which are vegetables and fruits such as carrot, radish, onion, spinach, bean and strawberry (Maas and Hoffman 1977), in the first year following gypsum application while no adverse effects can be expected with grasses or spring-sown cereals. It may be worth testing how cereals sown in autumn right after the application of gypsum will respond.

Even though Ca was added in gypsum in still higher quantities than S, the relative change was small because of the original abundance of Ca in soil. The added amount of Ca, 370 mg l^{-1}, when evenly distributed in a 20-cm deep plough layer, corresponds to 12% and 18% of what was originally in clay and coarse mineral soils, respectively. Compared to the typical CEC of clay and coarse mineral soils, 27 and 14 cmolc kg^{-1} (Räty et al. 2021), respectively, the present Ca addition corresponded to 4.8% and 9.3%, of the CEC, respectively. The Ca concentration was higher in the first spring after gypsum amendment in the gypsum plots (contrast ratio 0.796, $p < 0.001$, Table S3). However, the finding of gypsum having an effect on Ca concentrations was blurred by the fact that Ca concentration was also increased in the control plots at the same time (contrast ratio 0.796, $p < 0.0001$). Some liming had taken place according to a farmer survey, but the data was too sparse for a quantitative analysis.

In our soil material, the Mg concentrations were at the typical level of the Lieto municipality (Table 1), and the average Mg concentration in clay soils was roughly twofold compared to coarse mineral soils. The mixed-effect model also recognized a difference between the soil types ($p < 0.001$, Table S3). The control fields, both clay and coarse mineral soils, were initially higher in Mg than those amended with gypsum (contrast ratio 1.396, $p = 0.002$). Soil concentrations of Mg did not decrease upon gypsum application, suggesting no major leaching of Mg as a response to the amendment. For unknown reasons, there was substantial annual variation in the Mg concentrations, appearing generally higher in 2017 and lower in 2018 than in 2016 ($p < 0.001$, Table S3).

The molar Ca/Mg ratio was initially lower in clay soils (5.2, range 3.4–7.8) than in coarse mineral soils (8.0, range 5.7–12.4) indicating the relative abundance of Mg in clay soils. Gypsum application increased the ratio to 5.4 (4.1–7.9) in clay soils and to 9.3 (7.1–16.4) in coarse mineral soils in the first spring after gypsum application. In clay soils, none of the soils exceeded the level of 5.4–14.2, the target range calculated from the optimal cation ranges presented by Graham (1959). In coarse mineral soils, this threshold was exceeded after gypsum application in one glacial till out of the three coarse mineral soils. The equivalent sum of Ca, K and Mg is a rough estimate of the effective CEC, since the Na content is negligible, and there is no exchangeable Al in these soils that have an almost neutral pH. On average, 17% of this sum consisted of Mg while one glacial till, mentioned above, exhibited the minimum value of 6%, which has been proposed as the minimum proportion for Mg (Graham 1959). Since this basic cations saturation concept has been strongly challenged and claimed to have little experimental proof (e.g., Kopittke and Menzies 2007), it is unlikely that gypsum application would have an adverse effect on Mg supply to the crop grown in the soils of this study. High Ca/Mg ratios and low proportions of Mg most likely occur in soils that are tested low in Mg, indicating quantitative Mg deficiency, but no such soils were found in this material. According to soil test results of 2016–2020, only less than 1% of the soil samples were tested low or rather low (the two lowest categories in the seven-step interpretation in Finland) in Mg in the Lieto municipality, while the nationwide share was 12% (Eurofins Viljavuuspalvelu 2022).

It is typical that soil texture gets finer below the plough layer and many cultivated coarse mineral soils (particularly loam and finesand soils) of southern Finland have clayey subsoils (Kähäri et al. 1987, Yli-Halla et al. 2000). Soils that may have low Mg concentrations in the plough layer commonly have higher Mg concentrations in the subsoil. However, thick sandy soils are usually poor in Mg throughout the soil profile (Yli-Halla et al. 2000). Gypsum application is not likely to decrease Mg supply to crops in clay soils but has a potential to do so in soils that have a sandy texture both in the plough layer and in the subsoil. This risk may be greatest when growing dicotyledonous plants such as clover, oilseed crops, sugar beet, fruit and most vegetables.

Concentrations of K were not affected by gypsum application. Interestingly, after two winters, the K concentrations were generally lower than in 2016 ($p = 0.002$, Table S3).

Neither were there marked changes in easily soluble P that could be attributed to gypsum application (Fig. 1a). There were bigger differences in soil P concentration between the years in fields that had a high soil test P level, usually associated with a larger difference between the two replicates. At lower soil test P levels, the concentration remained stable irrespective of gypsum application.

The average soil pH was between 6.0 and 6.7. In spring 2017, the average pH was lowered by 0.4 and 0.2 units for clay and coarser soils, respectively, in the fields amended with gypsum (Table 1). This may be attributed to the higher EC of the soil, usually resulting in a slight decrease of the measured pH(H_2O) value. The results of the mixed-effects model showed that pH was significantly higher in the clay soils than in coarser soils ($p = 0.004$, Table S3).

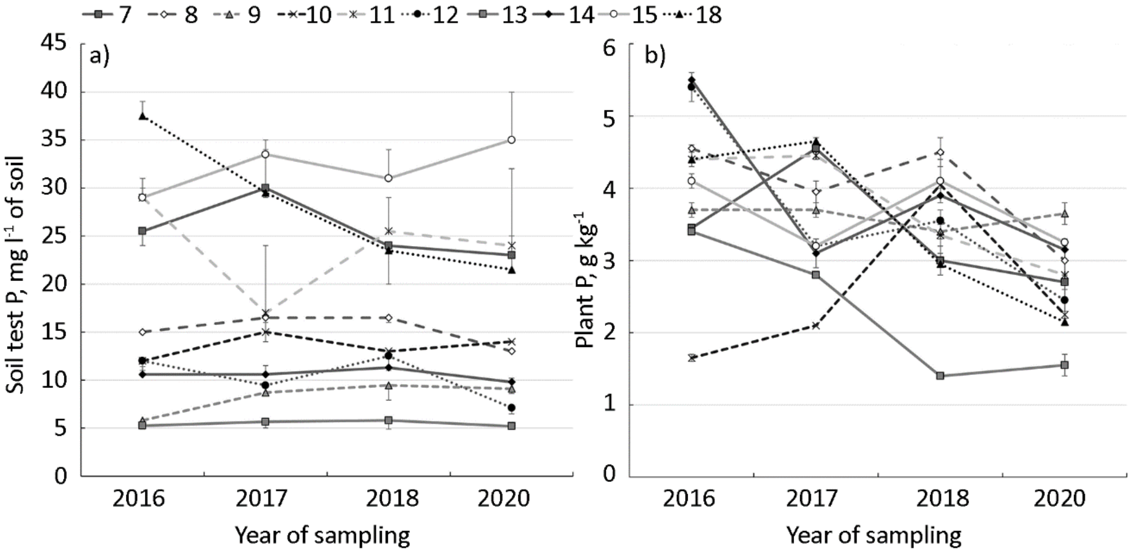

Fig. 1. Soil test P in the fields amended with gypsum (a) and plant P concentration in the same fields except field 18 that could not be sampled (b).

Changes of sulfur concentrations at different depths

The S concentration in the plough layer in spring 2017 was 11-to-12.5-fold as high as before the gypsum application in a glacial till (field 8) and clay loam soil (field 13), respectively (Fig. 2). Even during one winter of a relatively small amount of runoff (Table S1), added S had already been transported to the 20–40 cm layer, where the concentration was 4–8 times higher than initially. Even though the peak concentration decreased in the plough layer rapidly, the concentrations were substantially elevated at 20–40 cm and to some extent also at 40–60 cm at least two years after gypsum application. Sulfur appeared to be leached slightly more slowly from the clay soil than from the glacial till. While in the glacial till approximately only 3% of S added in gypsum was recovered in the 0–40 cm soil two years after the application, as much as 16% was recovered in the clay loam, and still 6% four years after the application. Because the 40–60 cm layer was not analyzed for S before gypsum application, the recovery could not be calculated at that depth.

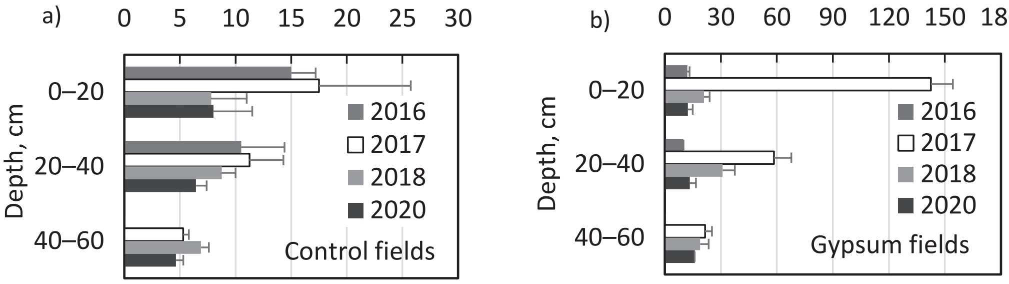

Fig. 2. Concentration of easily soluble sulfur in different layers of the control fields (a) and in the fields amended with gypsum (b). Observe the different scales of the x-axis in the two figures.

Concentrations of elements in plants

Before the gypsum amendment, the S concentration of the plants did not differ between the plots that were to be treated and the control plots (Table 2 and 3). Compared to the control plots, plant S concentration was roughly doubled upon gypsum application in the first summer after the treatment and it was still 63% higher in the second summer. The mixed-effects model results showed that the S concentration in the gypsum amended plots was significantly higher in the first (2017, $p = 0.002$) and the second summer (2018, $p = 0.021$) than in 2016, but not anymore in 2020 (Table S4), i.e., after four years' leaching and assimilation/uptake. There was also fluctuation of plant S concentration in the control plots so that, e.g., in 2016 the concentrations were lower than in 2017 but higher than in 2020 (Table 3). Plant S concentrations remaining elevated two summers after gypsum application shows that in spite of substantial S leaching out of the plough layer, part of added S had still been within the reach of the roots. The concentration of easily soluble S in soil was elevated eightfold in the first summer after gypsum application (Table 1) but the plant S concentration was only doubled (Table 2), reflecting the selective uptake of nutrients by plants. Uptake of S by the crop corresponded to about 5% of S added in gypsum, showing that the decrease of S in soil was predominantly attributable to leaching.

Gypsum amendment did not have a significant effect on plant Ca concentrations, reflecting the relatively small Ca application compared to the original stock of exchangeable Ca in soil. The Ca concentrations tended to be lower in 2020 than in other years both in gypsum amended and unamended plots (Table 2 and 3). The plant K concentration was not affected by gypsum but showed fluctuation from year to year being generally higher in 2017 than in the other years (Table S4). Gypsum application did not influence plant Mg concentrations either (Table 2), and the plant Mg concentration was not dependent on the Ca/Mg ratio in soil (Fig. S1). The efficient Mg uptake by the plants is demonstrated by the fact that in soil the average molar Ca/Mg ratio in the entire material was 5.6 while in the plants the ratio was 1.5, showing the preference of Mg over Ca.

Table 2. Concentrations of selected macronutrients in plant material collected from 10 fields amended with gypsum and from 15 control fields. The results are expressed as mean values and the range. G = Gypsum amendment. D = Concentration indicating deficiency

Variable	G	June 2016	June 2017	July 2018	June 2020	D	Typical
S	No	2.2 (1.0–3.5)	2.7 (1.3–3.9)	3.0 (0.9–10.0)	1.7 (0.8–2.5)	<0.7	1.0–1.6[3], 5[1]
g kg^{-1}	Yes	2.6 (1.0–4.5)	5.5 (2.7–10.5)	4.9 (2.3–13.0)	2.0 (1.5–2.7)		
Ca	No	3.8 (1.4–7.2)	4.5 (2.3–10)	3.6 (1.9–6.6)	3.1 (2.2–4.9)	<1.5[1]	2.6[4], 2.0–4.0[5]
g kg^{-1}	Yes	4.3 (2.5–7.4)	4.7 (2.4–6.9)	5.6 (1.5–18)	3.1 (1.6–5.6)		
K	No	26.2 (10–41.5)	31.9 (17.5–46.5)	24.0 (14–43.5)	22.8 (14.5–33.5)	<7.5[1]	23[4], 16–20[5]
g kg^{-1}	Yes	32.5 (13.5–53.5)	34.7 (16–48)	26.2 (10–37)	23.7 (15–34)		
Mg	No	1.7 (0.8–3.1)	1.7 (0.9–3.5)	1.7 (0.9–2.3)	1.5 (0.9–2.3)	<0.7	1.3[4], 0.6–1.8[5]
g kg^{-1}	Yes	1.6 (1.0–2.3)	1.5 (1.0–2.1)	1.6 (1.1–2.1)	1.4 (1.0–2.3)		
P	No	3.4 (1.5–4.9)	3.1 (2.1–5.0)	3.1 (1.9–4.7)	2.8 (1.9–4.3)	<1.5-0.24[2]	2.9[4], 1.8–2.5[5]
g kg^{-1}	Yes	4.1 (1.7–5.5)	3.6 (2.1–4.7)	3.4 (1.4–4.5)	2.7(1.6–3.7)		

[1] Youngest mature leaf (Riechelman et al. 2021); [2] Barley at tillering (Reuter and Robinson 1997); [3] Timothy hay at harvesting stage in July (Sillanpää and Jansson 1991); [4] Timothy at heading in late June (Kähäri and Nissinen 1978); [5] Timothy hay at harvesting stage in July (Jansson et al. 1985)

In all plant samples taken in 2016 before gypsum application, except one, plant P concentration was above 3.0 mg kg^{-1} (Fig. 1b), indicating abundant P supply. According to the results of the mixed-effects model, there were no significant differences in the plant P concentration associated to gypsum application. The examination of the contrast ratios of the estimated marginal means identified that plant P concentrations were higher in 2016 and 2018 than in 2020 both in amended and unamended (contrast ratios 1.237 and 1.129, $p = 0.001$ and 0.01, respectively, for both treatments) plots (Table S4). After gypsum application, most plant P concentrations were often the lowest in 2020 when, according to soil and plant S analyses, the effects of gypsum application had already been leached away (Fig. 1b). The lowest plant P concentration in 2016 (Field 10: 1.7 g kg^{-1}) was measured in perennial hay, representing an older physiological stage at sampling than the spring-sown crops where the P was not yet as diluted by the products of photosynthesis. A similar phenomenon was observed in 2017–2020 in the field that had the lowest soil test P value (Field 13), where somewhat low plant P concentrations were measured in winter wheat stands that were already at a rather mature stage at sampling. In turn, in the plant samples of fields 12 and 14 that had initially high plant P concentrations (5.4 g kg^{-1}), there was a substantial decrease in plant P concentration from 2016 to 2017. In 2016, the high P concentration was associated with high N, K, Ca, Mg and S concentrations, suggesting a presence of dicotyledonous plants. At least in one of the sites, the farmer later reported that, additional to barley recorded in the sampling notes, some white clover, inherited from previous years, had been growing in the field and may have been incorporated in the plant samples. These results do not indicate decreased plant P uptake that could be associated to gypsum application but the inconsistency of P concentrations, associated with uncontrolled variables in the survey material, call upon a systematic experiment on this issue.

Table 3. Post hoc analysis of the linear mixed effects model. Significant contrast ratios of the estimated marginal means for the concentrations of sulfur and calcium in plants in gypsum amended and unamended plots. ns. = not significant at $p < 0.05$. Contrast ratios are defined for the mean concentration in an earlier and a later year.

Variable	Gypsum	Year	2017	2018	2020
Sulfur	No	2016	0.789, $p = 0.049$	ns.	1.283, $p = 0.023$
		2017		ns.	1.628, $p < 0.001$
		2018			1.530, $p < 0.001$
Sulfur	Yes	2016	0.448, $p < 0.001$	0.517, $p < 0.001$	ns.
		2017		ns.	2.624, $p < 0.001$
		2018			2.276, $p < 0.001$
Calcium	No	2016	ns.	ns.	1.226, $p = 0.031$
		2017		ns.	1.391, $p < 0.001$
		2018			1.312, $p = 0.003$
Calcium	Yes	2016	ns.	ns.	1.226, $p = 0.031$
		2017		ns.	1.391, $p < 0.001$
		2018			1.312, $p = 0.003$

The annual changes of the average plant B, Cu, Zn, Mn and Fe concentrations (Table S5) could not be attributed to the gypsum application. The average plant Cu and Zn concentrations were the most stable over the years and the ranges were similar to those measured in other studies. The two redox sensitive micronutrients Mn and Fe showed much wider concentration ranges in plant samples with occasionally excessive values while the mean concentrations were mostly within the range usually measured in Finland. As for Mn, quite a few concentrations close to deficiency was measured every year, which, on the basis of practical farmer experience, is common in Finland. Occasional high B concentrations are attributed to pea and rapeseed that commonly receive additional doses of B compared to cereals.

The average plant Se concentration was substantially lower in the fields to be amended with gypsum already to start with as compared to the control fields ($p = 0.043$, Table S4). As much as about half of the results were below the determination limit of the analysis (0.02 mg kg^{-1}) irrespective of gypsum application, which makes it impossible to estimate the accurate mean Se concentration and the possible effect of gypsum application. Selenium fertilization was started in Finland in 1984 with the target to elevate grain Se concentration up to 0.1 mg kg^{-1} (Yläranta 1990a). The vegetative plant samples in our study to large extent had Se concentrations similar to the average Se concentration of timothy (0.007 mg kg^{-1}, Sippola 1979) before Se fertilization was started, far from the target values, which can be reached with recommended Se fertilization (Yläranta 1990a). It is likely that many fields in our study did not receive fertilizers that contain Se. This finding of many low Se concentrations is important as such and particularly so when combined with gypsum application, because sulfate additions are known to lower plant Se concentration (Yläranta 1990b). The effect of gypsum application on plant Se concentration requires further investigation, preferably in systematic experiments.

Conclusions

Application of gypsum at the rate of 4 t ha^{-1} resulted in 12–15-fold increase of easily soluble S in the plough layer at the beginning of the first growing season after the application. This relatively large change was due to the initially low level of S in soil. EC was also increased but remained lower than is harmful for plants. Concentration of Ca was not significantly elevated in soil because the added amount was relatively small compared to the native stock in soil. Changes in the concentrations of other elements, attributable to gypsum application, were not observed. Two years after the application, the plough layer still contained some more S than before the application but no effect was observed after four years. Sulfur seems to leach out slightly quicker from coarse mineral soils than from clay soils. High S concentration in soil was reflected as an elevated concentration in plants in the first and second summer after the application. Uptake of other nutrients, P and Mg in the first place, was not influenced by gypsum application. Commonly low Se content in plant samples suggests that Se fertilization was not comprehensively applied in the study area. This survey, performed predominantly on clay soils, did not detect problems of soil chemistry or plant nutrition that could be associated with gypsum application. Further research on plant nutrition is needed if gypsum application is extended to soils that are coarse-textured throughout the soil profile.

Acknowledgements

This study was part of the SAVE project (Improving the quality of the Archipelago Sea by applying gypsum to agricultural fields). We acknowledge Eliisa Punttila for organizing the soil and the plant sampling and analysis, ProAgria for taking the samples, Eurofins for the chemical analyses, Jouni Helske for help in the sampling approach, and Markku Ollikainen for initiating and participating in the SAVE project. We are grateful to the Ministry of the Environment for funding the research.

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