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Nitrous oxide emission from soil and from a nitrogen-15-labelled fertilizer with the new nitrification inhibitor 3,4-dimethylpyrazole phosphate (DMPP)

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Abstract Mineral-N fertilization can lead to a short-term enhancement of N₂O emission from cultivated land. The aim of this field study was the quantitative determination of the short-term N₂O emission after application of a fertilizer with the new nitrification inhibitor (NI) 3,4-dimethylpyrazole phosphate (DMPP) to winter wheat. NO₃⁻ and NH₄⁺ fertilizers labelled with ¹⁵N in liquid and granulated form were used in specific fertilizer strategies. N fertilizers with higher NO₃⁻ contents caused higher N₂O emission than NH₄⁺ fertilizers. For fertilizers with NIs, used in simplified fertilizer strategies with fewer applications and an earlier timing of the N fertilization, the N₂O release was reduced by about 20%. Of the total N₂O emission measured, 10–40% was attributed to fertilizer N and 60–90% originated from soil N. Besides the fertilizer NO₃⁻-N, the microbial available-N pool in the soil represented a further important source for N₂O losses. Compared to liquid fertilizers, the application in granulated form led to smaller N₂O emissions. For fertilizers with NIs, the decrease in the N₂O emission is mainly due to their low NO₃⁻-N content and the possibility of reducing the number of applications.

Keywords 3,4-Dimethylpyrazole phosphate · Inorganic fertilizer · Nitrification inhibitor · Nitrous oxide · Labelled nitrogen

Introduction

The release of N₂O, a trace gas which affects the global climate, is increasing due to more intensive agricultural practices. The contribution of agriculture to the global annual N₂O emission is estimated at approximately 35% (Isermann 1994). The amount of N₂O released is usually

related to the input of N in organic and mineral fertilizers and depends, in addition, on the form in which N is applied, on the location (soil and climate) and on the cultivated crop (Corre et al. 1995; Kaiser et al. 1998; Mac Kenzie 1998). The extensive survey by Eichner (1990) shows the enormous range of N₂O losses derived from fertilizers, together with different local conditions and forms of fertilizers. Kaiser et al. (1996) indicated a range of N₂O emissions for nine European agricultural soils of 0.2–6.6 kg N₂O-N ha⁻¹ year⁻¹, which seems to be low in relation to the findings of other studies (Granli and Bøckman 1994).

For several fertilizers formulated with nitrification inhibitors (NIs), a decreasing effect on N₂O losses has been shown (Bremner and Blackmer 1978; Bronson et al. 1992; Mosier 1994; De Klein et al. 1996; Delgado and Mosier 1996; Mosier et al. 1996; Michel and Wozniak 1998). A new, very effective NI is now available: 3,4-dimethylpyrazole phosphate (DMPP) (Zerulla et al. 2001). Specific application strategies for the use of stabilized fertilizers (NI-containing fertilizers) (Trenkel 1997) have been developed with the benefits of improved yield and reduced N losses by NO₃⁻ leaching, compared to conventional fertilizers (Gutser 1999; Linzmeier et al. 1999). The reduction in the number of fertilizer applications and the opportunity to apply fertilizer earlier, are further advantages of using stabilized fertilizers.

The aim of this study was to evaluate the short-term emission of N₂O after the application of fertilizer containing the new NI DMPP to winter wheat over 2 years of field trials. A decrease in N₂O emissions might be expected with stabilized fertilizers, particularly if the number of N applications can be reduced. The form of the fertilizer N (mainly NH₄⁺-N or NO₃⁻-N) is also likely to have an effect on the amount of N₂O released. The fertilizer-N- or soil-N-derived N₂O emissions were determined using ¹⁵N tracers.

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Materials and methods

Location and soil

The trials were conducted at the research station of the Chair of Plant Nutrition at the Technical University of Munich-Weihenstephan (Bavaria). Geologically the location is situated in the Tertiary hill landscape. The average annual precipitation is 800 mm and the average air temperature (2 m above soil) is 7.4°C. The soil is a brown earth with a silty loam texture (24% clay, 51% silt, 25% sand), an organic matter content of 2.3% and a pH value (in 0.01 M CaCl₂) of 6.4.

Experimental setup

Trials using top-dressed (160 kg N ha⁻¹) winter wheat (*Triticum aestivum* L. var. Astron) were conducted for 2 years (1998–1999) on different plots (Table 1). In these experiments, N as ammonium sulphate nitrate [NH₄NO₃(NH₄)₂SO₄; ASN] and as calcium ammonium nitrate [NH₄NO₃+CaCO₃; CAN] fertilizers was used. The NIs dicyandiamide (DCD) or DMPP were added to ASN (liquid form) and formulated on the fertilizer granules of ASN (granulated form), respectively. The fertilizers and NIs were provided by BASF (BASF Agricultural Centre, Limburgerhof, Germany).

Two investigations were carried out: in the first experiment (1998), the fertilizer ASN with and without NI (DMPP and DCD) was applied in a single application at the beginning of spring growth (27 March). The fertilizers were applied either in liquid (labelled N) or in granulated form. In the second experiment (1999), the most widely practised fertilizer system for winter wheat in this region was compared to that of a stabilized fertilizer, used in a reduced number of applications: CAN, split into three applications and ASN/DMPP, split into two applications. The ¹⁵N-labelled fertilizers were first dissolved in distilled water and then applied with a dispenser by spiking the plot in a symmetrical pattern. In 1998, 10 atom% ¹⁵NH₄⁺-N was applied to ASN (with or without NI). In 1999, the CAN and ASN/DMPP were double-labelled with ¹⁵NH₄⁺-N and ¹⁵NO₃⁻-N enriched to 30 atom% ¹⁵N.

Gas sampling

The N₂O fluxes were measured using the closed-chamber technique described by Hutchinson and Mosier (1981). In the randomized field experiments, in specific microplots, the gas sampling was replicated 8 times for each treatment.

Analysis

The gas samples (20 ml) were analysed by a mass spectrometer (ANCA-MS; Europa Scientific TG 20–20, Crewe, UK) to determine the total quantity of N₂O and the proportion of ¹⁵N. The inorganic N in the soil (N_{min}) was determined for NO₃⁻ by HPLC, after a 1-h extraction with 0.01 M CaCl₂ solution (soil:extracting solution=1:5) and for NH₄⁺, photometrically after a 1-h extraction with 1 M KCl solution (soil:extracting solution=1:5).

Statistical analysis

The statistical variance analysis was performed using the GLM procedures of the Statistical Analysis System (SAS Institute, Cary, N.C.). The minimum significant difference (*P*<0.05) was determined using Tukey's test of honestly significant difference.

Results

Effect of the stabilized NH₄⁺

The effect of the NIs DCD and DMPP on N₂O emission after the application of equal amounts of ASN at the beginning of spring growth was mainly investigated in 1998. In this experiment, the ¹⁵N enrichment was added to the fertilizer as NH₄⁺-N. Basically, the N₂O losses derived from the fertilizer NH₄⁺-N (Fig. 1) were very low, and reached approximately 5% of the total N₂O emission (Fig. 2). Within 4 weeks after fertilization, the NH₄⁺-N-fertilizer-derived N₂O losses were considerably reduced by about 50% by the NIs (Fig. 1). The course of the total N₂O emission (Fig. 2) differed significantly from the emission of the fertilizer NH₄⁺-N (Fig. 1).

Split application of fertilizer and form of N

In the experiment of 1998, for ASN and ASN/NI, significant N₂O peaks were detected within the first 3–4 weeks after fertilization (Fig. 2). On the whole, the time course

Table 1 Treatments and fertilizer characteristics. ASN Ammonium sulphate nitrate, NH₄NO₃·(NH₄)₂SO₄; DCD dicyandiamide; DMPP 3,4-dimethylpyrazole phosphate; CAN calcium ammonium nitrate, NH₄NO₃+CaCO₃

Treatment	Fertilizer form	Total N content (%)	N ratio NH ₄ ⁺ /NO ₃ ⁻ (%)	Nitrification inhibitor	¹⁵ N enrichment	Fertilizer: split N application (kg ha ⁻¹)
1998						
0	–	–	–	–	–	–
ASN	Liquid	26	71/29	–	NH ₄ ⁺ (10 atom%)	160
ASN/DCD	Liquid	27	71/29	DCD (17.35) ^b	NH ₄ ⁺ (10 atom%)	160
ASN/DMPP	Liquid	26	71/29	DMPP (1.61)	NH ₄ ⁺ (10 atom%)	160
ASN/DMPP	Granules	26	71/29	DMPP (1.61)	–	160
1999						
0	–	–	–	–	–	–
CAN ^a	Liquid	27	50/50	–	NH ₄ ⁺ /NH ₃ ⁻ (30 atom%)	70/40/50
ASN/DMPP	Liquid	26	71/29	DMPP (1.61)	NH ₄ ⁺ /NH ₃ ⁻ (30 atom%)	110/50

^a Equivalent amounts of SO₄⁻-S as for ASN treatments were given

^b Values in parentheses indicate the quantity of the nitrification inhibitor as percent of NH₄⁺-N

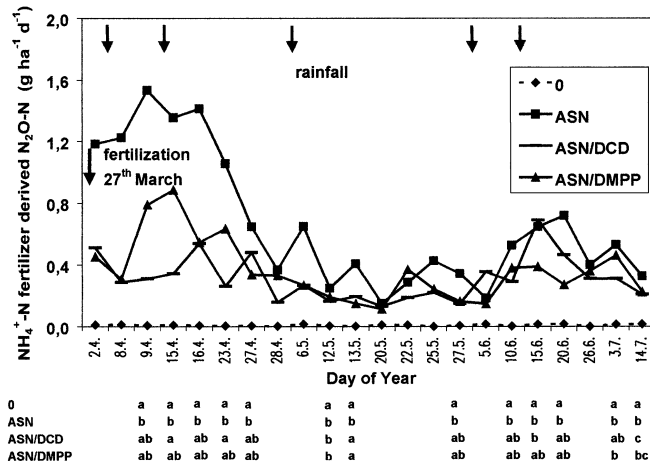


Fig. 1 N_2O emission from the fertilizer $\text{NH}_4^+\text{-N}$ (^{15}N tracer) after a single application of 160 kg N ha^{-1} of different N fertilizers in liquid form at the beginning of spring growth in 1998. *a, b, c* indicate significant differences ($P < 0.05$) among the treatments at the respective dates. 0 No N, ASN ammonium sulphate nitrate, $\text{NH}_4\text{NO}_3 \cdot (\text{NH}_4)_2\text{SO}_4$; ASN/DCD ASN plus dicyandiamide; ASN/DMPP ASN plus 3,4-dimethylpyrazole phosphate; *d* day

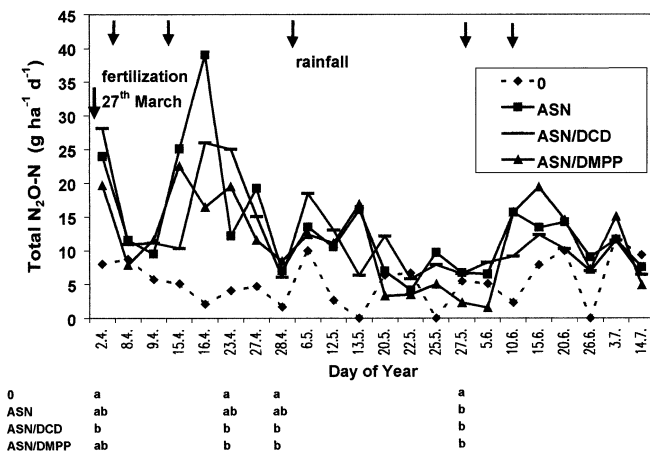


Fig. 2 Total N_2O emission (Σ fertilizer N and soil N) after fertilization with 160 kg N ha^{-1} of different N fertilizers in liquid form in a single application at the beginning of spring growth in 1998. *a, b, c* indicate significant differences ($P < 0.05$) among the treatments at the respective dates. For abbreviations, see Fig. 1

and the quantity of the N_2O emissions were not strongly influenced by the NIs. The quantity of N_2O emission was mainly affected by the amount of applied $\text{NO}_3^- \text{-N}$. At the beginning of spring growth the N treatments received equal amounts of $\text{NO}_3^- \text{-N}$.

Two fertilizing systems (CAN and ASN/DMPP) and one unfertilized treatment were compared in 1999. The fertilizing systems mainly differed in the relation of $\text{NH}_4^+\text{-N}$ to $\text{NO}_3^- \text{-N}$, the timing and the number of applications (Table 1). After the first application, at the beginning of spring growth (26 March), the N_2O emission of both fertilized treatments increased similarly up to a maximum emission rate of approximately $30 \text{ g N}_2\text{O-N ha}^{-1} \text{ day}^{-1}$ (Fig. 3). For the first fertilizer application, the

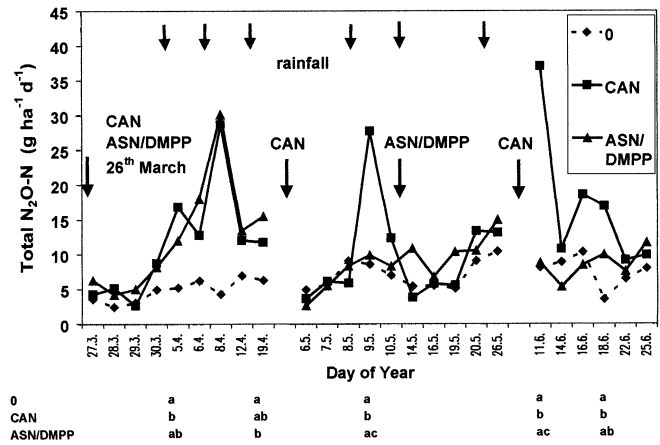


Fig. 3 Total N_2O emission (Σ fertilizer N and soil N) after application of 160 kg N ha^{-1} of different N fertilizers in liquid form with a fertilizer-specific split application of N in 1999. *a, b, c* indicate significant differences ($P < 0.05$) among the treatments at the respective dates. CAN Calcium ammonium nitrate, $\text{NH}_4\text{NO}_3 + \text{CaCO}_3$, in three applications (70/40/50); ASN/DMPP ASN plus DMPP in two applications (110/50); for other abbreviations, see Fig. 1

applied amount of $\text{NO}_3^- \text{-N}$ was almost the same for both systems: 35 kg as NO_3^- for CAN (70 kg N ha^{-1}) and about 32 kg as NO_3^- for ASN/DMPP (110 kg N ha^{-1}).

The following fertilizer applications were done at different dates: CAN – 4 May and 10 June, ASN/DMPP – 12 May. For CAN, the short-term N_2O emission increased considerably ($28 \text{ g N}_2\text{O-N ha}^{-1} \text{ day}^{-1}$ and $37 \text{ g N}_2\text{O-N ha}^{-1} \text{ day}^{-1}$) after the second and third application of fertilizer (40 kg N ha^{-1} and 50 kg N ha^{-1}), respectively. For ASN/DMPP, only a small peak was detectable following the second fertilizer application (50 kg N ha^{-1} , of which 14.5 kg was $\text{NO}_3^- \text{-N}$). Owing to the significant peaks following the second and third fertilizer application, the CAN treatment resulted in a 20% higher total N_2O emission than the ASN/DMPP treatment. On the unfertilized plot the N_2O emission rates increased during the growing season until the end of May (Fig. 3).

N_2O derived from soil N and fertilizer N

The use of ^{15}N -labelled fertilizer enables differentiation between fertilizer-N- or soil-N-derived N_2O emissions (Fig. 4). The course of the fertilizer-derived N_2O losses corresponded to the total N_2O emission (see Fig. 3). About 10 days after the first fertilization the highest N_2O fluxes were $10.4 \text{ g N}_2\text{O-N ha}^{-1} \text{ day}^{-1}$ for CAN and $12.8 \text{ g N}_2\text{O-N ha}^{-1} \text{ day}^{-1}$ for ASN/DMPP. Furthermore, significant peaks occurred after the application of CAN. The proportion of the fertilizer-N-derived N_2O from the total N_2O emission reached 30–40% and later decreased to 10–20%. The remaining part has to be assigned to soil N. Therefore, the emission from soil increased considerably in the later measuring periods.

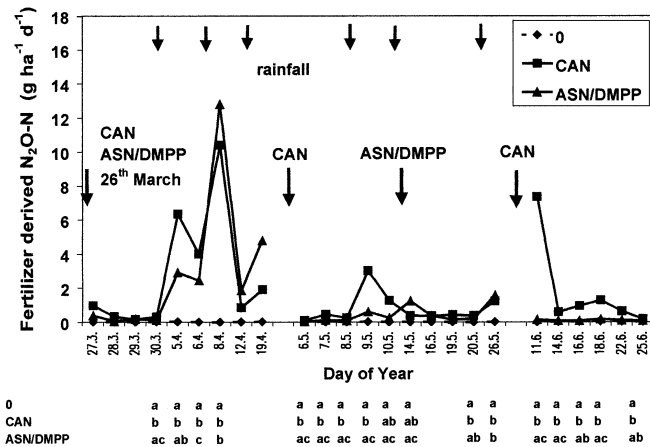


Fig. 4 N₂O emission from the fertilizer N (¹⁵N tracer) after application of 160 kg N ha⁻¹ of different N fertilizers in liquid form with a fertilizer-specific split application of N [CAN in three applications (70/40/50); ASN/DMPP in two applications (110/50)] in 1999. *a, b, c* indicate significant differences (*P*<0.05) among the treatments at the respective dates. For abbreviations, see Figs. 1 and 4

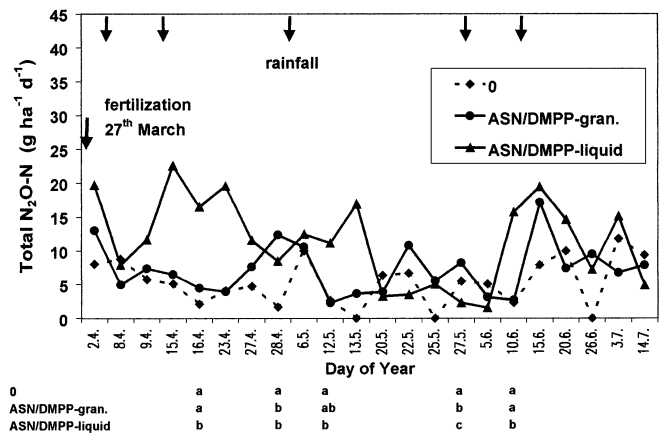


Fig. 5 Total N₂O emission after fertilization with 160 kg N ha⁻¹ of ASN/DMPP in a single application in liquid and granulated form at the beginning of spring growth in 1998. *a, b, c* indicate significant differences (*P*<0.05) among the treatments at the respective dates. For abbreviations, see Fig. 1

Influence of rainfall events on N₂O emission

In both years, the total N₂O emission as well as the fertilizer-N-derived N₂O emission were influenced by the weather conditions. Therefore, a significant N₂O release was not always found immediately after fertilization, but quite often after intensive rainfalls. In 1999, during the weeks following the fertilization, in spite of high NO₃⁻ concentrations in soil, N₂O was emitted only in combination with intensive rainfall events (Figs. 3, 4), which possibly led to partial water saturation in soils. The significant N₂O emission after the second and third CAN fertilization in May and June was also based on similar conditions (NO₃⁻-N and rainfall). In 1998, marked increases in N₂O emission rates occurred after rainfall events, which were independent of fertilization. In particular, the N₂O emission increased after high rainfall also on the unfertilized plot (Figs. 2, 5, sampling dates 6 May and 15 June).

Granules and liquid fertilizers

ASN/DMPP was also used as granules in the investigation of 1998. N₂O emission for the granulated form tended to be lower than for the liquid application (Fig. 5). This was mainly noticeable in the initial part of the measuring period. The treatment with granules did not differ from the unfertilized treatment, whereas the liquid fertilization showed remarkable higher N₂O losses.

Inorganic N in the soil

On CAN- and ASN/DMPP-fertilized plots, completely different time courses of NH₄⁺ and NO₃⁻ contents were

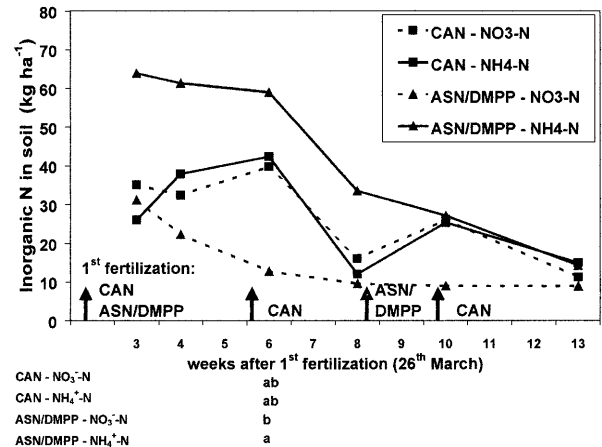


Fig. 6 Typical course of the NH₄⁺ and NO₃⁻ contents in the topsoil (silty loam) after fertilization of winter wheat with 160 kg N ha⁻¹ as CAN and ASN/DMPP in granulated form [CAN in three applications (70/40/50); ASN/DMPP in two applications (110/50)] in 1998. *a, b, c* indicate significant differences (*P*<0.05) among the treatments at the respective sampling dates. For abbreviations, see Figs. 1 and 4

found in the topsoil, probably influencing the N₂O release (Fig. 6). CAN generally led to higher NO₃⁻ contents in contrast to ASN/DMPP, with contents rapidly decreasing to a constant low level. Furthermore, rather constant NH₄⁺ contents were representative of ASN/DMPP, due to the NI DMPP, which resulted in the inhibition of nitrification which lasted for approximately 8 weeks. In particular, the subsequent applications of the fertilizer CAN produced higher NH₄⁺ and NO₃⁻ contents in the topsoil.

Discussion

In the 2 experimental years a remarkable influence of intensive rainfall events on N_2O emission was established. Despite high supplies of NO_3^- -N by fertilization, enhanced N_2O emission was determined only in combination with rainfalls and, therefore, mainly resulted from denitrification losses. Changing (aerobic/anaerobic) conditions in the soil favour the release of N_2O (Granli and Bøckman 1994). The effect of the rewetting of soil was recognized by an increase in the N_2O emission on the unfertilized plots.

The timing of fertilization and the amount of NO_3^- -N applied were found to be the main decisive factors for the quantity of N_2O emitted. The N_2O losses for CAN, split into three applications, compared to ASN/DMPP, split into two applications, were about 20% higher in 1999. After the first fertilization, the N_2O emission from the fertilizer systems with CAN and ASN/DMPP only differed marginally owing to similar amounts of applied NO_3^- -N. For the second and third fertilization, due to different NO_3^- -N applications, the N_2O emission increased in CAN-fertilized plots, but not in ASN/DMPP plots. For CAN, a rapid nitrification of the NH_4^+ -N was to be expected, resulting in rising NO_3^- concentrations in the soil. For ASN/DMPP, high NO_3^- concentrations in the soil were not to be expected after the second fertilization, due to the low NO_3^- content of the fertilizer and the retarded nitrification of the NH_4^+ -N by the NI DMPP (see Fig. 6).

When ASN was applied in a single spring application in 1998, the influence of NIs on the course of N_2O release differed only slightly. In contrast to our experiments with the application of DMPP in liquid form, Weiske et al. (2001) found a higher reduction of N_2O losses with single applications of DMPP formulated on ASN granules. Site-specific and environmental differences as well as the form in which the NI (liquid or granulated) was applied could account for these differences. Both studies show that a decreased number of fertilizer applications reduces N_2O losses.

The proportion of the soil-N-derived N_2O losses increased from 60% up to 90% for the subsequent fertilizer applications (mainly for CAN). Increased N_2O emission rates occurred after fertilization with NO_3^- -N. The N turnover in the soil was possibly stimulated by fertilization (Jenkinson et al. 1985) and developed an enhanced potential for N_2O losses. NO_3^- -N is subjected to losses as a consequence of denitrification, and due to its higher mobility, a larger area in the soil is included in a potential "priming effect" than, for example, for NH_4^+ -N. Fertilizers with NI (ASN/DMPP) showed this effect to a lesser extent (lower NO_3^- concentrations in the soil – see Fig. 6), thus the N_2O emission after fertilization decreased.

Fertilizer NH_4^+ -N had only a minor effect on the short-term N_2O release (3–4 weeks after fertilization) (Figs. 1, 2). NIs resulted in additional decreases in N_2O losses due to the inhibition of nitrification, a low NO_3^- -

concentration in the soil and therefore a reduction in the source for N_2O losses. In earlier experiments, we found enhanced N_2O emissions after late NO_3^- -N applications to wheat which was producing shoots, whereas an early application of NH_4^+ -N with an NI only caused a moderate N_2O release (Linzmeier et al. 1999). The immediate effect of the fertilizers on N_2O losses was small, whereas the indirect effect of influencing the N pool of the soil was of major importance.

The insignificant differences in N_2O emissions between ASN with and without NI can be explained as follows: no difference existed between the applied amounts of NO_3^- -N, which proved to be decisive for N_2O release. Moreover, the usually lower temperatures in spring generally slowed down nitrification, whereby the diminished effect of the NI on nitrification also became less important. As a consequence of drier conditions in May, no considerable denitrification was to be expected.

The results relating to the influence of the form in which the fertilizer was applied (liquid or granulated), with a considerably decreased N_2O release in the case of the granulated form, are quite interesting. This favourable effect might be attributed to several causes. Fertilizer granules stimulate the N turnover mainly in a few "hot spots". In particular, this seems to be very important in contrast to the mobility of NO_3^- -N (Wang et al. 1998). For granules, a dispersal of the fertilizers' NH_4^+ -N and NI is prevented, resulting in a longer lasting inhibition of nitrification and lower NO_3^- concentrations in the soil. Additionally, another positive aspect is the low mobility of DMPP in soil (Fettweis et al. 2001), in contrast to that of DCD (Abdel-Sabour et al. 1990). Further studies are required to consistently demonstrate positive effects of DMPP formulated on granules as compared to its application with fertilizers in a liquid form. The latter form is the preferred one for experimental reasons, however, most frequently solid fertilizers are used in conventional fertilization.

The soil-N-derived N_2O emission varying between 60–90% probably depends on the importance of the microbial available-N pool in the soil as a source for N_2O losses in interaction with the N fertilization. The regression set up by Bouwman (1996) between N_2O emission and N input [N_2O emission ($kg N_2O-N ha^{-1} year^{-1}$) = $1 + 0.0125 \times N$ input ($kg N ha^{-1} year^{-1}$)] enables only a rough estimate to be made of the N_2O emission. On the basis of long-term areal N balances (fertilizer-derived-N input minus N yield of the crops) the efficiency of the N fertilization and the available N pool are likely to improve the assessment. This is to be expected particularly for soils undergoing intensive organic fertilization.

Even though on a long-term basis organic fertilization is of great significance for N_2O release, short-term N_2O emission is also considerably influenced by mineral fertilization. These N_2O losses can be reduced by selecting the appropriate N form, in particular by using stabilized (DMPP) NH_4^+ fertilizers along with specific fertilizer strategies.

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