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Nitrous oxide emissions from red clover and winter wheat residues depend on interacting effects of distribution, soil N availability and moisture level

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Abstract

Aim The effects of residue type and distribution, soil moisture and NO_3^- availability were investigated in 43 days laboratory incubations (15 °C) on emissions of N₂O, CO₂, and for some treatments NO and NH₃.

Methods Two crop residues were considered (red clover, RC, and winter wheat, WW), and they were either mixed with topsoil, placed as a discrete layer in soil, or no addition. Soil NO_3^- was either at ambient level or increased. Water filled pore space (WFPS) was adjusted to either 40 or 60%. All treatments were analysed for mineral N, N₂O and CO₂ with manual sampling and gas chromatography. Selected treatments were analysed with a continuous-flow method of N₂O and CO₂ by laser spectroscopy, NO by photoluminescence and NH₃ by acid traps.

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Results The NH_3 and NO emissions was higher in mixed RC than control and WW treatment. The N_2O emission was many-fold higher with mixed than layered distribution, but only with high soil NO_3^- availability and high soil moisture. Emissions of N_2O from WW were an order of magnitude lower compared to RC, and decomposition was slower. Both batch and continuous-flow incubations resulted in similar emissions. Disregarding the extreme emissions in the high WFPS and NO_3^- treatment, the N_2O emission factors averaged 0.3 and 0.6% of residue N for WW and RC, respectively.

Conclusion Residue decomposition was enhanced by mixing, and N_2O emissions by higher soil water and NO_3^- content. The results show the importance of residue distribution and soil condition on estimating N_2O emission factors for crops.

Keywords Greenhouse gas emissions · Laboratory incubation · Crop residue quality · Soil nitrate · Incorporation method

Introduction

Each year, one billion metric tonnes of crop residues are produced in agricultural systems (Blanco-Canqui and Lal 2009). Recycling of residues to the soil by incorporation is an accepted way to maintain soil fertility, increase crop productivity, and help maintain soil carbon (C) stocks (Lehtinen et al. 2014; Lugato et al. 2014; Velthof et al. 2002). Decomposing crop residues serve as an energy provider and source of both C and nitrogen (N) for soil organisms. However, residue decomposition may also lead to environmental N losses depending on residue quality and local soil and environmental conditions (Baggs et al. 2000).

Nitrous oxide (N_2O) is a potent greenhouse gas and predicted to become the dominant ozone (O_3) depleting substance of the twenty-first century (IPCC 2013). Like N_2O , nitric oxide (NO) is a precursor of both tropospheric O₃ and nitric acid (HNO₃), a major component of acid deposition (Pilegaard 2013). Ammonia volatilisation from crop residues can also be significant depending on chemical composition and management (Xia et al. 2018). Incorporation of crop residues can, compared to surface application, reduce NH₃ losses, but will increase soil water holding capacity and oxygen (O_2) demand locally, which may fuel the development of anaerobic microsites as a result of intense microbial activity (Kuzyakov and Blagodatskaya 2015; Kravchenko et al. 2018; Kim et al. 2012).

Residue composition can vary with crop type and, for example, C:N ratios of crop residues range from <10 in leguminous crops used as green manure (Li et al. 2015) to 80 and higher in cereal straw (Robertson and Groffman 2007). Accordingly, the N mineralization potentials vary greatly (Li et al. 2020), and immobilization of inorganic N can occur at high C:N ratios (e.g. Yao et al. 2017). Ammonium (NH₄⁺) is the preferred N source for microbial growth, but also a substrate for autotrophic nitrification and subsequent denitrification, and hence there is competition for substrate between assimilatory and dissimilatory processes that can determine the fate of residue N (Burger and Jackson 2003).

Chen et al. (2013) observed that N_2O emissions from crop residues were less variable than those from synthetic fertilisers. This is because decomposer activity can lead to O_2 depletion around residue particles independent of soil moisture conditions (Li et al. 2016; Duan et al. 2017), and this oxic-anoxic gradient environment is favorable for the production of N_2O through coupled nitrifier and denitrifier activity (Chen et al. 2013; Rees et al. 2013). The presence of synthetic N fertilisers can significantly enhance N_2O emissions during crop residue decomposition by alleviating N limitations during microbial decomposition (Chen et al. 2013), and presumably this is more important for residues with a low net N mineralization potential. Since N_2O is an intermediate of denitrification, it follows that poor aeration and high decomposer activity may favor complete denitrification, as seen when straw was added to rice paddy soil (Ma et al. 2007; Yao et al. 2017), and such interactions further complicate the prediction of N_2O emissions.

Residue distribution, e.g., whether residues are left at the soil surface or mixed into the top soil by tillage, will influence gas and solute exchange between residues and soil (Angers and Recous 1997; Justes et al. 2009). Therefore, tillage or its absence also affects the magnitude of soil N2O emissions following residue management (Muhammad et al. 2019). Better distribution of residues, e.g. by rotovation or shallow tillage, will change the rate of decomposition (Loecke and Robertson 2009) and shift the balance towards aerobic decomposition compared to the more discrete distribution of residues achieved by inversion tillage. Leaving residues at the soil surface, with even less residue-soil contact, has been found to reduce N₂O emission when compared with incorporation (Muhammad et al. 2019). Hence, the magnitude and temporal dynamics of N₂O emissions are expected to vary depending on residue distribution and soil characteristics (Kravchenko et al. 2017).

Following guidelines from the Intergovernmental Panel on Climate Change (IPCC 2006), derived N₂O-N emissions from soil amendments are calculated by subtracting background emissions from an unamended control. Nitrous oxide emission factors express N₂O-N as percentage of the N added, and hence N content is the only characteristic of crop residues considered. National inventories of agricultural GHG emissions mostly rely on a default emission factor of 1% for calculating N_2O-N from the N in residues (IPCC 2006). In a refinement of the guidelines, IPCC (2019) suggested a change to 0.5 and 0.6%, respectively, for dry and wet climates. Yet, experimental studies show a much wider range of N₂O emission factors from crop residues (Chen et al. 2013; Jungkunst et al. 2006; Kravchenko et al. 2018), which indicates that factors other than N input should be considered to improve the reliability of emission estimates. Besides residue quality, this could include soil type, N availability, climate and management practices.

In this study, we examined effects of residue type and distribution, soil moisture, and soil NO₃⁻ availability on short-term emissions of N₂O and CO₂, and in some cases also on emissions of NO and NH₃. A standardised laboratory setup with manual flux measurements investigated treatment effects using a factorial experimental design. The temporal dynamics were examined in more detail with high-resolution automated flux measurements for selected treatment combinations. We hypothesized that: 1) a high residue C:N ratio would have lower N₂O emissions compared to a residue with low C:N ratio, especially when soil NO3⁻ availability was low; 2) 60% water-filled pore space (WFPS) would enhance emissions compared to 40% WFPS; 3) a concentrated distribution of residues would increase the magnitude and temporal stability of N₂O emissions, especially when residue C:N ratio was low.

Methodology

Soil

In spring 2018, soil was collected at the depth of 0–20 cm from the B-C blocks in the SITES Agroecological Field Experiment at Lönnstorp Field Station (55° 39′ 58.5072" N, 13° 6′ 57.0636″ E) belonging to the Department of Biosystems and Technology of the Swedish University of Agricultural Sciences (SLU). The selected field was under red beets (*Beta vulgaris* L.) in the previous year, and no cover crop was grown during winter. Any seedlings appearing after sampling were removed immediately. The soil was partially dried (to approximately 0.15 g H₂O g⁻¹ dw), sieved to <6 mm, subsampled for analysis of texture, total C, total N, and pH; and frozen at -20 °C. Soil was packed with insulating material before shipment to Denmark or Germany. The physicochemical characteristics of the soil are presented in Table 1.

Crop residues

The residues used for the incubation experiments were aboveground plant parts such as stems or leaves, which are typically left on the field, incorporated to varying degree depending on tillage practice, or fully removed.

Aboveground biomass of red clover (*Trifolium* pratense L.) was obtained from a long-term PK trial at the Norwegian University of Life Sciences (NMBU), 59° 39′ 45″ N 10° 45′ 46″ E (Byers et al. 2021). The red clover (RC) residues contained 445 g C and 25 g N kg⁻¹ DM, and they were dried to constant weight at 40 °C, and then cut to 1 cm length for the incubation experiment. Different fractions of RC were mixed in proportions similar to their occurrence

Table 1 Physico-chemical characteristics of SLU soil (left), and crop residues characteristics (right)

	Crop properties	Red clover	Winter wheat
158	C-content (g C 100 g^{-1} DM)	44.5	46.6
122	N-content (g N 100 g^{-1} DM)	2.5	0.5
102	C:N	17.9	90.9
307	Humidity (WM DM ⁻¹)	6.5	11.9
311	$NO_3^{-}-N (g N 100 g^{-1} DM)$	0.0	0.0
1.49	$NH_4^+-N (g N 100 g^{-1} DM)$	0.0	0.0
15	Water soluble carbohydrates (g C 100 g ⁻¹ DM)	8.5	1.6
<1	Water soluble N (g N 100 g^{-1} DM)	0.2	0.1
26	Soluble NDF (%DM)	48.2	12.2
10.1	HEM (%DM)	23.4	31.3
6.18	CEL (%DM)	22.8	44.2
15.5	LIG + Ash (%DM)	5.1	11.7
	158 122 102 307 311 1.49 15 <1 26 10.1 6.18 15.5	Crop properties 158 C-content (g C 100 g ⁻¹ DM) 122 N-content (g N 100 g ⁻¹ DM) 102 C:N 307 Humidity (WM DM ⁻¹) 311 NO ₃ N (g N 100 g ⁻¹ DM) 1.49 NH ₄ ⁺ -N (g N 100 g ⁻¹ DM) 15 Water soluble carbohydrates (g C 100 g ⁻¹ DM) 26 Soluble NDF (%DM) 10.1 HEM (%DM) 6.18 CEL (%DM) 15.5 LIG + Ash (%DM)	Crop propertiesRed clover158C-content (g C 100 g^{-1} DM)44.5122N-content (g N 100 g^{-1} DM)2.5102C:N17.9307Humidity (WM DM^{-1})6.5311 NO_3^- -N (g N 100 g^{-1} DM)0.01.49 NH_4^+ -N (g N 100 g^{-1} DM)0.015Water soluble carbohydrates (g C 100 g^{-1} DM)0.226Soluble NDF (%DM)48.210.1HEM (%DM)23.46.18CEL (%DM)22.815.5LIG + Ash (%DM)5.1

Clay (<2 µm), Fine silt (2/20 µm), Coarse silt (20/50 µm), Fine sand (50/200 µm), and Coarse sand (200/2000 µm). *NDF* Neutral detergent fiber, *HEM* Hemicellulose, *CEL* Cellulose, *LIG* lignin

in the field (37% stem, 34% flowers and leaves, 29% petioles). The residual humidity was determined by drying separate subsamples at 80 °C (6.3, 7.3, and 6.0 g 100 g⁻¹ DM for stems, flowers and leaves, and petioles, respectively). A moisture level corresponding to 80% of the final fresh weight was recommended by the residue provider (see acknowledgement) for RC residues, and accordingly residues were rewetted by adding deionised water corresponding to 4 g g⁻¹ DM to RC residues in preparation for the experiment.

Winter wheat (Triticum aestivum L.) straw was obtained from the INRAe experimental site of Estrées-Mons (49° 52' 23.88" N 3° 1' 53.04" E) in July 2017 when winter wheat (WW) was at senescence (Sauvadet et al. 2018). The site has been cultivated for decades with intensive agriculture and deep tillage. However, the agricultural practices on the soil have differed since 2010, notably with the establishment of shallow tillage. The sample consisted (in dry weight) of stems (50%) and leaves (50%) as recommended by the WW residue provider (see acknowledgement). The residues were gently dried at 35 °C, and then they were cut to 1 cm length for the incubations. A moisture level corresponding to 20% of the final fresh weight was selected for WW straw residues; since the residual humidity of the wheat straw after drying was 11.9 g 100 g⁻¹ DM, about 0.13 g water was added corresponding to a final water content of 0.25 g g^{-1} DM.

Additional physico-chemical characteristics of RC and WW residues were determined at INRAe in Reims, France (Table 1).

Both residue types were left to absorb the water for between 10 and 15 min in preparation of experimental treatments.

Experimental treatments

Separate experiments with soil-residue treatments (specified below) were carried out during autumn 2018 (RC) and spring 2019 (wheat straw) at Aarhus University (AU), and during autumn 2019 and spring 2020 (selected treatments) at Karlsruhe Institute of Technology (KIT). Table 2 presents an overview of the treatments at AU and KIT. All experiments investigated main and interactive effects of soil and residue properties with respect to N_2O emissions and soil mineral N dynamics. Experiments at the two locations

were set up according to common protocols and incubated at the same temperature, 15 °C. However, experiments at AU and KIT had different objectives and were thus complementary. At AU, a full factorial experiment with 24 treatments in three replicates, was conducted, with N₂O emission measurements on ten sampling days and four destructive samplings for soil mineral N during 43 days. At KIT, the experiments investigated the detailed temporal dynamics of the emission of N₂O, but also those of CO₂, NH₃ and NO, in four selected treatments (Table 2) with three replications using an automated mesocosm system (Arias-Navarro et al. 2017). These incubations included six destructive samplings for mineral N.

Residue treatments included RC or WW straw, or no amendment. Two residue management practices were mimicked, i.e., mixing at 0–4 cm depth to simulate shallow tillage or rotovation, or placement of residues in a discrete layer at 4 cm depth to simulate inversion tillage. Soil conditions included two levels of soil water-filled pore space (40 or 60% WFPS) to represent a range of soil moisture conditions that is typical during spring and autumn in wet temperate climates, and two levels of soil NO₃⁻-N content (Low = No amendment; High = 100 mg NO₃⁻-N kg⁻¹ dry wt. soil achieved by addition of KNO₃).

Preparation of samples

Before each experiment, soil was thawed and stored at +2 °C overnight. Soil moisture was determined by oven drying (105 °C for 24 h) to calculate adjustments needed to reach 40 or 60% WFPS based on the soil bulk density of 1.25 g cm⁻³ of the field site, which was also used for incubation experiments. Soil NO_3^{-} -N content was also determined prior to the experiment as described below.

Seven days prior to residue amendment, soil was packed to the target bulk density of 1.25 g cm^{-3} . The height of soil cores (8 cm) was identical in all experiments, but the diameter varied: At AU, two sets of samples were prepared in triplicate for each treatment, one set in 6.2 cm inner diam. Cylinders for gas sampling, and another set in 20 cm inner diam. Cylinders for all soil mineral N samplings. At KIT, the sample diameter was 12.7 cm, and two sets were prepared in triplicate for soil gas flux measurements and mineral N sampling, respectively. Soil cores were prepared by stepwise packing of the soil in layers of 1 cm, each time adjusting soil

Table 2 The combinations of crop residue type, residue distribution, soil NO_3^- , and soil moisture level in two factorial experiments (1–12 and 13–24) with batch incubation, as well as continuous flow incubations for selected treatments (25–28).

Treatments 1–12 and 13–24 constituted two sequentially completed experiments, which is why unamended treatments were included twice

Treatment §	Residue type	Residue distribution	Soil NO ₃ ⁻ -N Low = no addition, initial 12 mg NO ₃ ⁻ -N kg ⁻¹ , High = 100 mg NO ₃ ⁻ -N kg ⁻¹	Soil moisture Low=40% WFPS, High=60% WFPS
1	Red clover	Mixed	Low	Low
2	Red clover	Mixed	High	Low
3	Red clover	Mixed	Low	High
4	Red clover	Mixed	High	High
5	Red clover	Layered	Low	Low
6	Red clover	Layered	High	Low
7	Red clover	Layered	Low	High
8	Red clover	Layered	High	High
9	None	NA	Low	Low
10	None	NA	High	Low
11	None	NA	Low	High
12	None	NA	High	High
13	Winter wheat	Mixed	Low	Low
14	Winter wheat	Mixed	High	Low
15	Winter wheat	Mixed	Low	High
16	Winter wheat	Mixed	High	High
17	Winter wheat	Layered	Low	Low
18	Winter wheat	Layered	High	Low
19	Winter wheat	Layered	Low	High
20	Winter wheat	Layered	High	High
21	None	NA	Low	Low
22	None	NA	High	Low
23	None	NA	Low	High
24	None	NA	High	High
25=4	Red clover	Mixed	High	High
26=16	Winter wheat	Mixed	High	High
27 = 20	Winter wheat	Layered	High	High
28 = 12,24	None	NA	High	High

NA - Not applicable; § - Treatments 1-24 where conducted at AU, and treatments 25-28 at KIT, see text for details

moisture and NO_3^- content by adding deionised water, or a KNO₃ solution, to the soil surface with a pipette to reach the intended soil WFPS and NO_3^- level. At AU, both ends of the cylinders were covered with perforated plastic caps to minimise water loss while allowing gas exchange. The cylinders were then pre-incubated at 15 °C in boxes with a loosely fit cover and wet paper towels under a rack tray to further minimise evaporation losses. With the automated incubation system at KIT, gas fluxes were recorded already during the pre-incubation phase. On Day 0 of each experiment, crop residues were added at a rate of 0.04 g DM cm⁻² (corresponding to 4 Mg DM ha⁻¹). For treatments with residues at 4 cm depth, this was achieved by transferring one half of each soil core to a different cylinder, adding the crop residues on top of the soil, and then pushing soil and residues into another cylinder with soil. For treatments with residues at 0–4 cm depth, this part of each soil core was mixed with the plant material and repacked, allowing for the volume of residues as indicated by the layered treatment. All cylinders were

covered at both ends with perforated plastic caps and incubated at 15 °C as described above. Water loss was monitored by reweighing twice a week; this was negligible at AU, and also low at KIT where adjustment for evaporation losses took place only twice during the experiment.

Gas sampling and analysis

Batch incubations

For N_2O flux measurements, the samples were put in 1-L glass bottles with lids having a rubber septum fitted with a three-way stopcock for gas sampling; a vacuum-greased rubber gasket ensured an air-tight closure. After closure, a 10 mL gas sample was immediately taken with a plastic syringe, and additional samples after approximately 20, 40, and 60 min. Gas samples were transferred to 6-mL preevacuated exetainers (Labco, High Wycombe, UK).

Gas sampling was taking place on day 1, 3, 6, 9, 13, 16, 22, 29, 36, and 43. Concentrations of N₂O and CO₂ were determined using a dual-inlet gas chromatograph (model 7890A) equipped with electron capture and thermo-couple detectors and a CTC Combi-Pal Autosampler (Agilent; Nærum, Denmark). The details of instrument configuration were described by Petersen et al. (2012). Concentrations were quantified with reference to synthetic air and a calibration mixture containing 2013 nL L⁻¹ N₂O and 2000 μ L L⁻¹ CO₂ (Air Products; Diegem, Belgium).

Continuous-flow incubations

For validation of temporal dynamics of CO_2 and N_2O , and to investigate additional gas fluxes, selected treatments (Table 2) were incubated also using an automated system based on a dynamic chamber approach. Gas concentrations were measured before and after each mesocosm, with continuous flushing of the headspace with ambient air at a rate of approximately 333 mL min⁻¹. Incubation conditions (i.e. temperature, quantity and quality of the air supply) were controlled and monitored inside two thermostatic cabinets.

The system encompassed a set of 18 containers (12.7 cm inner diameter, 12.0 cm height). Automatic gas sampling took place according to a continuously repeated 180-min sequence, in which each container

was sampled for 6 min and alternating with background-air measurements. A set of solenoid valves automatically divert the airflow from each container to either an acid trap, to determine ammonia (NH_3) volatilization, or to various gas-measuring devices to measure the concentrations of N₂O, NO and CO₂.

Concentrations of N₂O and CO₂ were determined using cavity ring-down spectroscopy (CW-QC-TIL-DAS-76, Aerodyne Research Inc., MA, USA). The gas analyzer was calibrated every measuring cycle using a gas blend containing defined concentrations of N₂O (408 ppbv) and CO₂ (406 ppmv) in synthetic air (Air Liquide GmbH, Düsseldorf, Germany). Nitric oxide (NO) concentrations were quantified by a chemiluminescence detector (CLD88p, Eco Physics AG, Duernten, Switzerland) calibrated daily with four different NO concentrations in synthetic air: 0, 50, 200 and 500 ppbv NO, prepared from mixtures of 4 ppm NO in N₂ (Air Liquide GmbH, Germany) and synthetic air $(20\% \text{ O}_2 + 80\% \text{ N}_2)$ using a multigas calibration system (series 6100; Environics Inc., Tolland, CT, USA). Ammonia (NH₃) in acid traps containing 100 mL of a 0.1 M oxalic acid were subsampled several times to determine NH_4^+ concentrations using green-indophenol at 660 nm (Epoch Microplate Spectrophotometer, BioTek Instruments Inc., United States). Sampling frequency depended upon the expected NH₃ volatilization dynamics, i.e., twice in the first week after residue incubation, and later less frequently. The cumulative NH₃ volatilization was calculated from the marginal NH_4^+ increase in the acid traps between sampling times.

Flux calculations

For the batch incubation method, the accumulation of N_2O and CO_2 was used to calculate fluxes using the HMR flux estimation method (Pedersen et al. 2010), available as an add-on package in R (R Project 2019). Cumulative emissions were calculated by a trapezoidal approximation to the integral under the emission curve in R (R Core Team).

For the dynamic chamber measurements at KIT, the soil-headspace exchange rate of each trace gas was calculated from the mass balance between the inlet (ambient air) and outlet (chamber air) concentrations assuming mass flow equilibrium conditions (Pape et al. 2009).

$$F_{cham} = \frac{Q}{A} \times \rho \big(\mu_{chamb} - \mu_{amb} \big)$$

where F_{cham} stands for the trace gas flux (nmol m² s⁻¹); *A* denotes the surface area of the samples (m²); *Q* is the headspace air flow rate (m³ s⁻¹). μ_{cham} and μ_{amb} are the trace gas mixing ratios (nmol mol⁻¹) of the inlet and outlet air, respectively; and ρ is the molar density of dry air (mol m⁻³). During sampling, a dynamic equilibrium was soon reached, where μ_{cham} was effectively constant during the 6 min gas flux measurements.

Soil mineral N

All treatments were analyzed for soil mineral N several times during incubation. At AU, samples were collected from 20 cm diam. Cylinders with a miniauger (15 mm diameter) on day 1, 6, 22, and 43 of incubation. Three subsamples were taken from each treatment (0–8 cm) and pooled. At KIT, separate samples were destructively sampled on day 0, 4, 7, 14, 28, and 60.

Subsamples of around 10 g fresh wt. soil were extracted in 40 ml 1 *M* KCl by shaking end-over-end for 30 min and then filtered through 1.6 μ m glass microfiber filters (VWR, Sweden). The filtrates were stored at -20 °C until analyzed. At AU, ammonium-N (NH₄⁺-N) and nitrate-N (NO₃⁻-N) was performed with standard colorimetric methods on a Foss FIAstar 5000 flow injection analyzer (FOSS Denmark). At KIT, the samples were analyzed by an external laboratory (Raiffeisen Laboservice; Ormont, Germany). Gravimetric soil water content was determined by drying approximately 10 g of soil for 24 h at 105 °C.

Statistical analyses

Cumulative fluxes of CO_2 -C and N_2O -N were estimated from non-transformed data. The arithmetic means of emissions observed at each time point were calculated and integrated using the trapezoidal rule for integration (Levy et al. 2017). Nitrous oxide emission factors, and recovery of residue-C in CO_2 , were calculated by subtracting the appropriate control with respect to soil WFPS and NO_3^- availability.

The cumulative N_2O (determined at AU and KIT) and NO emissions (determined at KIT only) were

analyzed using a generalised linear model (GLM) with the response given by the integrated emissions for each experimental unit. The models were defined using an identity link function and the Gamma compound Poisson distribution. The Gamma compound Poisson distribution allows modelling of positive responses with significant variability, as well as values below the detection limit ("zero values"), see Cordeiro et al. (2021) and Jørgensen and Labouriau (2012). We analyzed emissions from the AU and the KIT studies separately, but using the same GLM. In both cases, we checked for the presence of fourth order interactions using a likelihood ratio test for GLMs and found that the interaction terms were statistically significant (p values < 0.00001). Note that main effects and lower order interaction effects do not have a meaningful interpretation in models with fourth order interactions; therefore, we present results for the effects of the treatment combinations (residue type, distribution method, NO₃⁻ level and WFPS level). Post-hoc analyses were performed to identify specific differences between treatments via pairwise comparison with p values corrected for multiple comparisons by the false discovery rate (FDR) method (Benjamini and Hochberg 1995). All the post-hoc analyses were conducted using the R-package "post-Hoc" (Labouriau 2020).

Statistical analysis of mineral N data was performed using Generalised Linear Mixed Models defined with the logarithm link function and a Gaussian random component designed to account for the dependence of the observations arising from the same experimental units, but at different time points. The analyses for RC and WW residues were performed separately in parallel analyses. The models used for NH₄⁺-N were constructed using the Gamma compound Poisson distribution, since several observations were below the detection limit. The models for NO₃⁻-N were based on the Gamma distributions. The presence of interactions between residue distribution, nitrate level WFPS level and experimental day was tested using likelihood ratio tests. Since we did not find statistically significant interactions, we report the main additive effects of each of the determining factors (note that additive factors act multiplicatively on the expected values due to the use of the logarithmic link function). The ratios for each level of the explanatory variables were calculated relative to one level chosen as reference.

Results and discussion

CO₂ and N₂O emissions from red clover residues

Similar patterns in CO_2 evolution were observed with mixed and layered RC residues, except for the treatment where residues were mixed with soil at 0–4 cm depth and 40% WFPS (Fig. 1). Here, CO_2 evolution rates were higher by day 1, but lower during the remaining part of the incubation, indicating an accelerated turnover of degradable substrates. Water, but not NO₃⁻-N addition to control soils stimulated CO_2 evolution. The detailed results on CO_2 evolution dynamics for the treatment with RC residues mixed into 0–4 cm soil at 60% WFPS (Fig. 2A) confirmed the dynamics captured in the factorial experiment.

The delay in residue decomposition at the higher water content, and with layered distribution, suggests that O₂ availability may have limited heterotrophic activity. This interpretation was supported by N₂O evolution rates, which were consistently low at 40% WFPS, but significantly higher and peaking on day 3 at 60% WFPS. In NO₃-amended soil, the rate increased to an extreme value of 60 mg N_2 O-N m⁻² h⁻¹ by day 3 in the mixed RC treatment of both batch and continuous incubations (Figs. 1 and 2B). Henderson et al. (2010) reported that extractable organic carbon in soil amended with RC residues was depleted after 2-3 days, and this may have contributed to the subsequent decline. Nitrous oxide evolution from the 60% WFPS treatment without nitrate amendment declined between day 1 and 3 and then remained low (Fig. 1) presumably because nitrate for denitrification was depleted (see below).

CO_2 and N_2O emissions from wheat straw residues

The control treatments without residue amendment in experiments with WW straw showed less CO_2 and N_2O evolution compared with the same treatments in the preceding experiment with RC (Figs. 1 and 2). Therefore, although the soil being stored at -20 °C between the two experiments, apparently most labile organic matter in the bulk soil had been lost at the time of the second incubation.

The CO_2 evolution rates in treatments amended with wheat straw showed consistent effects of residue distribution and soil NO_3^- availability in the factorial experiment (Fig. 1). The rates were higher by day 1 when residues were mixed with the soil as compared to a layered distribution (Fig. 1). Then followed a decline and a secondary increase, which peaked after 1-2 weeks. These dynamics were confirmed with automated measurements, except that the transient decline was deeper with layered distribution (Fig. 2). During the 2nd and 3rd week of incubation, there was evidence for faster degradation of the wheat straw in NO₃⁻-amended soil when straw was mixed with the soil, indicating that N was a limiting factor for WW straw decomposition in this period.

Nitrous oxide evolution rates were generally low compared to those associated with RC residues (Fig. 1). There was a tendency for higher rates at 60% WFPS, and in NO_3^{-} -amended soil, but treatment effects were generally low. This was confirmed in the experiment with continuous-flow incubations (Fig. 2A).

Soil mineral N dynamics with red clover residues

The level of NH_4^+ -N in RC treatments was significantly higher at 60 compared to 40% WFPS (Table 3). At 40% WFPS, net N mineralization was observed by day 6, and with almost complete disappearance of NH_4^+ from day 21 and onwards (Fig. 3). At 60% WFPS, however, NH_4^+ could be observed throughout the 43 d incubation, but with a trend towards depletion when residues and soil were mixed, and accumulation when RC residues were placed as a layer.

In unamended soil, NH₄⁺ concentrations was at the detection limit, while there were slight changes in NO_3^- concentrations over time. At 40% WFPS, a transient decline by day 6 was replaced by net accumulation of NO₃⁻ in the mixed treatments, while at 60% WFPS the NO₃⁻ levels did not increase. In contrast, with a layered distribution there was a declining trend in soil NO₃⁻ concentrations throughout the incubation period independent of NO_3^- and WFPS level (Fig. 3). In both cases, these trends were independent of NO₃⁻ amendment. There was a significant decline in soil NO₃⁻ concentration during the first week (Table 3), especially with mixed distribution of RC residues (Fig. 3), and no net increase throughout the 43 d incubation. The net decline in NO₃⁻ concentration between day 1 and day 6 in the NO₃⁻-amended treatment was c. 70 μ g NO₃⁻-N g⁻¹ dry wt. soil, which for an 8 cm soil phase with a bulk density of 1.25 g cm^{-3} would





Fig. 1 Carbon dioxide (CO_2) and nitrous oxide (N_2O) from RC (top two panels) and WW treatments (bottom two panels) in a factorial experiment with two levels of residue distribution

(mixed at 0–4 cm depth vs. layered at 4 cm depth), soil water content (40 vs. 60% WFPS) and NO_3^- availability (ambient vs. 100 mg N kg⁻¹)

be able to account for an average emission of 58 mg N_2O -N m⁻² h⁻¹ between day 1 and 6. In comparison, N₂O evolution observed with the mixed treatment in this period (Fig. 1) corresponded to c. 32 mg N₂O-N m⁻² h⁻¹, suggesting that N₂O and N₂ were both products of denitrification. In accordance with these numbers, Miller et al. (2008) incubated RC residues with 50 mg kg⁻¹ NO₃⁻⁻N and found that the N₂O/(N₂O+N₂) product ratio declined from 0.9 to 0.2 during this period. It should be mentioned, however, that Miller et al. (2008) also added 250 mg kg⁻¹ glucose to the soil, and so the

experimental designs and results cannot be directly compared.

In treatments with RC residues in a layer, the disappearance of NO_3^- was less than in mixed treatments at 60% WFPS despite the fact that very likely the extent and duration of anoxic conditions supporting denitrification were greater with discrete as opposed to mixed distribution, as shown with incubations of cattle manure (Petersen et al. 1992). The time course of N₂O emissions in this treatment indicated that denitrification occurred primarily during the first week. Petersen

Fig. 2 Temporal changes in (A) CO₂, (B) N₂O and (C) NO fluxes for selected treatments (Table 2) as observed with an automated sampling system. Ribbons indicate the standard error of the mean (n=3)



Table 3Statistical analysisof mineral N data from thefull factorial experimentsat AU with RC and WWresidues, respectively. Theeffects shown representratios relative to a referencevalue (reff.) calculated foreach explanatory variableusing generalised linearmixed models defined witha logarithmic link function

Variables	Red clover (RC)		Winter wheat (WW)		
	NH4 ⁺ - N	NO ₃ ⁻ -N	NH4 ⁺ -N	NO ₃ ⁻ -N	
Residue distrib	oution				
Mixed	12.58 (7.18-22.04) ^b	0.52 (0.29-0.99) ^a	2.99 (1.42-6.3) ^b	0.48 (0.34–0.67) ^a	
Layered	9.2 (5.23-16.18) ^b	0.73 (0.4-1.32) ^a	2.51 (1.18-5.32) ^b	0.6 (0.43–0.85) ^a	
None	1.00 (reff.) ^a	1.00 (reff.) ^a	1.00 (reff.) ^a	1.00 (reff.) ^b	
p value	< 0.0001	0.1524	0.0274	0.0060	
Nitrate level					
Low	1.00 (reff.) ^a	1.00(reff.) ^a	1.00(reff.) ^a	1.00(reff.) ^a	
High	0.81(0.52-1.26) ^a	6.36(3.92-10.32) ^b	3.05(1.67-5.57) ^b	8.07(6.12-10.64) ^b	
p value	0.3576	< 0.0001	0.00171	< 0.0001	
WFPS level					
Low	1.00 (reff.) ^a	1.00(reff.) ^b	1.00(reff.) ^b	1.00 (reff.) ^a	
High	2.01(1.29-3.14) ^b	$0.43(0.27-0.7)^{a}$	$0.42(0.23-0.76)^{a}$	0.8(0.61-1.05) ^a	
p value	0.0073	0.0065	0.0103	0.1305	
Day of experim	nent				
1	1.00 (reff.) ^{ab}	1.00(reff.) ^b	1.00(reff.) ^c	1.00(reff.) ^c	
6	1.83(0.99-3.4) ^b	$0.61(0.47-0.8)^{a}$	$0.18(0.09-0.4)^{b}$	$0.77(0.6-0.99)^{bc}$	
22	$0.47(0.25-0.89)^{a}$	0.78(0.6-1.02) ^{ab}	0.07(0.03-0.16) ^{ab}	0.57(0.44-0.74) ^{ab}	
43	0.74(0.39-1.39) ^a	0.83(0.63-1.08) ^{ab}	$0.03(0.01-0.06)^{a}$	0.52(0.4-0.68) ^a	
p value	0.0026	0.0061	< 0.0001	0.0002	
p value for tests of additivity	0.6293	0.07421	0.09504	0.44030	

95% confidence intervals are shown in parentheses for each reported ratio

Letters indicate significant differences for each explanatory variable reported (constructed with 5% significance level).

P-values for testing additivity (in the logarithmic scale) are reported in the last row of the table

et al. (1996) concluded, based on modelling of NO_3^- profiles in soil cores with a layer of cattle manure, that denitrification was constrained by NO_3^- availability during the first week, and that only soil NO_3^- supported denitrification for at least three days after manure application. Hence, transport of NO_3^- to the soil-residue interface probably limited denitrification during the phase of residue decomposition with a potential for N₂O emissions.

Soil mineral N dynamics with wheat straw residues

There was a small, but consistent increase in NH_4^+ -N content by day 1 after residue application, especially in the mixed treatment (Fig. 3). This suggests an accelerated turnover of labile organic matter with a low C:N ratio, possibly microbial biomass, and accords with elevated CO_2 evolution on day 1 (Fig. 1). The fact that NH_4^+ level was higher in NO_3^- amended soil (Table 3) suggests that

denitrifiers contributed to residue decomposition. There was a consistent decline in soil NO_3^- concentration in the presence of WW straw despite evidence of net N mineralization and nitrification in the bulk soil (Fig. 3).

The soil mineral N dynamics in WW treatments from automated chambers were comparable with batch incubations, except that here a peak in both NH_4^+ -N and NO_3^- -N were observed by day 4, which was also seen in unamended soil and hence could be associated with soil organic matter decomposition (Supplementary Information, Figure 1).

NO and NH₃ emissions from selected treatments at KIT

The automated incubation system captured a significant, but short-lived peak in evolution of NO from the treatment with RC mixed with soil at 60% WFPS and high



Fig. 3 Soil mineral N (NH₄⁺-N and NO₃⁻-N) determined in the all treatments at AU (error bars = + s.e.m.; n = 3)

 NO_3^- availability (Fig. 2c), which preceded the peak in N_2O evolution (Fig. 2B). Both nitrification or denitrification are sources of NO emission from soil, but there was significant net consumption of NO_3^- during the first week, and the timing of NO relative to N_2O indicates that NO was produced as an intermediate of denitrification.

The NH₃ volatilisation rates were similar in control and WW treatments (both mixed and layered), whereas NH₃ volatilisation was higher in mixed RC treatment with up to 0.7 kg NH₃-N ha⁻¹, and <0.2 kg NH₃-N ha⁻¹ in other treatments.

Cumulative emissions

The cumulative emissions of CO_2 and N_2O during 43 days in the factorial experiment, and of CO_2 , N_2O and NO in treatments incubated with continuous flow,

are shown in Table 4. Cumulative CO_2 was lower for WW as compared to RC treatments, with percentages of residue-C recovered in CO_2 evolution ranging from 26 to 44% vs. 41 to 67%. There were no consistent treatment effects. The lower degradability of WW straw may be related to the higher C:N ratio (as indicated by faster turnover in NO_3^- -amended soil) and higher lignin content (Table 1), in accordance with earlier studies (Fageria et al. 2007; Li et al. 2020).

The cumulative N_2O emissions were highly variable, and N_2O emission factors calculated with reference to residue N varied by one (WW) to two orders of magnitude (RC) with incubation conditions. As hypothesized, N_2O emissions were much higher with RC having lower C:N ratio, and there was a consistent, though non-significant trend towards higher emission at high soil NO_3^- availability. The

Table 4 Cumulative CO_2 and N_2O emissions from batch incubations (AU) and continuous flow incubations with an automated system (KIT). Nitrous oxide emission factors for

residue N were calculated with reference to the appropriate unamended control treatment

Residue	Distribution	NO ₃ ⁻	WFPS	Cumulative CO_2 (kg CO_2 -C ha ⁻¹)	Cumulative N ₂ O (kg N ₂ O-N ha ⁻¹)	N ₂ O Emission factor (%)	Cumula- tive NO(kg NO-N ha ⁻¹)
Batch incu	bation						
RC	Mixed	Low	Low	1090 [45] [§]	0.60 (0.20-1.0 abcd	0.59	
RC	Mixed	High	Low	938 [44]	0.76 (0.27-1.24) abcd	0.44	
RC	Mixed	Low	High	1660 [52]	2.24 (1.05-3.43) cde	0.53	
RC	Mixed	High	High	1559 [65]	40.65 (27.48–53.81) ^f	39.2	
RC	Layered	Low	Low	1294 [57]	0.29 (0.07-0.51) abc	0.24	
RC	Layered	High	Low	1346 [67]	1.19 (0.49-1.90) abed	1.0	
RC	Layered	Low	High	1450 [41]	2.87 (1.41-4.33) de	0.66	
RC	Layered	High	High	1421 [57]	7.36 (4.17–10.54) ^e	5.5	
None	NA	Low	Low	288	0.05 (0.00–0.11) ^a		
None	NA	High	Low	150	0.20 (0.04-0.36) abc		
None	NA	Low	High	726	2.36 (1.12-3.60) cde		
None	NA	High	High	408	1.92 (0.88-2.97) bcde		
WW	Mixed	Low	Low	640 [26]	0.02 (0.00–0.04) ^a	0.05	
WW	Mixed	High	Low	796 [38]	0.32 (0.08-0.56) abcd	0.98	
WW	Mixed	Low	High	800 [35]	0.10 (0.01–0.19) ^{ab}	0.39	
WW	Mixed	High	High	864 [41]	0.34 (0.09-0.59) abcd	1.3	
WW	Layered	Low	Low	666 [28]	$0.04 (-0.00-0.07)^{a}$	0.14	
WW	Layered	High	Low	643 [30]	0.09 (0.01–0.17) ^a	-0.21	
WW	Layered	Low	High	813 [44]	0.21 (0.04-0.37) abc	0.65	
WW	Layered	High	High	742 [40]	0.23 (0.05-0.41) abc	0.72	
None	NA	Low	Low	148	0.01 (0.00–0.03) ^a		
None	NA	High	Low	84	0.13 (0.02–0.24) ^{ab}		
None	NA	Low	High	150	0.04 (0.00–0.09) ^a		
None	NA	High	High	101	0.10 (0.01–0.19) ^{ab}		
Continuou	s flow						
RC	Mixed	High	High	1140 (57)	37.7(18.2–57.1) ^b	34.5	0.19 (0.14–0.24) ^b
WW	Mixed	High	High	853 (39)	3.4 (1.0–5.9) ^a	11.4	0.07 (0.04–0.10) ^a
WW	Layered	High	High	780 (35)	3.9 (1.1–6.7) ^a	14.1	0.05 (0.02–0.08) ^a
None	NA	High	High	124 b	1.3(0.2–2.3) ^a		0.15 (0.1–0.19) ^b

[§]Numbers in square brackets indicate the percentage of residue-C recovered as CO₂-C. Number in parentheses represent confidence intervals with 95% coverage

hypothesis that higher soil moisture would generally increase N_2O emissions was not confirmed, since it was only the case in combination with high NO_3^- availability. Also, there was not evidence for greater temporal stability of N_2O emissions with the distribution of residues as a layer in this study, where N_2O emissions were short-lived.

Some emission factors stood out as unrealistic, including the treatment in which RC residues were

mixed with soil amended with NO_3^- at 60% WFPS, where both batch and continuous flow incubation showed N₂O evolution corresponding to 35–40% of residue N applied. Soil NO_3^- likely contributed to these high emissions, which do not reflect known observations in field studies (Jungkunst et al. 2006). Further, emission factors for WW obtained with the continuous flow system exceeded 10%, but the temporal dynamics of emissions shown in Fig. 2A suggest that this could be due to a small carry-over effect within the analytical system. Discrete distribution of RC residues in a layer, as opposed to mixing, reduced the N₂O emission factor to 5.5%. The surface-to-volume ratio of a layer may be compared to that obtained with inversion tillage under field conditions. Disregarding the High NO₃⁻ + High WFPS treatments, the N₂O emission factors averaged 0.3 and 0.6% for WW and RC, respectively, which is comparable with the level of annual emission factors recently proposed for residue N (IPCC 2019).

The cumulative NO evolution was highest in this RC treatment, though not different from the control; both were higher than WW treatments (Table 4). It is widely considered that NO released from aerobic soils is mainly produced by nitrification, but both nitrification and denitrification can produce NO (Skiba et al. 1997), and a meta-analysis based on a limited number of studies reported that soil NO emissions mainly occur after fertiliser application (Liu et al. 2017). The time course of NO emissions showed a peak around Day 3, which suggested that in this study NO emissions were derived from denitrification (data not shown).

During decomposition the crop residues may, depending on C:N ratio, release ammoniacal N, which is both a source of NH_3 emissions and the starting point for a range of microbial processes that can produce both NO and N₂O, such as nitrification and denitrification (Butterbach-Bahl et al. 2013). The total amount of NH_3 volatilisation in the mixed RC treatment reached 0.6 kg NH_3 -N ha⁻¹ over 60 days, while no emission of NH_3 was observed with WW. Increases in NH_3 emissions following residue return can, in general, be attributed to net N mineralization, in accordance with the difference in net N mineralisation profiles between WW and RC residues in this study.

General discussion

Full factorial experiments were conducted with red clover and winter wheat residues, while the temporal dynamics of CO_2 and N_2O emissions were examined by independent near-continuous measurements for selected treatments. Both the magnitude and temporal dynamics of N_2O emissions were very similar for treatments where both incubation methods were used, suggesting that soil-air gas exchange was not affected

by the different boundary conditions of batch vs. continuous-flow incubation, but rather controlled by reactions and transport within the soil.

Previous studies have shown that residue effects on soil N₂O emissions vary with residue properties. For example, Millar and Baggs (2005) applied six crop residues with the same amount of N and found significant negative correlation between N₂O emission and the residues' soluble C:N ratio, in accordance with the contrast between RC and WW residues observed in the present study. More recently, Laville et al. (in prep.) screened 24 different crop residues in incubations with mixing of residues at 0–4 cm depth and 60% WFPS, but without NO₃⁻ amendment, and found N₂O emission factors ranging from 0.3 to 4.7%.

The present study focused on effects of the interactions between residue decomposition and the soil environment for N₂O emissions. Loecke and Robertson (2009) showed that crop residue aggregation suppressed decomposition compared to uniform mixing of residues and soil at 80% WFPS. It suggests that availability of soil resources such as O₂ is important for crop residue decomposition under field conditions, and O₂ diffusion may better meet O₂ consumption rates when crop residues are well distributed as opposed to aggregated.

The depth of incorporation in the field is often greater than in incubations such as the present study, and this could have influenced emissions if the diffusive supply of O2 and/or the soil accumulation of N2O were critical for soil microbial processes associated with the formation and consumption of N₂O. In our experiments, the higher soil moisture level of 60% WFPS already limited O₂ supply to the soil, allowing anoxic conditions to develop around decomposing residues and stimulate denitrification, even though residues were only mixed into the top 4 cm of soil. Parkin (1987) found that even a water film of 20 µm could be enough to support denitrification from residues exposed to air, and so distance from the soil surface per se may not be a critical factor when determining the N₂O production potential of residue incorporation. However, concentration gradients control the flux of N₂O, and shallow incorporation could therefore enhance emissions when N₂O is produced. This may have contributed to the extremely high, but transient rate of N2O emissions (>50 mg N₂O-N m⁻² h⁻¹) observed in the treatment where RC residues were mixed into soil at 0-4 cm depth at 60% WFPS. High soil NO3⁻ availability will

reduce the potential for N_2O reduction to N_2 in connection with denitrification (Giles et al. 2012), and this could also enhance N_2O emissions. The release of N_2O was an order of magnitude lower when residues were placed at a discrete layer at 4 cm soil depth and indicates that residue distribution is also important for the regulation of N_2O emissions.

Angers and Recous (1997) examined the effect of wheat straw particle size on decomposition and found a positive relationship between residue-soil contact area and the extent of decomposition after 21 d. They found that smaller particles mixed into the soil would decompose faster. A higher residue-soil contact area will increase O₂ availability for aerobic decomposition, but also the contact with NO₃⁻ in the soil solution. Justes et al. (2009) reported that the initial C mineralization from catch crop residues was positively correlated with their soluble C content, and therefore O2 consumption probably extends into the surrounding soil, as shown with soybean residues by Kravchenko et al. (2017) in a study using planar optodes. The high residue-soil contact area may thus have contributed to the extreme N₂O emissions in the treatment with RC residues mixed into soil at 60% WFPS and elevated NO₃⁻. Chen et al. (2013) found that N_2O emissions were enhanced more by crop residue amendment in laboratory experiments compared to field studies, and a greater soil-residue contact in mixed incubation systems may have been responsible for this effect. Thus, residue distribution can also affect the outcome of incubation studies to estimate the N₂O emission potential of crop residues.

For prediction of soil N₂O emissions under controlled conditions, the importance of soil microbiology must also be considered. Nitrification and denitrification are both potential sources of N2O. However, Attard et al. (2010) found that soil organic carbon, together with water-filled pore space and soil NO₃⁻ availability, could account for around 90% of the observed variability in N₂O emissions from two sites exposed to recent changes in land use and land management, including residue recycling. These authors concluded that the main reason for this variability was changes in soil denitrification activity. In the present study, the temporal dynamics of NO and N2O emissions also pointed to denitrification as the main source of N2O emissions induced by RC residues. Chèneby et al. (2009) observed a 20-27 fold increase in the abundance of nitrate reducing bacteria during the first week in soil amended with RC residues, confirming that this environment supports denitrification. Miller et al. (2008) observed an 8-fold increase in denitrifying enzyme activity after 72 h when incubating RC residues with extra NO₃⁻, and a much smaller stimulation without NO₃⁻ amendment, i.e., NO3⁻ availability constrained denitrifier growth, as discussed above. However, Attard et al. (2010) also concluded that the abundance and diversity of denitrifiers were of minor importance for the extent of denitrification in soil, and similar observations were reported by Duan et al. (2018) from a two-year field study comparing N₂O emissions during spring with and without a cover crop the previous winter. Also, Henderson et al. (2010) found that denitrifiers responded similarly to residues of different quality (red clover, soybean and barley). Together, these observations suggest that N₂O emissions may not be very sensitive to soil type or crop specific differences in denitrifying communities, and that instead the drivers directly affecting dentrification, i.e., electron donor and acceptor availability, are more critical.

Screening crop residues for N₂O emission potential under standardised conditions could be a way to quantify the effect of residue chemical composition. However, this study demonstrated significant interactions between residue decomposition and the physical and chemical environment, which eventually reflect on N₂O emissions. Soil NO₃⁻ availability was clearly important, in accordance with reports of higher N₂O emissions from residues under field conditions when synthetic fertilisers is applied (Guardia et al. 2016). There is a need to distinguish between N₂O emissions sustained by residue N and emissions resulting from interaction with other soil N pools. This may be N from recent applications of fertilisers or manure, or N mineralised from soil organic matter ("background" emissions); in both cases the O_2 sink capacity of crop residues may be the driver of N₂O emissions not actually derived from residue N. In addition, soil moisture conditions will define the O_2 supply and hence potential for anaerobic decomposition. It implies that assessment of N2O emission factors must take place under conditions of O_2 supply representative of the field situation when residues are recycled.

Conclusions

This study, investigating N₂O emissions from crop residues under controlled laboratory conditions, confirmed that residue N is a poor predictor of N₂O emissions. Treatment effects showed strong interactions between residue distribution and soil properties (notably soil aeration, NO₃⁻ availability), and hence site-specific conditions and management are likely important for N2O emissions. The temporal dynamics of soil NO_3^- and N_2O emissions indicated that denitrification was the main source of N₂O. With most treatment combinations, the short-term incubations showed N2O emission factors in the range expected for annual emissions under field conditions with both batch incubation and continuous-flow systems. Based on this we conclude that the development of improved N₂O emission factors for crop residues may be supported by incubation experiments representing field conditions with respect to O2 and NO3⁻ availability, and residue distribution.

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