Received: 10 October 2022 | Revised: 13 April 2023 | Accepted: 12 May 2023

DOI: 10.1111/sum.12913

RESEARCH ARTICLE

Soil Use and Management
BRITSH SOCIETY OF SOIL SCIENCE
WILEY

Effect of calcareous and siliceous amendments on N_2O emissions of a grassland soil

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Funding information
FACCE ERA-GAS; Norges Forskningsråd

Abstract

Liming of acidic agricultural soils has been proposed as a strategy to mitigate nitrous oxide (N_2O) emissions, as increased soil pH reduces the N_2O/N_2 product ratio of denitrification. The capacity of different calcareous (calcite and dolomite) and siliceous minerals to increase soil pH and reduce N_2O emissions was assessed in a 2-year grassland field experiment. An associated pot experiment was conducted using homogenized field soils for controlling spatial soil variability. Nitrous oxide emissions were highly episodic with emission peaks in response to freezing–thawing and application of NPK fertilizer. Liming with dolomite caused a pH increase from 5.1 to 6.2 and reduced N_2O emissions by 30% and 60% after application of NPK fertilizer and freezing–thawing events, respectively. Over the course of the 2-year field trial, N_2O emissions were significantly lower in dolomite-limed than non-limed soil ($p < .05$), although this effect was variable over time. Unexpectedly, no significant reduction of N_2O emission was found in the calcite treatment, despite the largest pH increase in all tested minerals. We tentatively attribute this to increased N_2O production by overall increase in nitrogen turnover rates (both nitrification and denitrification) following rapid pH increase in the first year after liming. Siliceous materials showed little pH effect and had no significant effect on N_2O emissions probably because of their lower buffering capacity and lower cation content. In the pot experiment using soils taken from the field plots 3 years after liming and exposing them to natural freezing–thawing, both calcite ($p < .01$) and dolomite ($p < .05$) significantly reduced cumulative N_2O emission by 50% and 30%, respectively, relative to the non-limed control. These results demonstrate that the overall effect of liming is to reduce N_2O emission, although high lime doses may lead to a transiently enhanced emission.

KEYWORDS

denitrification, dolomite, grassland, liming, siliceous minerals, soil pH

1 | INTRODUCTION

Acidity is well known to increase the N_2O/N_2 product ratio of denitrification (Nömmik, 1956; Simek & Cooper, 2002; Wijler & Delwiche, 1954) and therefore raising the pH of acidic soils by liming can decrease the N_2O emission and hence serve as a N_2O mitigation tool (Hénault et al., 2019; Kunhikrishnan et al., 2016; Page et al., 2009; Russenes et al., 2016). Liming is a common agronomic practice used to counteract soil acidification by intensive cultivation, and to improve soil fertility of acidic soils (Fageria & Baligar, 2008). Most commonly used are calcareous materials (calcite and dolomite), but powdered siliceous mine tailings can be considered, as their dissolution under acidic conditions releases base cations which react with CO_2 or HCO_2^- and H^+ in the soil, thereby increasing the soil pH (Van Noort et al., 2018). Soil pH has direct and indirect effects on microbial nitrogen (N) transformations and associated production and consumption of N_2O. Directly, low pH inhibits the maturation of N_2O reductase in the periplasm (Bergaust et al., 2010). While the N_2O emission from denitrification decreases with increasing soil pH, the trend for nitrification appears to be opposite: the N_2O yield of nitrification (N_2O/NO_3^-) increases with increasing pH (Nadeem et al., 2020). Indirectly, liming may also control the potential of a soil to emit N_2O by affecting the microbial community composition over time (Braker et al., 2012; Jones et al., 2014) and, in the long run, by altering physical and chemical properties of the soil (Weil & Brady, 2017).

In intensively managed grasslands, the N_2O budget is usually dominated by fertilizer-induced N_2O emissions, often triggered by rainfall (Dobbie & Smith, 2003). Soils experiencing cycles of drying and wetting emit N_2O mainly produced by denitrification (Congreves et al., 2018). After rewetting of dry soil, a flush of microbial activity depletes O_2, thus inducing denitrification and associated N_2O emissions (Saggar et al., 2013). Likewise, in colder climates, cycles of freezing and thawing can trigger N_2O emission peaks, which may contribute up to 50% of the total annual N_2O emission (Wagner-Riddle et al., 2017; Wallman et al., 2022). Freezing–thawing induced N_2O emission peaks are commonly attributed to the release of readily degradable organic matter from died-off microbial biomass and plant residues, which may fuel both nitrification and denitrification, and lower the O_2 availability (Byers et al., 2021; Christensen & Tiedje, 1990; Sturite et al., 2021).

While increasing soil pH can be expected to decrease N_2O emissions originating from denitrification, N_2O production by nitrification may increase (Nadeem et al., 2020), thus reducing or cancelling out the mitigation effect of liming. As with denitrification, liming has direct and indirect effects on nitrification and its N_2O yield: it instantaneously accelerates the nitrification rate because of shifting the NH_4^+/NH_3 equilibrium towards NH_3, and has an indirect effect by increasing the relative activity and abundance of ammonia oxidizing bacteria (AOB) over ammonia oxidizing archaea (AOA; Hink et al., 2018). AOB produce inherently more N_2O per unit oxidized NH_3 than AOA (Hink et al., 2017). Therefore, liming may increase N_2O emissions from nitrification.

Several other factors affect N_2O emissions such as soil texture, soil organic matter content, inorganic N availability, moisture and functional microbial community composition (Abdalla et al., 2022; Wessén et al., 2011). Spatial variation of these factors may obscure the pH effect on N_2O emissions. For instance, a central factor for processes producing and consuming N_2O is soil moisture which greatly depends on local drainage conditions. Also, the trajectory of pH rise may affect N_2O processes; rapid pH rise by fast-reacting liming agents such as colloidal calcite may boost nitrification and increase available organic C, resulting in a transient increase in emission (Baggs et al., 2010). In addition, there still exists a gap in knowledge about impacts of liming on N_2O emissions from grasslands (Abdalla et al., 2022).

To expand on the 2 years of flux measurements after liming in the field experiment, we used homogenized field soils from the same liming treatments in the third year to perform a more controlled pot experiment, in which we simulated ploughing of grassland and mineral N addition, before exposing the pots to freeze–thaw conditions. Measurement campaigns were conducted both during the growing period, including application of NPK fertilizer and rain events, and during off-season in autumn and early spring to capture emission peaks triggered by freezing–thawing. We compared two calcareous and three siliceous liming treatments. Siliceous minerals were included as a possible alternative to traditional carbonates to explore the possibility for reducing N_2O emissions without liberating geochemically bound carbon as carbon dioxide (CO_2; IPCC, 2006; Wang et al., 2021). To test the pH effect on N_2O emissions with less variability in drainage conditions as compared with the experimental field, we set up a freely draining, outdoor pot experiment, using excavated and homogenized soils from the field plots.

We hypothesized that (1) N_2O emissions decrease with increasing soil pH, (2) rapid pH increase after applying finely dispersed calcite to the soil would lead to transient increase in N_2O emissions and (3) the effect of more slowly dissolving dolomite or siliceous rock powder on N_2O emissions emerges gradually over time.

2 | MATERIALS AND METHODS

2.1 | Field experiment

A field liming experiment was established in 2014 at the experimental farm of the Norwegian University of Life Sciences (NMBU) in SE Norway, Ås (59°49’ N, 10°47′ E, 75 m a.s.l). The soil is classified as an Albeluvisol (WRB, 2006; and has a clay loam texture, 27% clay, 48% silt, 25% sand – pH($CaCl_2$) = 5.0 before liming; total carbon 3%, total nitrogen content 0.28%, C:N = 11.03) and had been under a crop rotation since the 1950s (Manojlović et al., 2004). The field was last limed in the 1970s. The experimental layout consisted of five liming treatments and a control, with four replications per treatment randomly distributed in three rows. Calcareous materials were applied in autumn 2014 at rates chosen to achieve distinct pH values, whereas the mafic minerals were applied at a uniform rate of 30 t ha^{-1}. Larvikite, norite and calcite were by-product of mining industries and applied as slurries (Table 1). Dolomite was applied as granulate and olivine as a powder. To distribute the materials throughout the plough layer, liming was done in two consecutive rounds; first, half of the material was added and ploughed under to 20 cm. Thereafter, the other half was applied and harrowed to 10 cm depth.

Directly after liming in autumn 2014, winter wheat was sown. The wheat failed to establish, and the field was ploughed again in spring 2015. In early June 2015, a grass mixture (20% Timothy, 25% Perennial ryegrass, 25% Meadow fescue, 20% Tall fescue and 10% Meadow-grass) was undersown to spring barley, which served as a cover crop for grass establishment, receiving 100 kg N ha^{-1} as NPK fertilizer (YaraMila NPK, 22:3:10). The cover crop was harvested on 31st July 2015. There were no grass harvests during the establishment year 2015. Instead, aboveground biomass was cut and mulched in July and October 2015. In spring 2016, the sward was fertilized with urea-N and NPK after the first and second harvest, respectively. A third cut occurred in September.

2.2 | Pot experiment

In spring 2017, soil was excavated from 0 to 25 cm depth in each plot to set up a pot experiment. The soils were sieved (6 mm) and homogenized before placing each of them in two freely draining 9 L white plastic pots (total of 48 pots, dimensions 0.22 × 0.235 × 0.22 m, L × W × H). To provide similar drainage and packing conditions (average 7.5 kg soil dry weight per pot), a layer of gravel, isolated by a cloth (to prevent root ingrowth) was placed beneath the

TABLE 1 Liming materials: composition, producer, grain size and application rate.

Treatment	Dominant mineral[a]	Producer	Grain size[b]	Application	CCE (%)[c]
Dolomite	$Ca(Mg)CO_3$	Franzenfoss	granulated	24 t ha^{-1}[d]	109
Calcite	$CaCO_3$	Hustad marmor, Elnesvågen, Norway	Finely dispersed slurry <0.4–1.5 μm	30 t ha^{-1}	100
Larvikite	32.2% feldspar, ($Na_{0.52}K_{0.29}Ca_{0.19}Al_{1.19}Si_{2.81}O_8$), 24.5% pyroxene ($Mg_{0.24}Fe_{0.30}Ca_{0.46}SiO_3$), 20.7% biotite, 17.5% nepheline(($Na_{0.78}K_{0.22}$) $AlSiO_4$), 2.9% calcite, and 2.2% sphene	Lundhs Real Stone, Larvik, Norway	Slurry <63 μm	30 t ha^{-1}	47
Norite	65.3% plagioclase ($Na_{0.54}Ca_{0.46}Al_{1.46}Si_{2.54}O_8$), 14.4% pyroxene, 11.4% ilmenite (containing some Mg), 7.3% biotite, 1.0% magnetite, and 0.7% periclase	Titania, Hauge i Dalane, Norway	Slurry <300 μm	30 t ha^{-1}	61
Olivine	95.1% olivine ($Mg_{1.86}Fe_{0.14}SiO_4$), with 3.0% orthopyroxene and 1.9% chlorite	Silbeco, Åheim, Norway	Powder <63 μm	30 t ha^{-1}	65.7

[a] Minerology and the dissolution rates have been described in a study by Van Noort et al. (2018).

[b] Particle size as mentioned by Nadeem et al. (2020).

[c] CCE Calcium carbonate equivalent or neutralizing value of the material compared to pure (100%) of CaCO3. Calculated as a ratio of molecular weight of CaCO3 and chemical composition of liming treatments (Havlin et al., 2014).

[d] Dolomite was applied at a lower rate to adjust for its highest buffering capacity (which is 110% times of CaCO3) to achieve the same targeted pH increase pH(CaCl2) = 6.5.

soil, thus ensuring that the lower soil level would be above the drainage hole. Ryegrass was sown, to simulate forage production and the pots were kept outside, under natural conditions on white plastic sheets to prevent heating.

After establishing the sward throughout summer, the soil was removed from each pot and coarsely homogenized by hand (using a knife) before filling it back into the pot to mimic ploughing of a grass sward. Additional grass (500 g ryegrass m^{-2}) and KNO_3 (equivalent to 50 kg N ha^{-1}) was added to provoke conditions conducive to denitrification. Thereafter, the pots were placed outside and monitored for N_2O emissions (1st November–15th December; 44 days). Strong weather fluctuations led to repeated freezing and thawing of the pots. On 6st November time domain reflectometry (TDR) soil probes (Decagon, ECH2O GS3) were installed to measure soil moisture and temperature.

Overview of the field activities and experiments is presented in Table 2.

2.3 | N_2O measurements and flux calculations

2.3.1 | Field flux robot

Fluxes were measured by an autonomous field flux robot (FFR), using a fast box chamber technique (Hensen et al., 2006) with on-board gas analytics. The robot is equipped with a weatherproof instrument chamber and powered by a lithium-ion battery pack. It navigates autonomously along predefined way points by real-time kinematic (RTK) GPS, achieving centimetre-level precise positioning.

The robot is an open platform designed to carry various equipment (Molstad et al., 2014). In our experiments, it was equipped with a tunable diode laser spectrometer (DLT-100, Los Gatos Research) that measures N_2O, CO, and H_2O, and a CO_2/H_2O infrared gas analyser (LI-840A, LI-COR Inc.). The robot carries two static aluminium chambers (0.5×0.6×0.5 m, L×W×H) mounted to a 4 m wide vertical boom. The robot chambers fit the pots entirely on top of them. To deploy the chambers, the boom is lowered automatically, pressing the chambers on the soil surface. Close contact with the surface is ensured by a ring of ribbed foam rubber attached to the soil surface with a predefined pressure. An additional ring of nylon brush hairs serves as wind break.

For each measurement, the chambers were deployed for 3 min, during which gas circulated through the chambers and the optical cells of the two instruments, alternating between the left and right chambers (20 s each), thus providing data for simultaneous estimation of N_2O and CO_2 fluxes from both chambers. A sonic anemometer mounted to the robot at 2.3 m height delivered continuous data on wind speed and direction.

Leakage testing was performed by deploying chambers on a steel plate with channels (to mimic uneven soil surface and rubber ring interference) and injecting a known concentration of N_2O into both of chambers (Molstad et al., 2014). Leakage was found to be negligible, affecting flux estimated by <3% (unpublished results).

Flux calculations

N_2O concentrations measured (at ~1 Hz) during deployment were stored and postprocessed by a Python script to estimate flux rates (Molstad, 2015). For this, the first 6 s after each switch between the two chambers was omitted and a regression window of 126 s used to find the period of most linear CO_2 accumulation, which was then used to estimate the N_2O emission rate.

Raw data were plotted and inspected to identify outliers because of instrument failure, multiplexer failure, clogged tubing or actual negative fluxes. All together 5% of the measured fluxes were omitted.

N_2O fluxes were calculated based on the linear slope estimates of N_2O concentration change over time, according to equation 1:

$$F_{N_2O} = \frac{\mathrm{d}\left[\mathrm{N_2O}\right]}{\mathrm{d}t} \times \frac{V_c\, M_n}{A\, V_m} \tag{1}$$

where F_{N_2O} is the flux (μg N_2O-N m^{-2} h^{-1}), $\mathrm{d}\left[\mathrm{N_2O}\right]/\mathrm{d}t$ is the rate of N_2O concentration change in the chamber over time (ppmv h^{-1}), V_c is the chamber volume (L), A is the area covered by the chamber (m^2), M_n is the molecular mass of N in N_2O (g mol^{-1}) and V_m is the molar volume (L mol^{-1}) at chamber temperature (Tan et al., 2009). $V_m$was calculated according to the ideal gas law:

$$V_m = R\,\frac{T_c + 273.15}{P} \tag{2}$$

where R is the ideal gas constant, T_c is the temperature in degrees Celsius and P is the pressure. Given the short deployment time, chamber temperature was assumed to be equal to air temperature, which was taken from downscaled meteorological data closest in time to the recorded flux estimate (Institute, T. N. M, 2015–2016). For the pot experiment, Equation 1 was modified to take account for the pot volume and the surface area of the pots.

Quality-checked data were used to calculate cumulative fluxes by linear interpolation (i.e. trapezoidal integration) in R studio, where values were sorted according to time order and aggregated to a cumulative flux (gasfluxes package version 0.4–1), R software 4.1.

TABLE 2 Field activities, management inputs and event dates for the field and pot experiment.

		Field activities
Field experiment date		
October	2014	The field was limed with 30 t ha^{-1} calcite, norite, larvikite and olivine. Dolomite was applied at a rate of 24 t ha^{-1}. During the same month winter wheat was sown.
18th May	2015	Field was ploughed with crop residues of winter wheat that failed to establish.
29th May	2015	A grass mixture was under sown to spring barley. Field was fertilized on the same date with 100 kg ha^{-1} NPK-N (YaraMila NPK, 22:2:10).
15th July	2015	*N_2O measuring campaign begins for the year 2015.*
31st July	2015	Aboveground biomass was cut and mulched.
10th October	2015	Aboveground biomass was cut and mulched.
21st October	2015	Application of NPK fertilizer with 100 kg ha^{-1} NPK-N (YaraMila NPK, 22:2:10).
15th December	2015	*N_2O measuring campaign ends for the year 2015.*
14th March	2016	*N_2O measuring campaign begins for the year 2016.*
6th May	2016	Application of NPK fertilizer with urea, 120 kg ha^{-1} N.
9th June	2016	First harvest. Application of NPK fertilizer with 90 kg ha^{-1} NPK-N (YaraMila NPK, 22:2:10).
19th July	2016	Second harvest.
22nd July	2016	Application of NPK fertilizer with 60 kg ha^{-1} NPK-N (YaraMila NPK, 22:2:10).
8th September	2016	Third harvest.
5th November	2016	*N_2O measuring campaign ends for the year 2016.*
Pot experiment date		
Spring	2017	Soil excavation from the field and setting up of the pot experiment. Ryegrass was sown to simulate forage production.
2nd November	2017	Soil was taken out of the pots, re-homogenized, mixed with 50 kg ha^{-1} N as KNO_3 and 500 g m^{-2} dry ryegrass, Densely packed back into the pots and left out in the field.
3rd November	2017	*N_2O measuring campaign begins for the year 2017.*
6th November	2017	TDR soil probes were installed in the pot experiment.
15th December	2017	*N_2O measuring campaign ends for the year 2017.*

2.4 | Soil variables

Soil temperature and volumetric water content were measured by TDR probes (Decagon, ECH2O GS3, 0–10 cm depth, 70-MHz frequency), which were placed in the south and north part of the field. Water filled pore space (WFPS) was calculated as the ratio of volumetric soil water content to total soil porosity, as inferred from the field's average bulk density (Paul, 2015; Equation 3).

$$\%\,\text{WFPS} = \frac{\text{soil water content x 100}}{1 - \frac{\text{bulk density}}{2.65\ \text{g}/\text{cm}^3}} \tag{3}$$

The probes were removed during application of NPK fertilizer, harvests and sowing.

Total carbon content of soil was determined according to the dry combustion method as described in Nelson and Sommers (1983). Soil samples were crushed by a mortar before weighing and analysing them on a Leco CHN628 element analyser. The samples were dried at 55°C prior to analysis to remove residual moisture.

In 2013 prior to liming, soil samples were analysed for texture by a Beckman Coulter LS 13320, particle size analyser, measuring the size distribution of the particles suspended in a liquid (Aqueous Liquid Module, ALM) after removing all organic material by H_2O_2 according to the method of Pye and Blott (2004). Nine samples were analysed by the pipette method (Gee & Bauder, 1986) to calibrate the particle counter.

Throughout 2015 and 2016, composite samples for each plot were sampled twice or once a month for measuring pH both in water and 0.01 M $CaCl_2$ using a Thermo Scientific ROSS Ultra pH/ATC Triode electrode.

Soil was sampled prior to and after application of NPK fertilizer events (0–10 cm depth) for mineral nitrogen analysis. Twenty-five grams of fresh soil were extracted with 40 mL 1 M KCl. Subsamples of 1 mL were frozen for later analysis. The subsamples were thawed and centrifuged at

10,000*g* for 15 min at 4°C. NO_3^- and NO_2^- concentrations were determined by Griess reaction with Vanadium (III) chloride (Doane & Horwáth, 2003) using a microplate reader (Infinite F50, TECAN Austria GmbH) at 540 nm. NH_4^+ was analysed calorimetrically by the sodium salicylate method at 660 nm (Keeney & Nelson, 1982), using the same microplate reader (Infinite F50, TECAN Austria GmbH).

Soil texture, total carbon and total nitrogen were provided by Bleken (M. Bleken, personal communication).

2.5 | Ancillary variables

Precipitation and air temperature data were obtained from the nearby (~1 km to the east on a comparable field) NMBU weather station that is part of the Norwegian meteorological network (MET, 2015–2016).

2.6 | Statistics

All flux rates were tested for assumptions of linear relationship and gamma distribution of the responses. A generalized additive linear mixed model was used to test for treatments (fixed effects, six levels) on hourly flux estimates N_2O emissions using glmer function, 4.2.2 package lme4 (Bates et al., 2014). Temporal variation in N_2O emissions (μg N_2O-N m^2 h^{-1}) between 24 different plots and between the 209 different sampling days was modelled by means of random effects. To avoid negative values of the responses a linear shift was performed by adding 0.000001 to negative values (5% of data). The same model was used to test differences between liming treatments in the pot experiment. The proportion of the random effect variance attributable to each random effect was found by dividing each random effect by the sum of all random effects (i.e. the total variance of the random effects).

Differences in soil pH were tested with a general linear model (GLM), 4.0.2 package MASS (Venables & Ripley, 2002) with soil pH as response variable and liming treatments as predictor. The pH effect on cumulative N_2O emissions was tested with ANOVA factorial model using the ANOVA function in R.

All statistics were performed by R software, 4.2.2. version.

3 | RESULTS

3.1 | Soil pH

Liming with calcite ($p < .01$) and dolomite ($p < .01$) in 2014 resulted in significantly higher pH values in 2015 compared to the control (Figure 1). Soil treated with silicious minerals in 2015 showed no increase in pH compared to control (pH 5.5). Except for the calcite treatment, soil pH increased throughout March to May, peaking shortly after application of NPK fertilizer (18th May) for all treatments. In late May, olivine started to separate from this grouping with slightly higher pH but decreased again during June and July. During the rest of the year 2015 there was a trend of decreasing pH in all treatments.

In May 2016 soil pH was similar to the last pH measurement in October 2015 and there was a positive trend over the year, but not significant (Figure 1). In May 2016, there was on average half a pH unit decrease in norite, control, larvikite and olivine between 17th April (before application of NPK fertilizer with urea) and 20th May (after application of NPK fertilizer with urea). During this time dolomite decreased soil pH by almost one pH unit (approx. 0.8), while calcite remained stable. Later in July mafic minerals and dolomite returned to their pre-urea application of NPK fertilizer pH values. In September dolomite, calcite and olivine increased pH, while norite, larvikite and control were stable, showing similar values as in July.

3.2 | N_2O emissions

3.2.1 | Field experiment

Over the course of the 2-year field experiment, N_2O emissions showed distinct emission peaks (Figure 2a), which were related to harvesting (31st July 2015 and 14th June 2016), application of NPK fertilizer (22nd October 2015, 10th May and 14th June 2016) and freezing and thawing events (23rd–22nd November 2015 and 16th–30th March 2016). The first peak in 2015 was observed after harvesting the cover crop (31st July 2015) coinciding with increasing WFPS because of abundant rain (Figure 2b). Another pronounced emission peak in 2015 was observed after mulching of the grass sward on 10th October 2015 and application of NPK fertilizer on 20th October which coincided with a period of diurnal freeze–thaw cycles, eliciting a large N_2O emission peak.

Application of NPK fertilizer with urea increased N_2O emissions in early May 2016 and even more in June 2016 (after first harvest). During the latter event the highest emission peak was recorded for both years (~1600 μg N_2O-N m^2 day^{-1}). This peak was triggered by rainfall after application of NPK fertilizer. Application of NPK fertilizer after the second harvest did not result in any distinct emission peaks.

Emission peaks at the end of November 2015 coincided with some rain and decreasing soil temperatures,

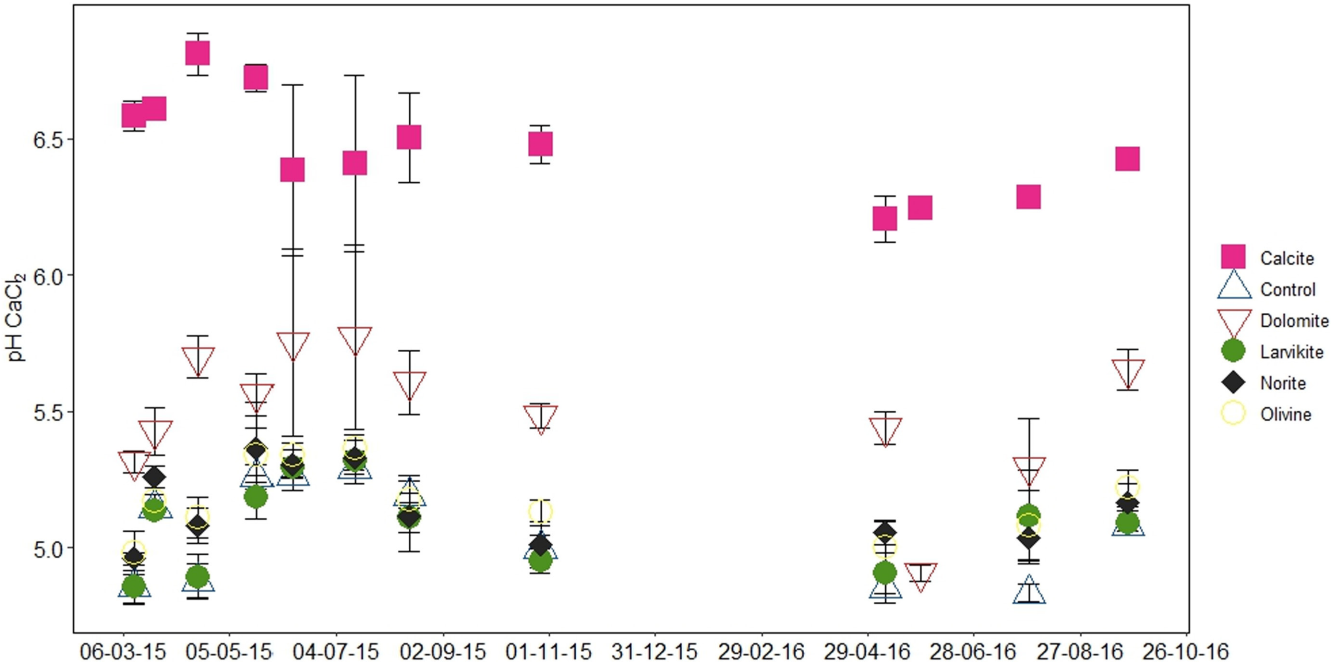

FIGURE 1 Average topsoil (0–15 cm) pH_{CaCl2} (±SE) from March 2015 to October 2016 for all treatments. $N = 4$ for each treatment and sampling date.

followed by two smaller emission peaks triggered by freezing and thawing cycles (highest peak ~900 µg N_2O-N m^2 day^{-1}). During snowmelt and spring thaw in March 2016, N_2O emissions strongly fluctuated with peak emissions of up to ~1600 µg N_2O-N m^2 day^{-1}, which is approximately 600 µg N_2O-N m^2 day^{-1} higher than the highest emission peak observed in autumn 2015 (Figure 2a). By contrast, highest recorded emission for the first freezing and thawing event in autumn 2016 did not exceed 1000 µg m^2 day^{-1} N_2O-N.

3.2.2 | Treatment effects

There was no overall significant ($p = .54$) relationship between N_2O emissions and soil pH for the field trial based on cumulative N_2O emissions. However, N_2O emissions in the dolomite treatment were significantly lower than in the control ($p < .05$; Table 3), while no other liming treatment affected N_2O emissions significantly. Most of the variation seen in N_2O emissions was left unexplained (47%). A large part of the variation was left explained by day-to-day flux dynamics (day number, 45% of the variation), and plot number explained only 8% of the variation in N_2O emissions.

N_2O cumulative emissions across seasons revealed that dolomite had generally smaller N_2O emissions compared to the control (Figures 3a-c), although this was not statistically significant. Calcite, which was the treatment with the highest pH raise (Figure 1) appeared to emit less N_2O than the control in the first growing season (Figure 3a), but this difference was not significant because of the high variability of emission fluxes in the control. In the following seasons, the average cumulative N_2O emission of the calcite treatment was numerically almost equal that of the control.

Overall, silicious minerals had no significant effect on N_2O emissions (Tables 3 and 4). Olivine had smaller, though not statistically significant, emissions than the control in the first summer, but this effect disappeared throughout the following seasons. (Figure 3). Larvikite and norite had variable effects on N_2O emissions. Growing season emissions in 2016 did not show any treatment effect (Figure 3d).

3.3 | Pot experiment

In the pot experiment, frequent freezing and thawing triggered exceptionally high emission rates (Figure 4a). Cumulated over the entire experimental period (November–December 2017) N_2O emissions were strongly affected by pH (Figure 4) with calcite ($p < .01$) and dolomite ($p < .05$) having 50% and 30%, respectively, smaller emissions than the control (Figure 5).

The variation in N_2O emissions explained by treatment was by approximately 17% higher in the pot experiment than in the field experiment (Tables 3 and 4). Day-to-day flux variation (day number) explained 70%, plot number 0% and unexplained variation was 30%. It is important to

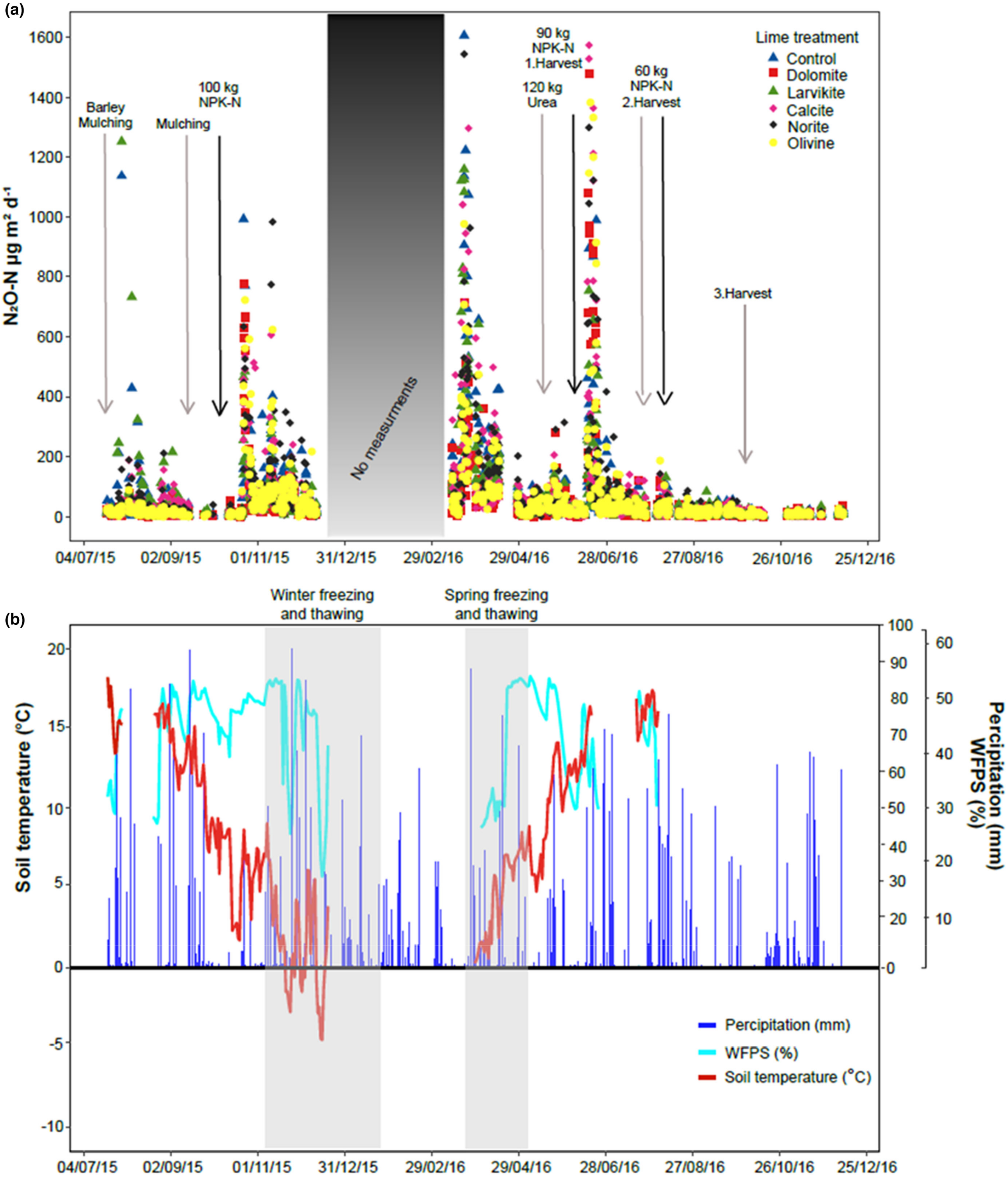

FIGURE 2 Mean N_2O emission in the field experiment (in µg N m^2 day^{-1}) in 2015 (July to December) and 2016 (March to December) (a); for clarity, no standard deviations are shown; with no significant differences in emission rates. (b) Average daily soil temperature (0–10 cm depth), water filled pore space (%) and daily precipitation in mm.

note that the field experiment consisted of 209 sampling dates while the pot experiment was sampled in only 44 dates, which inherently reduces day-to-day variation and overall unexplained variability (error).

4 | DISCUSSION

Based on previous laboratory studies of denitrification in soil (Simek & Cooper, 2002; Wijler & Delwiche, 1954), and

TABLE 3 Parameter estimates of a linear mixed effects model for mean hourly N_2O emissions (µg N_2O-N m^2 h^{-1}) in the field experiment for fixed (treatment) and random effects (plots, day number).

2015–2016				
Fixed effects treatment	**Estimate (µg N_2O-N m^2 h^{-1})**	**N_2O-N emissions, *p* value ($p < .05$)**	**Random effects**	**Variance**
Dolomite	−0.24 ± 0.11	.01	Day number	0.32
Larvikite	−0.00 ± 0.10	.99	Plot number	0.01
Calcite	−0.01 ± 0.01	.87	Residuals	0.35
Norite	0.04 ± 001	.63		
Olivine	−0.11 ± 0. 10	.23		

Note: *p* values indicate differences between treatments and control (not limed).

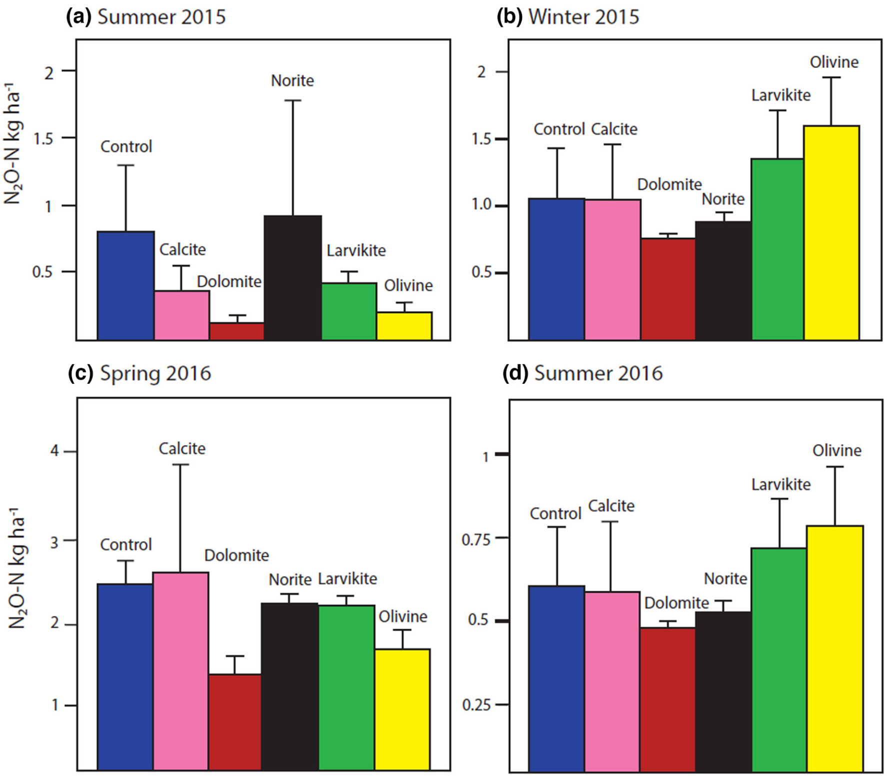

FIGURE 3 Cumulative N_2O emissions (N kg ha^{-1} $period^{-1}$) in the field experiment: (a) growing period 2015 (20th July–14th September 2015); (b) Fall 2015 (20th October–20th November 2015); (c) Spring thaw 2016 (14th March–15th April 2016); (d) Growing season 2016 (20th July–15th September 2016). Error bars are standard error ($N = 4$).

subsequent documentation that the maturation of N_2O reductase is hindered by low pH (Bergaust et al., 2010; Liu et al., 2014) we hypothesized that N_2O emissions would be negatively related with soil pH. Such a negative relationship has also been documented for field fluxes in a meta study by Wang et al. (2018). In a review paper by Abdalla et al. (2022), in 4 out of 10 field studies N_2O emissions decreased after increased pH by liming. However, under field conditions, the pH effect may be overridden by other factors. Particularly under conditions of shifting O_2 availability, the well-documented suppression of N_2O emission from denitrification by pH rise may be cancelled out by increased N_2O emissions from nitrification. Nadeem et al. (2020), using soils from the same field plots in a laboratory study, demonstrated that, after NH_4^+ addition, soil moisture played a key role for the overall pH effect on N_2O emissions; at lower soil moistures, nitrification prevailed and N_2O emission was positively correlated with soil pH, while at higher soil moistures, O_2 consumption by nitrification induced coupled nitrification–denitrification, the N_2O production of which was negatively correlated with soil pH. Under field conditions, a multitude of factors determines O_2 availability in the soil (root respiration, C availability, NH_4^+ availability, soil moisture), which makes it difficult to predict dynamics of nitrification and denitrification in situ. Next to the temporal dynamics of nitrification and denitrification, their relative importance may show considerable spatial variation, which compromises the statistical power of plot trials. In the present study, large differences in soil texture and hence aeration conditions, were detected across the field plots (Supplementary data, Figure S1). This may explain the large standard errors of cumulative N_2O emissions we found, for instance, in the unlimed control (Figure 3) and the overall low explanatory power of treatment in the mixed effect models (Tables 3 and 4). The high spatial variability of N_2O fluxes in the field trial prompted us to conduct a more controlled pot experiment, in which we attempted to equalize drainage conditions by letting the pots freely drain and, in addition, stimulated denitrification by mixing in grass litter and NO_3^-. Exposing the pots to natural freezing–thawing cycles elicited vigorous N_2O emissions which, when cumulated, showed a pH-proportional response with significantly less N_2O emissions the higher the pH (Figure 5). This corroborated that liming has a direct effect on N_2O emissions when originating from denitrification.

Another critical factor for the effect of liming on N_2O emissions may be the trajectory of pH rise. While granulated dolomite dissolved slowly, the finely dispersed calcite slurry used in our study instantly raised the pH from pH 5.00 to pH 6.62 (Figure 1). Rapid pH raise has multiple effects in soil, among them release of adsorbed DOC to the soil solution, increased microbial activity and, most notably, a strong stimulation of nitrification (Li et al., 2020). Overshooting NH_3 oxidation may result in transient nitrite (NO_2^-) accumulation as shown for our soils in the laboratory experiment by Nadeem et al. (2020). Nitrite accumulation, in turn, may induce additional N_2O production by chemical hydroxylamine oxidation (Liu et al., 2019), hybrid N_2O formation (Terada et al., 2017) or nitrifier denitrification (Kremen et al., 2005). In our field experiment, calcite addition caused variable effects resulting in large standard errors and non-significant differences to the control. We therefore conclude that rapid pH raise by calcite slurries is not suitable for mitigating N_2O emissions, most likely because pH raise perturbs N cycling in general, and nitrification in particular.

It is noteworthy that liming did not affect N_2O emissions during freezing–thawing cycles in autumn 2015 (shortly after mineral N addition) nor in spring 2016 (spring thaw). A possible explanation for the lack of a pH effect on N_2O emissions during freezing–thawing may be strongly reductive conditions in partly frozen soils (Öquist et al., 2004). As shown by Byers et al. (2021), N_2O lingers in the subsoil for extended periods while pO_2 declines and also de novo production of N_2O during thawing often occurs when soils are fully saturated. Under these conditions, N_2O reductase may be induced unconditionally because of a lack of electron acceptors, thus overriding the effect of pH on denitrification product stoichiometry. Interestingly, Russenes et al. (2016) working in a wheat stubble field in SE Norway found a negative relationship between N_2O emissions and naturally occurring pH variation during spring thaw, probably reflecting less reductive conditions in a wheat stubble as compared to a grass ley. More research is needed to elucidate whether pH interactions with non-growing season N_2O processes differ in annual and perennial cropping systems.

Contrary to our field experiment, we found a significant negative relationship between soil pH and N_2O in the pot experiment (Figure 4). This was partly because allowing for free drainage removed some of the intra-treatment variability observed in the field experiment as can be seen from a decrease in the proportion of variance explained by plot number between the pot and field experiment, 8% and 0%, respectively. More importantly, there was a clear pH effect because addition of plant litter and nitrate and exposure to freezing–thawing triggered denitrification as the dominant N_2O source. This confirms that the recurrently reported observation of a liming effect on denitrification product stoichiometry can be reproduced under near-field conditions.

Despite the fact that N_2O field emissions did not scale proportionally with soil pH, we found that dolomite

TABLE 4 Parameter estimates of a linear mixed effects model for mean hourly N_2O emissions (µg N_2O-N m^2 h^{-1}) in the pot experiment for fixed (all treatments) and random effects (plots, day number).

Pot experiment				
1st November–15th December 2017				
Fixed effects Treatment	**Estimate (µg N_2O-N m^2 h^{-1})**	**N_2O-N emissions *p* value ($p < .05$)**	**Random effects**	**Variance**
Dolomite	−0.42 ± 0.13	<.01	Day number	0.79
Larvikite	−0.12 ± 0.13	.33	Plot number	0.01
Calcite	−0.71 ± 0. 13	<.01	Residual	0.33
Norite	−0.02 ± 0.13	.91		
Olivine	−0.08 ± 0.13	.51		

Note: *p* values indicate differences between treatments and control (not limed).

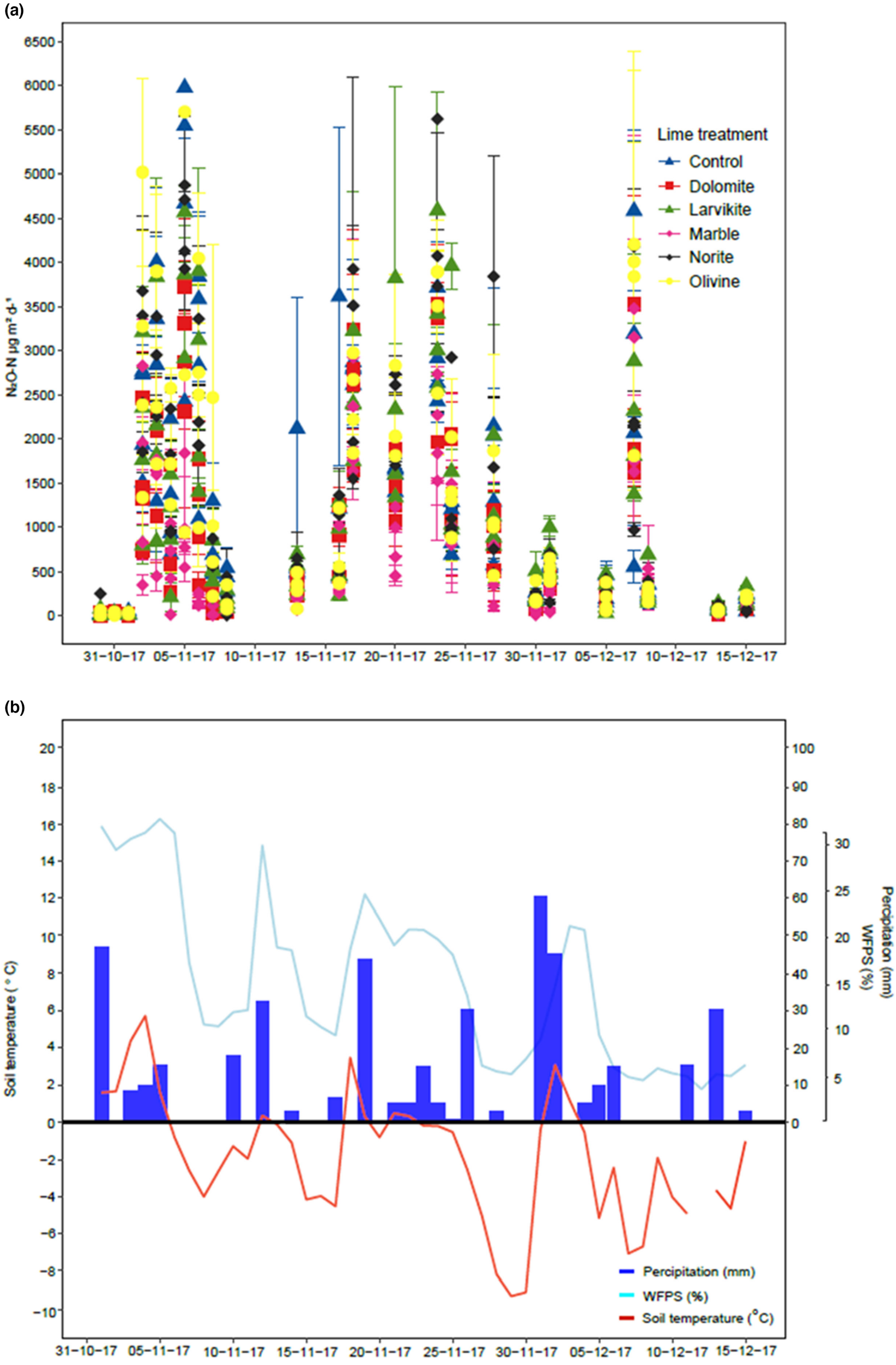
(a)
N₂O-N µg m² d⁻¹
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
31-10-17
05-11-17
10-11-17
15-11-17
20-11-17
25-11-17
30-11-17
05-12-17
10-12-17
15-12-17
Lime treatment
Control
Dolomite
Larvikite
Marble
Norite
Olivine
(b)
Soil temperature (°C)
20
18
16
14
12
10
8
6
4
2
0
−2
−4
−6
−8
−10
100
90
80
70
60
50
40
30
20
10
0
30
25
20
15
10
5
Percipitation (mm)
WFPS (%)
Percipitation (mm)
WFPS (%)
Soil temperature (°C)
31-10-17
05-11-17
10-11-17
15-11-17
20-11-17
25-11-17
30-11-17
05-12-17
10-12-17
15-12-17

FIGURE 4 Mean daily N_2O emission in the pot experiment (in µg N m^2 day^{-1}) in 2017 (31st October–15th December) (a); for clarity, no standard deviations are shown; with no significant differences in emission rates. (b) Average daily soil temperature (0–10 cm depth), water filled pore space (%) and daily precipitation in mm.

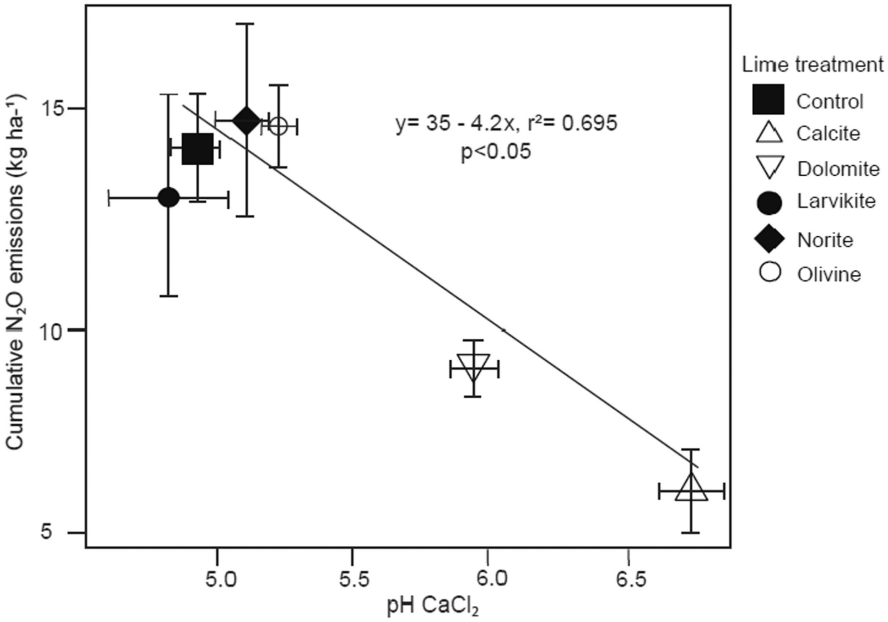

FIGURE 5 Relationship between cumulative N_2O emissions and measured soil pH in the pot trial. Shown are average cumulative N_2O-N fluxes in kg ha^{-1} over a period of 44 days (1st November–15th December 2017) plotted over average soil pH in 0.01 M $CaCl_2$ with standard errors for both variables.

reduced N_2O emissions in all application of NPK fertilizer events by on average 30% (Figure 1a, four application of NPK fertilizer events total) which is in accordance with results of Hénault et al. (2019) who found that liming of acidic soils to neutrality reduces N_2O emissions after fertilizing events. However, this application of NPK fertilizer event coincided with an autumn freezing and thawing event (Figure 1) and therefore it is hard to conclude if the dolomite led to reduced N_2O emissions derived from application of NPK fertilizer and/or freezing–thawing event. Nevertheless, dolomite successfully reduced fertilization induced emissions and we believe these emissions were predominantly denitrification induced, because of a combination of rain and NH_4^+ induced O_2 consumption.

Overall, we conclude there was no liming effect on field N_2O emissions. Temporal variations of N_2O emissions in the field experiment seem to have been more influenced by field heterogeneity, climate and management events (Figure 2). Siliceous minerals did not show promising results as an alternative to traditional limes (calcite and dolomite). As such, we do not recommend liming as a management option to mitigate soil emissions. However, we conclude this based on two and a half year N_2O measurement campaign without investigations on the soil CO_2 emissions and soil organic carbon changes. Liming is an important and necessary soil management measure to ensure optimal soil health and crop productivity. Thus, liming should be applied sporadically but in smaller doses and not to achieve pH > 6.5 as it may lead to enhanced emissions.

ACKNOWLEDGEMENTS

We thank Trygve Fredriksen and Øyvind Peder Vartdal for their help with field and laboratory work. This research was financed by Norwegian Research Council project number 234382 (MIGMIN) and the FACCE ERA-GAS project, MAGGE pH under Grant Agreement No. 696356.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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How to cite this article: Vekic, T. T., Nadeem, S., Molstad, L., Martinsen, V., Hiis, E. G., Bakken, L., Rütting, T., Klemedtsson, L., & Dörsch, P. (2023). Effect of calcareous and siliceous amendments on N_2O emissions of a grassland soil. *Soil Use and Management*, *39*, 1082–1095. https://doi.org/10.1111/sum.12913