



Predicting nitrous oxide emissions from manure properties and soil moisture: An incubation experiment



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ABSTRACT

Field-applied manure is a source of essential plant nutrients, but benefits may be partly offset by high rates of nitrous oxide (N₂O) emissions, as modified by manure characteristics and soil properties. In a 28-d incubation experiment we quantified short-term emissions of N₂O from a sandy loam soil amended with digestate (*DI*), pig slurry (*PS*) or cattle slurry (*CS*), and unamended soil (*Ctrl*), when incubated at 60, 70 and 80% water-filled pore space (WFPS). The soil was amended with ¹⁵N-labelled nitrate to distinguish sources of N₂O. Emissions of N₂O were not related to N input and corresponded to between 0.04 and 2.42% of manure N, decreasing in the order *CS* > *DI* > *PS* > *Ctrl* within each WFPS level. Recovery of ¹⁵N in N₂O indicated that heterotrophic denitrification constituted at least 64–77% of total emissions at 70 and 80% WFPS, while nitrification was more important for the low emissions at 60% WFPS. The results were further analyzed with a static two-compartment model of N₂O emissions from manure. Experimental results showed a much stronger response to soil moisture than predicted by the model, and therefore a new term was introduced linking the balance between aerobic and anaerobic decomposition to relative soil gas diffusivity. Model parameters for sources of N₂O, estimated from experimental results by multiple linear regression, indicated that denitrification was responsible for 79–98% of N₂O emissions at 70 and 80% WFPS, and 45–59% at 60% WFPS.

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1. Introduction

Globally, net anthropogenic emissions of nitrous oxide (N₂O) to the atmosphere are estimated at 5.3 Tg N₂O–N yr⁻¹, with a 66% share from agriculture (Davidson and Kanter, 2014). Field application of manure on livestock farms is a large, but highly variable source of agricultural N₂O emissions, typically in the range from <0.1 to 3% of total applied nitrogen (N) (Chadwick et al., 2011). Growth and intensification of livestock production leads to increasing volumes of manure to be managed. Since large farms are dominated by liquid manure management (Eurostat, 2015), the growth in liquid manure volumes is particularly high, and methods to better predict and, in turn, reduce N₂O emissions are urgently needed.

The methodology used in national inventories estimates N₂O emissions from field-applied manure as a percentage of the N input (IPCC, 2006). While mineral N is a precondition for N₂O emissions

via nitrification and denitrification activity, it is not the only driver. In particular, oxygen (O₂) limitation is a key factor that may stimulate emissions of N₂O from both nitrification (Khalil et al., 2004) and denitrification (Coyne, 2008). It is well known that wet or compacted soil promotes N₂O emissions (Balaine et al., 2013), but Weier et al. (1993) found that, for a sandy soil with up to 75% water-filled pore space (WFPS), emissions of N₂O were low unless amended with degradable carbon (C). When degradable C is applied to soil in liquid manure, the distribution is highly heterogeneous. Direct measurements of O₂ distribution with micro-sensors (Markfoged et al., 2011) and optodes (Zhu et al., 2015) have shown how O₂ depletion in the soil coincides with the distribution of manure organic matter, and Markfoged et al. (2011) further linked zones of O₂ depletion with N₂O accumulation. Hence, decomposition of manure organic matter (volatile solids, VS) in local hotspots could be an important driver for N₂O emissions in unsaturated soil.

Manure properties are modified by animal diet, treatment, and storage conditions (Møller et al., 2014) and therefore knowing manure characteristics at the time of field application may be important for predicting N₂O emissions. Petersen et al. (2003)

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found that, upon field application, a fraction of liquid manure will be absorbed by the surrounding soil, and a fraction retained in a manure-saturated environment with high moisture and high biological activity. Furthermore, the results indicated that infiltration of manure liquid could be predicted from manure VS and soil water potential (Petersen et al., 2003). In bulk soil the potential for N₂O production is likely to be different from manure-saturated volumes. Sommer et al. (2004) proposed an empirical two-component model predicting redistribution of labile C and N between manure hot-spots and bulk soil, and subsequent N₂O emissions from nitrification and denitrification. However, model parameters were based on literature data, and the two-compartment model has not been tested experimentally.

We conducted an incubation experiment with the objective to quantify effects of manure properties and soil moisture on N₂O emissions. A second objective was to evaluate the model of Sommer et al. (2004) against experimental results. Sources of N₂O were estimated in two different ways: a) by ¹⁵N labelling of soil NO₃⁻ and then measuring recovery of ¹⁵N in emitted N₂O (Stevens and Laughlin, 2001), and b) by estimation of model parameters from multiple linear regression analysis of observed N₂O emissions. We hypothesized that i) manure VS will increase N₂O emissions independent of WFPS; and that ii) the importance of denitrification as source of N₂O will increase with manure VS concentration.

2. Materials and methods

2.1. Soil characteristics

Soil for the incubation experiment was collected at Foulumgaard Experimental Station (56°29'N, 9°34'E), Denmark in autumn 2013, after harvest of winter wheat. The soil in the area is classified as a Typic Hapludult (coarse sandy loam) with 3.5% organic matter, 90 g kg⁻¹ clay, 50 μS cm⁻¹ electrical conductivity, and 6.1 soil pH_{H2O} 1:2.5. The soil was collected from 0 to 25 cm depth after removing a thin layer of surface litter, sieved <6 mm and thoroughly mixed, and then stored at 10 °C until commencement of the experiment.

2.2. Characteristics of slurries and digestate

Cattle and pig slurry were collected from fully mixed storage tanks located at Foulumgaard Experimental Station. Cattle slurry originated mainly from dairy cows, and pig slurry from finishing pigs and farrowing sows, delivered from the livestock production facilities at the Research Centre AU-Foulum; both materials had been stored for several months at the time of collection. Digestate was taken from a storage tank at a biogas plant near Foulum Research Station with an 1100-m³ active reactor volume and operated with a hydraulic retention time of 13–14 d and 52 °C reactor temperature. Various organic materials, including maize silage and glycerol/fish silage (c. 20% by volume) were co-digested with cattle and pig slurry. The digestate was sampled from a secondary storage tank to which digestate is transferred after the cooling phase. To minimize changes in manure properties prior to use, the collected materials were stored at 2 °C.

Total ammoniacal N (TAN), total nitrogen (TN), pH, electrical conductivity (EC), dry matter (DM) and ash content of slurries and digestate were analyzed prior to incubation (Table 1). A distillation procedure (Gerhardt, Napodest 10s) was used for determination of slurry NH₄⁺-N concentrations (Sommer et al., 1992), and the Kjeldahl procedure (Foss, Kjeltecttm 2300) for TN. Slurry pH and EC were determined by a pH/Conductivity meter (CyberScan PC 300, EUTECH Instruments; Singapore). Dry matter was determined by drying c. 10 g fresh manure at 105 °C until constant weight, and ash

Table 1

Characteristics of digestate and untreated slurries (mean ± SD) used in the incubation experiment.

	Digestate	Pig slurry	Cattle slurry
Total N (g kg ⁻¹ fw)	3.45 ± 0.01	3.31 ± 0.00	2.97 ± 0.04
Ammoniacal-N (g kg ⁻¹ fw)	1.82 ± 0.01	2.54 ± 0.02	1.35 ± 0.03
pH	7.99 ± 0.02	7.68 ± 0.01	7.81 ± 0.02
EC (mS cm ⁻¹)	7.47 ± 0.03	10.26 ± 0.03	6.11 ± 0.05
Dry matter (g kg ⁻¹ fw)	60.91 ± 0.37	27.32 ± 1.58	83.09 ± 2.83
Volatile solids (g kg ⁻¹ fw)	43.61 ± 0.23	19.47 ± 0.76	61.37 ± 0.88
TOC (g kg ⁻¹ fw)	18.44 ± 0.60	9.66 ± 1.32	23.40 ± 1.70
VS _d (% TOC)	27.44 ± 9.44	74.37 ± 6.05	23.55 ± 5.27

EC, electrical conductivity; fw, fresh weight; TOC, total organic carbon; VS_d, easily degradable volatile solids.

content after an additional 6 h at 500 °C.

The fraction of easily degradable VS (VS_d) was estimated with an aerobic biodegradability test (see Supplemental Information for details). Briefly, net evolution of CO₂-C from manure applied to the sandy loam soil was determined by incubation for 26 d in a Respiromet VI respirometer (A. Nordgren Innovation AB, Bygdeå, Sweden), and total organic C in slurry or digestate using TOC cuvette test LCK 387 (DR 3900, HACH Lange, Germany). VS_d was then estimated as:

$$VS_{d,i} = \Delta CO_2 - C_i / TOC_i \quad (1)$$

where ΔCO₂-C is the evolution of CO₂-C from manure material *i* during a 26 d period after correction for emissions from an unamended control.

2.3. Incubation experiment

Main effects and interactions between manure type and soil moisture level with respect to N₂O emissions were examined in an incubation experiment with four manure treatments, i.e., a control (*Ctrl*; no amendment), digestate (*DI*), pig slurry (*PS*), and cattle slurry (*CS*), and three soil moisture levels (60, 70 or 80% WFPS). All 12 treatments were represented in each of three blocks, corresponding to replicates, which were initiated on consecutive days to make the sampling procedure manageable. Fifteen replicates of each treatment were prepared to allow for five destructive samplings.

For the experiment, sieved soil was packed to a bulk density of 1.3 g cm⁻³ in 100-cm³ stainless steel cylinders (inner diameter of 6.10 cm, and height of 3.42 cm) in four portions. Each portion was compressed with a purpose-fit piston and the surface then loosened by gentle scratching to improve contact with the next portion. Demineralized water and a 10 atom% K¹⁵NO₃ solution were added during packing of samples to introduce 25 mg NO₃⁻-N kg⁻¹ dry soil and reach the target WFPS (following addition of slurry/digestate). After 2 h the manure materials were applied to 50% of the soil surface. The application rate corresponded to 100 kg NH₄⁺-N ha⁻¹ if distributed to 25 cm depth, corresponding to 1.33 g NH₄⁺-N m⁻². Both ends of the cylinders were closed with perforated lids to allow for gas exchange while minimizing water loss. The samples were incubated at 20 °C in plastic boxes with a loosely fit cover and wet paper towels at the bottom to further minimize evaporation losses. The maximum loss of water by evaporation was 0.5 g sample⁻¹, which translates into c. 1% change in WFPS. Sampling took place after 1, 7, 14, 21 and 28 d incubation.

2.4. Sampling procedure and analyses

At the time of sampling, one replicate per treatment and block

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