Nitrous oxide flux from nitric-acid-treated cattle slurry applied to grassland under semi-controlled conditions

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Abstract

Nitrous oxide (N₂O) fluxes from cattle slurry after surface application to grassland were measured under semi-controlled environmental conditions during three periods in 1991. Three types of cattle slurry were examined: untreated slurry and slurries treated with nitric acid (HNO₃) to pH 6.0 and 4.5. Treatment with HNO₃ is a proposed technique to reduce ammonia volatilization from slurry during storage, and during and after surface application. N₂O flux was determined one to four times a day for 7 to 18 days after application of 0.64 to 3.8 kg slurry m⁻². Slurry-derived fluxes were much higher from treated slurries than from untreated slurries. Cumulative N₂O losses ranged from less than 0.2 % of the mineral N applied via untreated slurry to a maximum of 3.4 % of mineral N for treated slurry of pH 6.0, which was stored for one week before application. N₂O fluxes from slurry of pH 6.0 and those from slurry of pH 4.5 were similar, provided that the pH-6.0 slurry was treated one hour before application. Differences between treatments with treated slurry and those with calcium ammonium nitrate were relatively small; apparently the amount of NO₃ N applied controlled the N₂O flux. Regular irrigation strongly increased N₂O fluxes.

Keywords: nitrous oxide, grassland, cattle slurry, treated cattle slurry, nitric acid

Introduction

One of the measures to reduce ammonia (NH₃) emission from animal slurries is the acidification of these slurries. Studies in the United Kingdom have shown that acidification of animal slurry to pH 5.5-6.0 with sulphuric acid (H₂SO₄) reduced NH₃ losses by 30-95 % after surface application (Frost et al., 1990; Pain et al., 1990; Stevens et al. 1989; 1992). Similar results were obtained by Bussink & Bruins (1992) for slurries treated with nitric acid (HNO₃). Addition of HNO₃ also increases the fertilizer value of the slurry because the entrapped NH₃ and added nitrate (NO₃) are both directly available for uptake by plants.

However, addition of HNO₃ to generally anaerobic and carbon-rich slurry may promote denitrification, the microbial reduction of NO₃ into gaseous nitrous oxide (N₂O) and dinitrogen (N₂) (Firestone & Davidson, 1989). Denitrification leads to a loss of plant available N and also to an increase in slurry pH (Oenema & Velthof, 1993). Furthermore, there may be an increased emission of N₂O as it is one of the

volatile products of denitrification. Indeed, N₂O fluxes from cattle slurry treated with HNO₃ were much higher than those from untreated slurry during storage for up to six months (Oenema & Velthof, 1993). N₂O is suspected of a probable contribution to global warming and to destruction of ozone in the stratosphere (Bouwman, 1990). The aim of the experiments described here was to determine the N₂O flux from grassland after surface application of untreated slurry and slurries treated with HNO₃ to pH 6.0 and 4.5.

Currently, two variants of the slurry acidification procedure are tested in practice, i.e. acidification to pH 6.0 just before surface application and acidification to pH 4.0 to 4.5 immediately after deposition of the slurry by the cattle in the barn. In the latter variant, the HNO₃ treated slurry may be stored for up to 6 months before surface application. Slurries of both variants, with some modifications, were examined.

A soil cover chamber technique was chosen to measure the N₂O flux, because this technique is most appropriate for measuring small areas that are manipulated by fertilization (Mosier, 1989). A major problem in making reliable estimates of the flux from (un)fertilized grassland, based upon chamber measurements, is spatial variability. We assumed that this problem could be reduced by using a newly established sward on homogeneous soil in columns in which the groundwater level was fixed. We also assumed that irrigation increases soil moisture content and, thereby, N₂O flux.

Materials and methods

Materials

Measurements were carried out with cattle slurries in three experiments during periods from 10 to 17 June, 21 to 30 August and 30 September to 17 October 1991. The slurries were applied to a grass sward in stainless steel columns (internal diameter 20 cm, height 30 cm). Each column contained 12 kg sandy soil with a pH (KCl) of 4.74, 7.05 % organic matter, 4.6 % clay, 10.3 % silt, 59.1 % fine sand and 26.0 % coarse sand. Lolium perenne L. was the dominant grass species in the one-year-old sward. The columns were placed in the open, on a dish with 2 cm of water to keep the soil moisture status constant. During nights and during periods of rainfall, a mobile greenhouse covered the columns.

Cattle slurry was obtained from a local dairy farm. The slurry was acidified to pH 6.0 and 4.5 with 10.3 M HNO₃. In the first experiment, the slurry was acidified one week before application. In the second and third experiments slurries of pH 6.0 were acidified one hour and those of pH 4.5 one week before application. During storage at 15 °C for one week, the slurries were homogenized regularly and the pH was kept at the target pH by addition of HNO₃ if necessary. The untreated slurry was also homogenized regularly.

Concentrations of NH₄, NO₃ and N₂O in the slurries were measured according to the procedures described by Oenema et al. (1993). The chemical composition and application rates of the slurries in all three experiments are given in Table 1. The homogenized slurries were applied with a pipette to the sward, which was cut one day before application.

Experimental design

The first two experiments had a split-plot design with treatments in four replicates. Irrigation was the main factor. There were five treatments in type and rate of slurry: untreated slurry, treated slurry of pH 6.0, treated slurry of pH 4.5 at two application rates and a control. All these treatments were carried out with and without irrigation. When irrigated, treatments received about 5 mm water per day from a sprinkler installation. N₂O flux was measured once a day between 9.00 and 12.00 a.m., for 7 to 9 consecutive days.

The third experiment had a randomized block design with treatments in four replicates. The treatments were: untreated slurry, treated slurries of pH 6.0 and 4.5, calcium ammonium nitrate (CAN), a control and an additional treatment in which treated slurry of pH 4.5 was applied to inert quartzitic sand without a grass sward. The latter treatment was included to determine the effect of soil and sward on N₂O flux. All treatments were irrigated every two days with 5 mm water. N₂O fluxes were measured four times a day during the first four days, usually starting at 3.00 and 9.00 a.m., and at 3.00 and 9.00 p.m.; thereafter one to two times per day, starting at 9.00 a.m. and 15.00 p.m.

Flux measurements

Measurements were carried out with vented closed chambers (Mosier, 1989). The circular PVC chambers (internal diameter 25 cm and height 10 cm) had a collar of insulation foam attached to the bottom to minimize gas exchange with the outside atmosphere. The flux was calculated from the increase in N₂O concentration in the headspace of the chambers with time. Slurry-derived N₂O flux was calculated by substracting the average N₂O flux from the unfertilized control treatment.

In experiment I, the headspace of the chambers was sampled with 12 ml polypropylene syringes via rubber septa in the lid three times, usually after 0, 30 and 60 minutes. All samples were analyzed by gaschromatography within two days after sampling.

Because of the time consuming procedure and the possible errors introduced via sampling and sample storage, N₂O was measured with a photo-acoustic infra-red gas analyzer in experiments II and III. The analyzer was directly attached to the chambers, using polytetrafluorethylene tubes (internal diameter 0.3 cm, length 75 cm) with traps of soda lime and magnesium perchlorate to remove CO₂ and H₂O, respectively. A gas sample was taken and analyzed for N₂O automatically about every minute, after the air in the headspace of the chamber was pumped around for 30 s at a flow rate of 30 ml s⁻¹. All 5 or 6 treatments within one replicate were measured sequentially. This procedure was repeated four or five times, so that all treatments were measured four or five times at regular time intervals during an enclosure period of 30 to 40 minutes. Immediately thereafter, the treatments in the other three replicates were measured sequentially.

The increase in the N_2O concentration in the headspace was linear with time. Generally, the linear correlation coefficient (r^2) was ≥ 0.98 and the precision of the

calculated flux was high. However, if the N_2O concentration increased less than about 3 ppbv per minute and the flux was less than about 0.03 mg N m⁻² hr⁻¹, r² was usually less than 0.98 (Fig. 1). Thus, the precision of the calculated flux increased with increasing flux. The unit ppbv is equivalent to μl m⁻³.

Analytical procedures

NO₃ and NH₄⁺ were analyzed by standard auto-analyzer methods (Technicon: US no 824-87T and US no 795-86T, respectively). N₂O was measured via a PU 4400 gas-chromatograph equipped with 27.5 m Porapak Q wide bore column and a ⁶³Ni electron capture detector (ECD) or via a photo-acoustic spectroscopic Gas Monitor Type 1302 of Brüel & Kjær, equipped with the optical filters UA 0982, UA 0985 and SB 0527. Analysis of gas standards with N₂O concentation in the 300-1000 ppbv range, showed that the accuracy of the N₂O analyses was within 5 % when measured with the gas monitor in the field.

Results and discussion

Composition of slurries

The large differences in slurry composition (Table 1) were due to slurry origin and treatment. The initial pH, dry matter and NH₄ contents, and acid neutralizing capacity of the slurry of experiment I were higher than those of the slurries of experiments II and III. Differences between slurries within one experiment were the result of slurry acidification. Addition of HNO₃ increased the NO₃ content and decreased NH₃ volatilization so that treated slurries had higher NH₄ contents than untreated

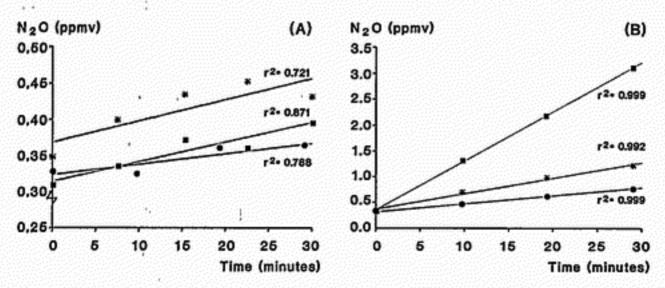


Fig. 1. Changes in the N₂O concentration in the headspace of vented closed chambers on a fertilized grass sward with time, illustrating the effect of flux rate on precision of the flux. Relatively low concentration increases and a corresponding low linear correlation coefficient (r²) are shown in A; rapid increases and high r² in B. The N₂O concentration was measured with a photo-acoustic infra-red gas analyzer directly attached to the chamber.

| Table 1. Some chemical properties and application rates of the slurries. | Samples were taken just before |
|--|--------------------------------|
| application to the grass sward. | |

| 100000000000000000000000000000000000000 | | | | | | <u> </u> | 21.4.2 |
|---|-----------|-----|--|---|---|--|---|
| Expt | Slurry | pН | Dry matter (g kg ^{-l}) | NH ₄ -N (g kg ⁻¹) | NO ₃ -N (g kg ⁻¹) | N ₂ O-N (mg kg ⁻¹) | Application rates (kg m ⁻²) |
| ſ | Untreated | 7.4 | 110 | 2.18 | 0.02 | 0.02 | 1.27 |
| | pH 6.0* | 6.0 | 113 | 2.40 | 1.94 | 6.34 | 1.27 |
| | pH 4.5* | 4.4 | 123 | 2.79 | 4.41 | 0.07 | 0.64 1.27 |
| II | Untreated | 7.0 | 90 | 1.32 | 0.00 | 0.01 | 1.27 |
| | pH 6,0** | 6.0 | 108 | 1.82 | 1.19 | 0.41 | 1.27 |
| | pH 4.5* | 4.5 | 109 | 1.89 | 3.58 | 0.13 | 0.64 1.27 |
| 111 | Untreated | 6.9 | 112 | 1.91 | 0.00 | _*** | 3.82 |
| | pH 6.0** | 6.0 | | 2.23 | 1.32 | <u> -</u> | 2.22 |
| | pH 4.5* | 4.5 | | 2.51 | 4.07 | | 1.27 |
| | CAN | _ | _ | 13.50 | 13.50 | _ | 0.03 |

^{*} Slurry acidified one week before application. ** Slurry acidified one hour before application. *** Not analyzed,

slurries, which contained no or only traces of NO3.

The dissolved N₂O content of the pH-6.0 slurry was 15 times higher in the first experiment than in the second experiment (Table 1). Apparently, N₂O accumulated in the slurry during storage for one week in the first experiment. The relatively low N₂O contents in the untreated and in the pH-4.5 slurries suggest that N₂O production rate was low in these slurries. Under conditions favourable for denitrification up to 500 mg N₂O-N per kg slurry can accumulate in slurries of pH 6.0 (Oenema & Velthof, 1993).

Variability of N2O fluxes

Fluxes of N₂O were rather low during most measurements, but in some treatments and on some dates, the flux was very high. The frequency distributions of the fluxes in the three experiments were highly skewed and lognormal.

The coefficient of variation of the average flux of four replicates ranged from 4 to 200 % with an average of 83 % (Fig. 2). A high coefficient of variation is typical for N₂O fluxes from grassland and other terrestrial systems, when measured with a flux chamber technique (Mosier, 1989). In an attempt to reduce the variability within treatments we performed the measurements under semi-controlled conditions, using thoroughly homogenized sandy soil with a uniform and constant soil moisture status. However, the results of this study suggest that the within-treatment variability may be also large in columns with uniform sward and treatment. Probably, slight differences between columns in percolation rate may have increased the variability. The variability within treatments remained high throughout the experimental period; columns with a relatively high flux at the start of the experiment kept a strong source

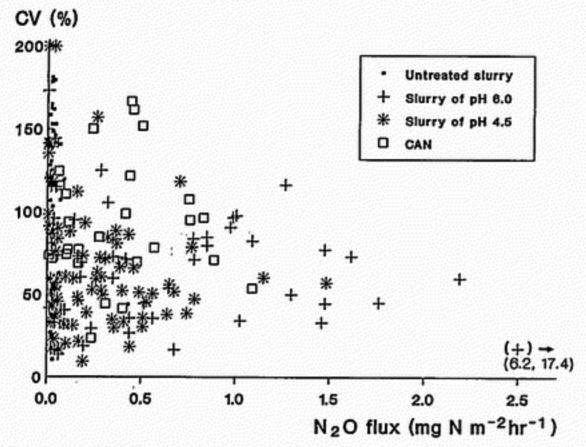


Fig. 2. Mean N₂O fluxes versus coefficients of variation (CV). Means and CVs were obtained from four flux measurements,

strength; low initial rates remained low.

Type of slurry and fertilizer did not clearly affect the coefficient of variation. Coefficients of variation slightly decreased with an increase in the N₂O flux (Fig. 2).

N2O fluxes: experiments I and II

Fluxes of N₂O from the control treatments were usually lower than 0.01 mg N m⁻² hr⁻¹. Slurry-derived fluxes from treatments with untreated slurry were also low, i.e. less than 0.04 mg N m⁻² hr⁻¹ in both experiments. Slurry-derived fluxes were 1 to 2 orders of magnitude higher from HNO₃ treated slurries than from untreated slurry in both experiments and for both pH levels.

There are two main biological sources of N₂O, viz. denitrification and nitrification (Firestone & Davidson, 1989). The fact that N₂O flux was high after application of treated slurry and low after application of NO₃ free untreated slurry suggests that denitrification was the dominant source of N₂O in the experiments. Nitrification of slurry NH₄⁺, possibly in combination with subsequent denitrification, was a minor source of N₂O, as total amounts of N₂O emitted after application of untreated slurry were low.

The mean flux from the pH 6.0 treatment was much higher in the first than in the second experiment (Fig. 3). This could be an effect of the duration of slurry treatment before application; in experiment I, HNO₃ was added one week, and in experiment II, one hour before application. Apparently, the denitrifying population was

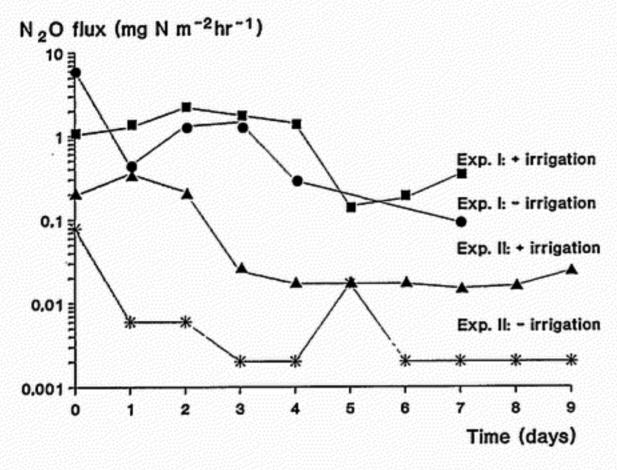


Fig. 3, Slurry-derived N₂O fluxes from treated cattle slurry of pH 6.0 applied at a rate of 1.27 kg m⁻² to the surface of a grass sward with and without daily irrigation. Means of four replicates, from experiments I and II. Note logarithmic scale of Y axis.

much more active one week after addition of HNO₃ than one hour after addition. The fact that the concentration of dissolved N₂O was higher in the one-week-old than in the one-hour-old pH-6.0 slurry (Table 1) supports this suggestion, just as the large flux immediately after application of one-week-old slurry (Fig. 3).

In experiment I, N₂O flux was lower from the pH 4.5 treatment than from the pH 6.0 treatment (Table 2). In experiment II, fluxes showed little variation, indicating that slurry acidified to pH 6.0 one hour before application behaved similar to one-week-old slurry of pH 4.5.

An increase in application rate from 0.64 to 1.27 kg m⁻² increased the flux from the pH 4.5 treatment (Figure 4). This agrees with the conclusion of Eichner (1990) and Bouwman (1990) that N₂O fluxes generally increase with increasing N application rate.

Irrigation increased N₂O fluxes from all treatments in experiments I and II (Fig. 3). In some treatments irrigation increased fluxes by more than one order of magnitude, The effect of irrigation may suggest that denitrification was the dominant source of N₂O. Water in slurry and soil may act as a barrier to oxygen (O₂) diffusion and may stimulate microbial consumption of trapped O₂, resulting in reducing conditions and increased denitrification in slurry and soil.

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Table 2, Summed slurry- and CAN-derived N₂O losses (mg N m⁻²) and as a percentage of mineral N and NO₃-N applied during the three experiments.

| Expt | Treatment | N application rate | | Regular irrigation | N ₂ O-N emitted | | |
|------|------------------|---------------------------------|---|-----------------------|----------------------------|----------------|--------------------|
| | | | | | mg m ⁻² | % of N applied | |
| | | mineral (g m ⁻²) | NO ₃ (g m ⁻²) | | | mineral | NO ₃ -N |
| I | Untreated | 2.8 | 0.0 | _ | <2 | <0.1 | _ |
| | | | | + | <3 | <0.2 | _ |
| | pH 6.0 | 5.6 | 2.5 | <u> </u> | 163 | 2.9 | 6.5 |
| | | | | + | 193 | 3.4 | 7.7 |
| | pH 4.5 | 9.1 | 5.6 | | <6 | <0.1 | <0.1 |
| | | | | + | 48 | 0.5 | 0.9 |
| | pH 4.5 | 4.6 | 2.8 | | <2 | <0.1 | <0.1 |
| | | | | + | 36 | 8.0 | 1.3 |
| 11 | Untreated | 1.7 | 0.0 | _ | <2 | <0.2 | _ |
| | • | | | + | 6 | 0.4 | _ |
| | pH 6.0 | 3.8 | 1.5 | <u> </u> | <3 | <0.1 | <0.2 |
| | | | | + | 21 | 0.6 | 1.4 |
| | pH 4.5 | 7.0 | 4.6 | _ | <3 | <0.1 | <0.1 |
| | | | | + | 26 | 0.4 | 0.6 |
| | pH 4.5 | 3,5 | 2.3 | - | <1 | <0.1 | <0.1 |
| | | | | + | 14 | 0.4 | 0.6 |
| ш. | Untreated | 7.3 | 0.0 | + | 10 | 0.1 | _ |
| | pH 6.0 | 7.9 | 2.9 | + | 107 | 1.4 | 3.7 |
| | pH 4.5 | 8.4 | 5.2 | + | 132 | 1.6 | 2.5 |
| | CAN pH 4.5 on | 7.2 | 3.6 | + | 90 | 1.2 | 2.5 |
| | sand only | 8.4 | 5.2 | + | 9 | 0.1 | 0.2 |

N2O fluxes: experiment III

In experiment III, N₂O fluxes increased in the order: untreated slurry, CAN, treated slurries (Fig. 5A and Table 2). The low but significant slurry-derived flux from the NO₃ free untreated slurry suggests that N₂O was released from nitrification of NH₄, possibly in combination with subsequent denitrification.

The flux from treated slurries immediately after application, probably originated from the N₂O pool in the slurries. No flux could be detected immediately after application of CAN. The highest slurry- and CAN-derived fluxes occurred during the second day. After this peak, fluxes decreased gradually with time and 17 days after application fluxes from fertilized columns were similar to those from unfertilized columns. There was no clear diurnal pattern in fluxes related to changes in soil temperature (Fig. 5A and B) as found by Blackmer et al. (1982) and Denmead et al. (1979). Possibly, the effect of temperature variations was masked by the effect of regular irrigation.

Fluxes from slurry of pH 4.5 were equal to or only slightly higher than those from

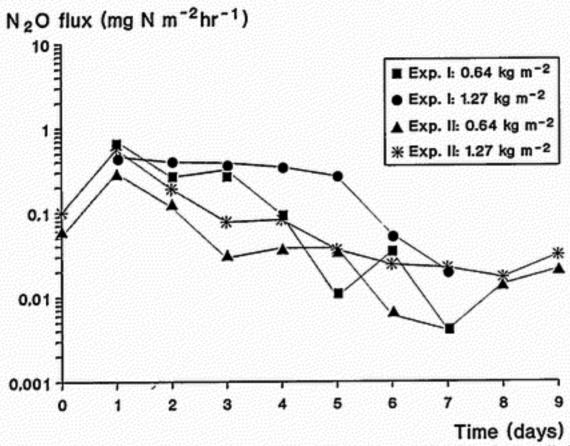


Fig. 4. Slurry-derived N₂O fluxes from treated cattle slurry of pH 4.5 surface-applied to a grass sward. Slurry was applied at rates of 0.64 and 1.27 kg m⁻². All treatments were irrigated daily. Means of four replicates, from experiments I and II. Note logarithmic scale of Y axis.

the slurry of pH 6.0. This corresponds with the results of experiment II and indicates that there was not an immediate effect of pH on N_2O flux, provided that slurry of pH 6.0 was treated immediately before application.

Surprisingly, fluxes from CAN were on average only slightly lower than those from treated slurries. This suggests that the sandy soil contained sufficient amounts of readily available carbon and sites of anaerobiosis for denitrification and N₂O production. With treated slurry both NO₃ and available carbon are applied. Application of slurry may also seal the soil, thereby creating anaerobic microsites in the soil with high denitrification activity, due to decreased O₂ diffusion and increased O₂ consumption (Beauchamp et al., 1989). The relatively small differences between CAN and treated slurries indicates that application of NO₃ was the most critical factor and that the soil was the dominant site of N₂O production.

The flux pattern of the treatment with pH-4.5 slurry applied to quartzitic sand is shown in Fig. 6. The flux from quartzitic sand was assumed to be negligible. During the first days no significant amounts of N₂O were emitted, except immediately after slurry application. A small but significant flux was measured from day 5 onwards. Apparently, it took some time for N₂O production to start in the sand-slurry environment. However, the fluxes were much lower from the quartzitic sand than from the sandy soil with sward. There are several possible explanations for these low fluxes, like the absence of soil organic matter, roots and a well-established denitrifying bacteria population in the quartzitic sand. Albeit unclear, the results of this treatment

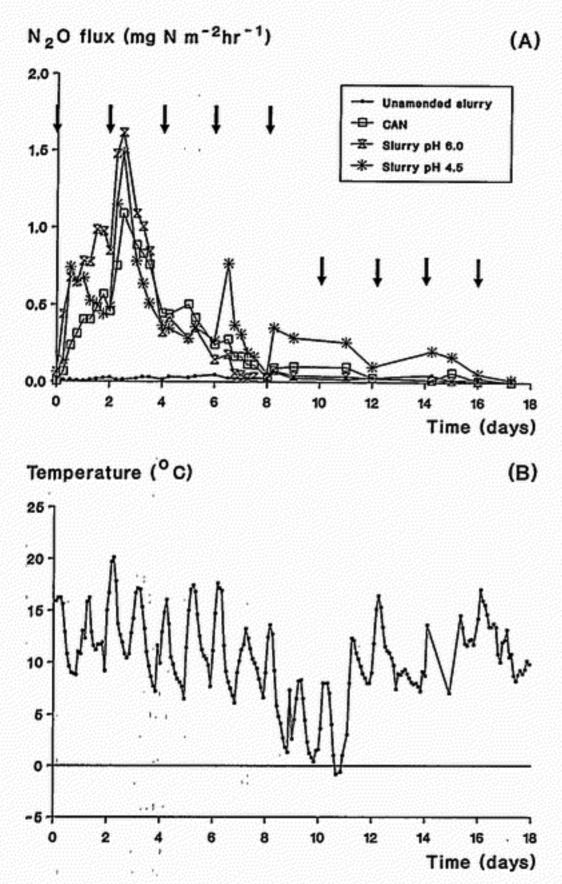


Fig. 5. (A) Slurry-derived N₂O flux from untreated slurry, treated slurries of pH 6.0 and pH 4.5 and fertilized-derived flux from CAN, surface-applied to a grass sward. Arrows indicate time of irrigation. Means of four replicates, from experiment III. (B) Course of the air temperature during experiment III.

confirm the suggestion that HNO₃ treated slurry itself was not the dominant site of N₂O production.

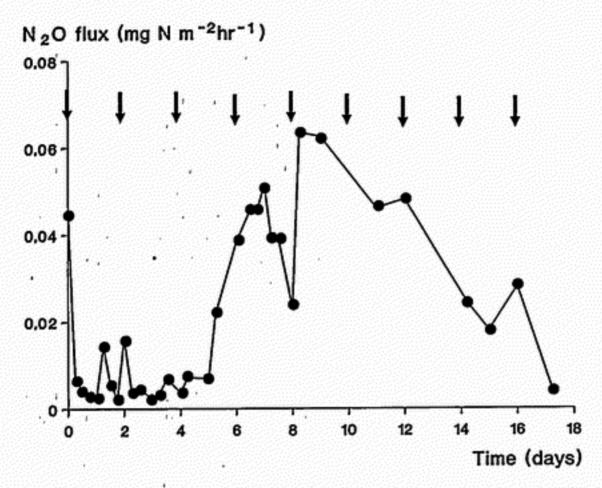


Fig. 6. Flux of N₂O from slurry of pH 4.5 surface-applied to quartzitic sand without a grass sward. Means of four replicates, from experiment III.

Summed N2O losses

The summed slurry-derived N₂O losses were estimated by integrating the N₂O flux over time. Summed losses were lower in experiment II than in experiments I and III (Table 2). Part of this difference may be attributed to differences in slurry composition and in the duration of the measurement periods. Differences in weather conditions and temperature may also have contributed (Table 3). During the warm and sunny weather conditions of experiment II, grass growth and N uptake was faster than during the colder and cloudy weather conditions of experiments II and III. Obviously, the faster N is absorbed by a crop, the smaller the risk of denitrification and N₂O production. The overall effect of temperature on N₂O flux is difficult to predict.

Table 3, Mean, minimum and maximum temperatures during experiments I, II and III.

| Expt | | | Temperature | Temperature (°C) | | | | |
|------|------------|-----|-------------|------------------|---------|--|--|--|
| e e | • | , , | mean | minimum | maximum | | | |
| I, | 10/6-17/6 | | 13.4 | 7.2 | 20.2 | | | |
| 11, | 21/8-30/8 | | 19.3 | 12.3 | 31.4 | | | |
| III. | 30/9-17/10 | | 10.3 | -0.8 | 20.1 | | | |

The denitrification rate increases with increasing temperatures, but the ratio between N₂O and N₂ produced during denitrification generally decreases (e.g. Keeney et al., 1979).

When irrigated, N₂O fluxes (mg N m⁻² hr⁻¹) and summed N₂O losses (in % of N applied) were relatively high, but both are in the range of data reported in the literature (Eichner, 1990; Bouwman, 1990). Under non-irrigated conditions, losses were low. From an agricultural point of view, the total N₂O-N losses were relatively small; less than 3.4% of mineral N applied was emitted as N₂O (Table 2). However, the proportion of N₂O in the total N loss through denitrification may range from less than 5 up to more than 50 % (Ryden & Rolston, 1983). Hence, N₂O-N losses are a lower estimate of total N losses. The wide range indicates that it is impossible to deduce accurately total N losses from N₂O production data only.

Conclusions

The major conclusions from these experiments are:

- Coefficients of variation of average N₂O fluxes were relatively independent of slurry type and flux and, under the semi-controlled conditions of our experiments, as high as those reported for field conditions.
- HNO₃ treated cattle slurry emitted much more N₂O than untreated slurry; the amounts emitted by CAN were similar to those of treated cattle slurry.
- Irrigation markedly increased N₂O fluxes from HNO₃ treated slurries and CAN.
- N₂O fluxes were highest during the first three days after application; subsequently decreased to rates equal to that of the unfertilized treatments within 5-14 days.
- Slurry pH had no clear effect on N₂O flux if the slurry was treated one hour before application; if treated one week before application, fluxes were much higher from pH-6.0 slurry than from pH-4.5 slurry.

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