Ammonia volatilisation from pig slurry and ANS with DMPP applied to Westerwold ryegrass (*Lolium multiflorum* Lam., cv. Trinova) under Mediterranean conditions

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Abstract

Ammonia volatilisation from agriculture represents an important nitrogen (N) loss with both environmental and economic impacts. In regions with large amounts of manures available, there is a need to find appropriate management strategies that help to reuse them without increasing ammonia volatilisation. A study was made of the effect on ammonia volatilisation and yield of fertilising ryegrass with pig slurry (PS) and ammonium nitrosulphate (ANS-26) alone and with the 3,4-dimethylpyrazol phosphate (DMPP) nitrification inhibitor added to them. The study was conducted under Mediterranean conditions at two different sites. The treatments (control, PS, PS+DMPP, ANS-26 and ENTEC®) were established in a randomised block design with three replicates. Ammonia was sampled daily after each fertilisation using semi-static volatilisation chambers. We hypothesised that PS could replace mineral fertiliser without substantially increasing ammonia volatilisation in the studied systems.

Temperature positively correlated with ammonia emissions. On the whole, during the two years of the study, the PS treatments presented higher average cumulative ammonia volatilisation (25% of total ammonium nitrogen (TAN) applied at Site 1; 21% of TAN applied at Site 2) than the mineral ones (11% of TAN applied at Site 1; 10% of TAN applied at Site 2). At pre-sowing, ammonia volatilisation was significantly ($p < 0.05$) lower (51% at Site 1; 55% at Site 2) than after ryegrass cuts due to burying PS immediately after application. Overall, applying DMPP had no effect on ammonia volatilisation. There were no significant differences in average yield (from 13.7 to 15.8 kg ha^{-1} at Site 1; from 11.6 to 13.5 kg ha^{-1} at Site 2) between the fertilised treatments, though ENTEC® tended to increase it. Applying PS (pre-sowing fertilisation) in combination with mineral N or processed PS fractions after ryegrass cuts could be an interesting option for the recycling of this livestock by-product without increasing ammonia volatilisation while maintaining yields.

Keywords: NH_3, Pig slurry, Nitrification inhibitor, Slurry incorporation, ENTEC®

HIGHLIGHTS

- Applying pig slurry at pre-sowing did not increase ammonia volatilisation
- Burying pig slurry after application reduced ammonia emissions
- Applying mineral fertilisers when the crop is absorbing N minimises nitrate leaching
- DMPP application had no effect on ammonia volatilisation

1. Introduction

Global livestock production has rapidly grown as world population has risen and become steadily wealthier (ten Hoeve et al., 2016). In 2018, Catalonia (NE Spain) had a total herd of almost 8 million pigs (including breeding mothers, males, fattening pigs and piglets; Institut d'Estadística de Catalunya, n.d.). Within the European Union, Spain is the second highest slaughterer of pigs (19%) after Germany (23%) (EUROSTAT, n.d.). Spain is also the world's fourth largest pig producer (MAPA, n.d.). It is necessary to find sustainable ways of applying the large amount of pig slurry (PS) produced in Spain to high nitrogen demanding crops for which this is not yet common practice.

There are several benefits associated with applying farm by-products as fertilisers (compared with mineral ones) including energy saving and avoiding emissions associated with the industrial process of N fixation (Sanz-Cobena et al., 2017). Such applications allow meeting the organic matter and nutrient requirements of agricultural soils (Aguilera et al., 2013). In particular, PS is widely recognised as a valuable source of N and P for crops (Guillaumes and Villar, 2004). It is also interesting to apply PS as a fertiliser within the framework of the Nitrates Directive, as long as it is applied in appropriate agronomic doses in order to avoid NO_3^--N leaching (Teira-Esmatges and Flotats, 2003; ten Hoeve et al., 2016). Due to mechanical constraints (it is hard to apply a dose of PS below a certain threshold), PS should be administered to crops with relatively high nutrient needs (not to extensive Mediterranean crops such as olives, almonds, vineyards or fruit trees) such as wheat, barley, rice, maize and ryegrass, among others. To our knowledge, there is little information available on the effects of PS applied to ryegrass under Mediterranean conditions. As PS has low dry matter (2-

6%; AHDB, 2017) and contains nitrogen (N) which is mainly in the form of NH_4^+ (~70%) (Martínez et al., 2017a), it constitutes a source of readily available N. If not managed appropriately, applying PS may have some negative impacts on the environment (Guillaumes et al., 2006; Martínez et al., 2017b). Although ammonia is not a direct greenhouse gas (GHG), its emission into the atmosphere affects air quality: it is a precursor of low diameter particulate matter ($PM_{2.5}$) and has been linked to the loss of nutrients (Tian et al., 2015). Furthermore, ammonia volatilisation has a negative impact on biodiversity: it is associated with eutrophication, soil acidification and has direct toxic effects on aquatic animals (Guthrie et al., 2018). The loss of N in the form of ammonia therefore represents a substantial financial cost for farmers (Pan et al., 2016).

Ammonia is lost via volatilisation, which is one of the foremost pathways of N loss in agricultural systems (Pan et al., 2016). In fact, agriculture accounts for about 50% of all ammonia emissions worldwide (Sommer et al., 2004). Previous studies have reported ammonia losses of up to 56% associated with applying N fertiliser; individual rates may depend on such factors as soil moisture, temperature, soil pH, wind speed, fertiliser type, etc. (Singh et al., 2013). An average of 10-14% of the N applied with mineral fertilisers is lost via volatilisation (Zhang et al., 2018a), while livestock manure is responsible for roughly 40% of global ammonia emissions and 70% of the ammonia emissions in Europe (ten Hoeve et al., 2016).

It has been reported that temperature, wind speed, rainfall and the dry matter content of cattle and pig manure and slurry influence ammonia emissions after their surface application in the field (Huijsmans et al., 2001; Misselbrook et al., 2005). This means that the moment of fertiliser application can be critical if significant losses of N from the soil are to be avoided (Bell et al., 2016). As for ryegrass, pig slurry is usually applied at pre-sowing. Although it makes sense to apply it at other moments according to crop needs and the availability of this fertiliser resource, there is a gap in the knowledge about its effect on volatilisation after ryegrass cuts. It has been observed that the immediate incorporation of animal slurry and manure applied at pre-sowing can effectively reduce ammonia volatilisation from arable land (Sommer and Hutchings, 2001; Webb et al., 2014). This, in turn, helps to minimise its exposure to the air. This practice has become generalised in Catalonia following the introduction of Decree 153/2019 (DARP, 2019). According to Pan et al. (2016), adjusting irrigation inputs (which could be applied after PS application after ryegrass cuts) may, for example, mitigate ammonia volatilisation by between 47 and 90%.

Nitrification inhibitors (NIs) slow down the conversion of ammonium (NH_4^+) into nitrate (NO_3^-) (Zhang et al., 2018b). Kim et al. (2012) reported that NIs could increase, reduce or leave ammonia emissions unchanged. The 3,4-dimethylpyrazole phosphate (DMPP) is generally more effective and longer lasting than other NIs and requires lower application doses (0.5-1.5 kg DMPP ha^{-1}; Rowlings et al., 2016). DMPP has a similar level of mobility to NH_4^+; this limits the likelihood of leaching (Subbarao et al., 2006). However, as the use of NIs may also prolong the retention of NH_4^+ in the soil, they could cause increased ammonia volatilisation (Lam et al., 2016). NIs are widely used to control ammonia emissions resulting from organic and mineral fertilisation (Soares et al., 2012; Recio et al., 2018; Mariano et al., 2019). However, little is known about the effect of those inhibitors on PS applied to ryegrass (*Lolium multiflorum* Lam.).

Many publications have studied ammonia volatilisation from other crops: wheat (Meade et al., 2011; Nyord et al., 2012); wheat and barley (Bosch-Serra et al., 2014); barley (Yagüe et al., 2019); maize-oat rotation (Aita et al., 2019); maize (Recio et al., 2018), and grasslands (Misselbrook et al., 1996; Rodhe et al., 2006). Nevertheless, there is little available information about the effects of PS fertilisation on ammonia losses from ryegrass (Park et al., 2018).

The objective of the current study was therefore to compare the effects of applying pig slurry and mineral fertilisers, both with and without the addition of NI, on ammonia volatilisation and yield of a Westerwold ryegrass crop grown under Mediterranean irrigated and rainfed conditions.

The hypotheses formulated for this study were: i) PS may offer an alternative to mineral fertilisers when seeking to fertilise ryegrass without substantially increasing ammonia volatilisation; ii) DMPP addition could stimulate ammonia volatilisation.

2. Material and methods

2.1. Site description

2.1.1. Location, climate, soil type and crops

The study was conducted at two different sites in order to cover the thermal range of the plain cropping area in Catalonia (Meteorological Service of Catalonia, n.d.a) during two cropping years (2005 to 2007). One site was located on a commercial farm at Torreneral

(Catalonia, NE Spain). This field had an area of 0.9 ha. The other site was established at the experimental site of a research institute: IRTA Mas Badia (Catalonia, NE Spain). The location of both sites and the land use can be seen in Fig. 1.

The climate of the study area at Torreneral (hereinafter Site 1) is Mediterranean semiarid continental, with warm, dry summers and cold winters (Fig. 2a). The average annual rainfall is about 390 mm and the annual average temperature is 13.5 ºC.

The soil is very deep and is coarse loamy, mixed, calcareous and mesic; it is classified as a Typic Calcixerept (SSS, 2003). The main soil properties are presented in Table 1.

Meteorological data were obtained from the closest meteorological stations, located at El Poal and Tornabous (Meteorological Service of Catalonia, n.d.b). Average daily air temperature and relative humidity during fertilisations are shown in Fig. 3 (see Supplementary material).

At Mas Badia (hereinafter Site 2), the climate is typical of the Mediterranean coast: mild and generally warm (Fig. 2b). The average annual rainfall is about 600 mm while the annual average temperature is 15.9 ºC. The soil is very deep, coarse loamy, mixed, calcareous, thermic; it is classified as a Typic Xerofluvent (SSS, 2003). The main soil properties can be found in Table 1. Meteorological data were obtained from an automated meteorological station set up at the experimental site (Fig. 4, see Supplementary material).

The usual crops in the areas of study can be seen in Fig. 1. Prior to the present study, at Site 1 the regular crop rotation was maize-fodder (alfalfa or ryegrass)-wheat. In addition, the plots were fallow the year prior to the study. At Site 2, the usual crop rotation was rainfed winter cereals and, in some years, maize irrigated by furrows. At both sites, previous fertilisation was mainly mineral.

2.1.2. Hydrologic characterisation

Site 1 is on the modern Holocene alluvial fan of the Ondara river (distal part). There is a good hydraulic connection between the aquifer and the surface water. The unit has high hydraulic permeability (IGCC, 2017). The area belongs to the irrigation system of Canals d'Urgell.

At the end of July 2005 (year 1), the soil profile was excavated down to 165 cm at Site 1. No water table was found nor any signs of soil hydromorphism. Prior to the start of the study, passive Gee capillary lysimeters (Decagon Devices, Inc.) were installed at each plot (15 at each site) in order to monitor NO_3^- leaching. However, at Site 1 there was a flash-flood of the Ondara river at the end of 2005. The water table rose and impeded lysimeter functioning as the water entered through the lower end of the divergence control tube leading to a false leachate registry. Water also entered through the leachate evacuation port, mixing with the actual leachate. As a result, no nitrate leaching data could be analysed. A few drainage water samples from two of the elementary plots were available, suggesting that nitrate leaching was not relevant (data not shown). The use of these lysimeters in this study proved to be important for the firm that manufactured them, and they were subsequently modified after this episode.

Site 2 is on the alluvial plain of the Ter river, exactly on the "Pla de Canet", 1 to 2 km from the Ter river. It is a well drained unit with high hydraulic permeability and a very good connection between the groundwater and surface waters. There is an oscillating water table at 5 to 10 m depth and other deeper ones (IGC, ACA and ICC, 2011).

One year later than in Site 1, Gee capillary lysimeters also were installed at each plot in Site 2, where they worked as expected. Fig. 5 (see Supplementary material) shows the average drainage volume recorded per treatment from September 2006 till the end of the ammonia sampling and a few months more. The leachate nitrate concentrations were low for all the treatments (Table 2). No statistical analysis of these data was done because the data matrix was not complete.

2.2. Experimental set up

Westerwold ryegrass (*Lolium multiflorum* Lam., cv. Trinova) was sown each year in September (Table 3). The seeding rates were 20 kg ha^{-1} at Site 1, and 30 kg ha^{-1} at Site 2. The individual plots were 10 m wide x 24 m long at Site 1, and 5 m wide x 8 m long at Site 2.

At both sites, the experimental set-up had a completely randomised block design, with three replicates. There were five fertiliser treatments: i) Control (C), without any N application, ii) ammonium nitrosulphate (ANS-26); iii) ENTEC® (ANS-26+DMPP); iv) pig slurry (PS), and v) PS+DMPP (4 L ha^{-1} of a 2% DMPP solution). At both Site 1 and Site 2, the objective was to apply about 250 kg N ha^{-1} (except during the year 1 at Site

1). The exact doses of PS and mineral fertiliser applied before sowing (pre-sowing) and after the first and second cuts are shown in Table 4.

Due to the high initial soil P and K contents at Site 1, no P or K fertilisation was applied to the mineral fertiliser treatments throughout the study. In contrast, mineral P and K were applied at Site 2 in order to obtain equal amounts of both macronutrients on both the plots to which PS was applied and those to which it was not.

The PS was analysed (Table 5) before application and mechanically incorporated into the soil on the same day of its application. At Site 1, the amount of PS applied was measured on site for each plot and application time; this was done using trays laid on the soil. At Site 2, pig slurry was applied manually and the amount administered was carefully prepared beforehand.

At Site 1, as ryegrass cultivation is not possible without irrigation, about 150 mm irrigation was applied immediately after sowing. A second dose of irrigation (60 mm) was then applied a couple of days later, using a mobile rain gun sprinkler. The crop was then irrigated according to the farmer's criterion. The irrigation water was of good quality, with low NO_3^- and low electrical conductivity ($EC < 0.5$ dS m^{-1}). Nitrogen deposition via precipitation was negligible (data not shown).

2.3. Ammonia sampling and quantification

Before fertilisation, ammonia was sampled to know the background volatilisation. Following fertilisation, ammonia from the fertilised treatments was quantified following the methodology described in Bosch-Serra et al. (2014), with some minor modifications. In this study, one sponge (trap) per chamber was used; the chambers mainly consisted of 23.4 cm diameter, 15 cm high, low-density polyethylene terephthalate (LD PET) cylinders; at Site 2, some of them were 27 cm in diameter and 12.5 cm in height.

Unlike the methodology reported by Bosch-Serra et al. (2014), in our study, once in the laboratory, each sponge was soaked in distilled water (four times with 100 mL) and then made up to 500 mL in a volumetric flask. Within this total volume, a 100 mL aliquot was then mixed with 10 mL of NaOH (40% w/v); this allowed the release of ammonia in the form of gas. This process was repeated twice per sponge. The ammonia quantification was carried out using ammonia selective electrodes (Crison, micropH 2002 at Site 1, and Mettler-Toledo^{TM} S230 at Site 2).

During rainfall events, the chambers were covered with plastic bags. Given these modifications, it is not possible to speak about open chambers here, but rather of closed chambers.

On the first day of sampling (after fertilisation application), the traps were changed twice. During the following 10 to 13 days of sampling, the traps were changed once every 24 h. Thereafter, given the lower ammonia volatilisation rate, the traps were changed two to three times per week. All the results were calculated and reported as daily (24 h) volatilisation.

Cumulative volatilisation is expressed as the mass of NH_3-N per hectare, and as the percentage of the total N applied and of the total ammonium nitrogen (TAN) applied, lost over the sampling periods.

2.4. Soil sampling and analysis

Soil samples were taken at four different moments: before sowing and after each cut. They were taken using an Edelman auger. At Site 1, some extra samples were taken before and after the 1st cut. These samples were composite: at both sites, they were obtained from three different subsamples and taken from the same plot at a depth of 0-30 cm (the results down to 120 cm are not shown). The samples were then placed in a sealed and labelled plastic bag and kept in a cold store at 4 ºC until analysis. Soil moisture (%) was determined by drying at 105 ºC to constant weight. Soil NO_3^--N was quantified from a soil water extract (1:2.5) by spectrophotometry performed with a continuous flow autoanalyser (λ= 520 nm). Quantification was based on the modified Griess-Ilosvay method (method of Barnes and Folkard, 1951, and Bremner, 1965, in Page et al., 1982). Ammonium nitrogen was extracted with KCl and quantified by visible UV spectrophotometry (λ= 660 nm), based on the Berthelot reaction (Berthelot 1859 in Page et al., 1982).

The nitric nitrogen in the drainage water collected from the lysimeters was analysed. Two readings were taken of each water sample. The determination of nitric nitrogen was made by means of a nitrate test with Merckoquant® indicator strips, providing a semi-quantitative determination of nitrate ions. The determinations were performed using a Nitracheck® reflectometer. Readings of a known nitrate concentration pattern

(100 ppm) were taken with the aim of establishing a correction factor for the temperature and the batch of indicator strips.

The correction factor for the readings was calculated using the following formula:

$$f = \frac{100}{\overline{\mathrm{X}}},$$

where, f= correction factor and $\overline{\mathrm{X}}$ = average of the pattern measurements.

To correct the readings, it is necessary to apply the correction factor to the group of determined samples. We applied the following formula:

$\mathrm{Cr} = f \times \mathrm{R}$

where, Cr= corrected reading of nitric nitrogen (ppm); f = correction factor; and R= uncorrected reading

To change the nitrogen value in ppm obtained with the Nitracheck® reflectometer to kg of nitric nitrogen leached per hectare, the following formula was used:

LN= Cr x Dv x 0.00226

where, LN= leached or washed nitrogen (kg NO_3^--N ha^{-1}); Cr= corrected reading of nitric nitrogen (ppm); Dv= drainage volume collected by the lysimeters (m^2 L^{-1}). 0.00226 is the factor to obtain the result in kg of nitrogen per hectare and was obtained from the following calculation:

$$\frac{\mathrm{Cr\ mg\ NO_3^-}}{\mathrm{L}} \mathrm{x} \frac{\mathrm{D_v\ L}}{\mathrm{m^2}} \mathrm{x} \frac{\mathrm{10^4\ m^2}}{\mathrm{1\ ha}} \mathrm{x} \frac{\mathrm{1\ g}}{\mathrm{10^3\ mg}} \mathrm{x} \frac{\mathrm{1\ mol\ NO_3^-}}{\mathrm{62\ g}} \mathrm{x} \frac{\mathrm{14\ g\ N}}{\mathrm{1\ mol\ NO_3^-}} \mathrm{x} \frac{\mathrm{1\ kg\ N}}{\mathrm{10^3\ g}} = \mathrm{Cr\ x\ Vd\ 0.00266}\ \frac{\mathrm{kg\ N}}{\mathrm{ha}}$$

The sampled drainage water had the nitrate concentrations reported in Table 2. Only the averages and the cumulative leachate were calculated.

2.5. Harvesting and plant analysis

Each year three cuts were made, with a total of six cuts at each site. The controlled ryegrass was hand-harvested at Site 1 and cut with a motor harvester (TRIUNFO TK 600 D - Motor KUBOTA) at Site 2, on the dates shown in Table 3.

At Site 1, the sampled area was 0.50 m^2 in the first year and 1.00 m^2 in the second year. At Site 2, the central part of the plot (9.6 m^2) was sampled in both years. The ryegrass dry matter produced in each cut was determined.

2.6. Calculations and statistical analysis

Data normality was assessed using the Shapiro-Wilk test. Data were studied by analysis of variance, and separation of means was carried out using the Student's *t*-test (effect of PS vs. mineral fertiliser and effect of applying or not DMPP) and Tukey's multiple range test (cumulative ammonia volatilisation; percentage of total N and of total ammonium nitrogen (TAN) applied; yield and fertilisation application moment) at a 5% level of significance. In order to compare between years and fertilisation regimes, the statistical analysis of volatilisation was only carried out until the 22nd day of sampling.
The non-parametric Spearman's rank coefficient test and an ANOVA (at a $p < 0.05$ level of significance) were carried out to analyse the correlation and the effect between ammonia volatilisation and the driving factors (daily air temperature (average and

maximum), daily rainfall and daily rainfall plus irrigation (Site 1), wind speed, soil organic matter content, and slurry dry matter).

The statistical analyses were performed using the JMP statistical package (JMP® version 13; SAS Institute Inc., Cary, NC, 2015).

3. Results and discussion

Ammonia volatilisation sharply increased shortly after fertilisation (Figs. 6 and 7), especially for the treatments with pig slurry (PS). At Site 1, cumulative volatilisation ranged between 5 and 49 kg NH_3-N ha^{-1} in year 1, and between 3 and 56 kg NH_3-N ha^{-1} in year 2. At Site 2, the cumulative volatilisation ranged between 4 and 39 kg NH_3-N ha^{-1} in year 1, and between 2 and 31 kg NH_3-N ha^{-1} in year 2. Rochette et al. (2008) reported similar cumulative ammonia losses (6-53 kg NH_3-N ha^{-1}) to those reported in this study when applying PS to a crop of timothy (*Phleum pratense* L.), although the PS dose applied was lower than in the present study.

At Site 1, the average ammonia volatilisation from both pig slurry treatments and all applications was 13% of total N applied in year 1, and 27% in year 2, while all the mineral treatments averaged a volatilisation rate of 8% of N applied in year 1, and 10% in year 2. At Site 2, the average ammonia volatilisation from both pig slurry treatments and all applications of the two studied years together was 15% of total N applied, while all the mineral treatments averaged a volatilisation rate of 7% for both years together. A higher percentage (close to 30% of total N applied) than those reported here was found by Nicholson et al. (2017) for a livestock (cattle and pig) slurry, and an even higher one

(42% of total N applied) was reported by Yagüe and Bosch-Serra (2013) for a treatment with PS (without incorporation) applied on a bare soil.

Recio et al. (2018) found similar cumulative volatilisation to those of the present study for PS (14.56% of the total N applied) and PS+DMPP (9.96% of the total N applied) applied at pre-sowing. Bourdin et al. (2014) reported higher ammonia volatilisation (30.3-70.8%) from total N applied after PS application in comparison with the present study. Likewise, Misselbrook et al. (2000) reported that 1.5% of total N applied volatilised after applying ammonium nitrate, compared to 59% after applying cattle slurry.

The average ammonia volatilisation (both study years) from TAN applied at pre-sowing and after cuts was 11% and 10% from the mineral treatments vs. 25% and 21% from the PS treatments at Site 1 and Site 2, respectively.

3.1. Effect of the type of fertiliser on volatilisation; incorporation and moment of fertilisation

The type of fertiliser used had a significant effect on volatilisation (Table 6). In half of the sampled periods, the percentage of ammonia volatilisation from total N and TAN applied was significantly higher in the pig slurry (PS) treatments than the mineral ones, in both years of the study and sites (Figs. 6 and 7). This could be related with the low dry matter (2-6%) and total organic matter content (< 40%) of PS (AHDB, 2017; Danés and Boixadera, 2001), which facilitate rapid volatilisation following PS application. As PS can be assimilated to an ammonium liquid fertiliser, it can be ready for ammonia volatilisation once in contact with air. Bosch-Serra et al. (2014) also observed a higher cumulative volatilisation from PS than from mineral fertiliser (ammonium nitrate) applied to rainfed winter cereals. In a ryegrass (*Lolium perenne*) crop, Bourdin et al. (2014) also observed significantly higher volatilisation from cattle slurry (with 2.06-7.42% dry matter) than from a mineral fertiliser (calcium ammonium nitrate).

The moment of application had a significant effect on ammonia volatilisation (Table 7). The percentage of cumulative ammonia volatilised with regard to the quantity of TAN applied was significantly lower at pre-sowing than at the 1st cut fertilisation. At pre-sowing in year 1 at Site 1, and also in year 1 at Site 2, there was no difference between PS treatments and mineral treatments in terms of the percentage of applied TAN that volatilised. The lower volatilisation from the present study was possibly due to burying PS as soon as possible after its application. This was in agreement with the ammonia abating measures recommended by Bittman et al. (2014). Likewise, probably, higher volatilisation in the 1st cut can be associated with exposure to the atmosphere of the PS in this fertilisation, when burying was not possible without damaging the crop. Relating with the moment of application, Bell et al. (2016) observed different ammonia losses in spring (15.71% of the total N applied) than in autumn (11.44%) from treatments of cattle and broiler manure.

At both sites of the present study, ammonia volatilisation significantly differed according to the moment of fertilisation; fertilisation after ryegrass cuts was associated with significantly higher cumulative ammonia losses (almost the double in some occasions) than at pre-sowing (Figs. 6 and 7).

For a perennial forage grass, Rochette et al. (2008) obtained an average volatilisation of 32% (of TAN applied) from PS treatments applied by splash-plate; this was higher than the results of the present study. Sanz et al. (2010) reported a case of 20% of ammonia volatilisation (of TAN applied) for PS applied to the soil surface of a bare soil.

Burying PS after applying pre-sowing fertilisation helped to reduce ammonia volatilisation until 2 and 3 days after pre-sowing fertilisation (Figs. 6a and 7c). According to Webb et al. (2010), the incorporation of manures into the soil immediately after application also influences the extent of ammonia emissions. In contrast, after the 1st and 2nd cuts, the fertiliser could not be buried and the recorded volatilisation was higher than at pre-sowing fertilisation. At Site 1, immediately burying the fertiliser after the pre-sowing application even counteracted the effects of higher ETo during that sampling period (autumn) than after the 1st cut (winter).

At 1st cut, the PS+DMPP treatment showed significantly higher cumulative ammonia volatilisation (35% of TAN applied) than the rest of the treatments (Fig. 7). This was probably due to DMPP application, which inhibits NH_4^+-N oxidation to NO_3^--N favouring ammonia volatilisation. Thus, it would be an interesting management strategy to replace ANS-26 with PS (without DMPP) especially at pre-sowing (PS can be incorporated).

It should be noted that it rained shortly after the pre-sowing fertilisation (on day five at Site 1 and on day three at Site 2); this together with burying PS diminished volatilisation (Figs. 6a and 7c). It is known that rainfall can reduce ammonia volatilisation. Sanz-Cobena et al. (2019) reported greater reductions in ammonia volatilisation after heavy rainfall in the Mediterranean area with a barley crop. In such cases, a useful management strategy to reduce ammonia losses would involve irrigating after fertilisation (Viero et al., 2015). Applying a small amount of water (e.g. 10 mm) is enough to mitigate these losses (Misselbrook, 2019). This management strategy was also successfully used at Site 1 after pre-sowing fertilisation (Fig. 6a).

After the 1st cut, in both years, the PS treatments (PS and PS+DMPP) registered higher volatilisation: on average 50% (Site 1) and 55% (Site 2), respectively, within the first 48 h after fertilising. These findings are in line with those reported by Soares et al. (2012). Park et al. (2018) observed that >50% of the total ammonia emissions occurred within the first 14 days after applying PS in the case of a perennial ryegrass sward. In contrast, Sanz et al. (2010) reported that 63-75% of total NH_3-N volatilisation occurred in the first 24 h after PS application to a bare soil. Yagüe and Bosch-Serra (2013) found that 50% of the total ammonia losses happened within 17 h (spring) and 8 h (summer) following PS application on a bare soil under Mediterranean conditions.

3.2. Effect of DMPP

Applying DMPP did not have a consistent effect on ammonia volatilisation either at Site 1 or at Site 2 in either of the study years (Table 6). For the two study years, volatilisation as a percentage of TAN applied from the treatments with DMPP ranged from 7% to 50% at Site 1 and from 6% to 35% at Site 2. Lower ammonia losses (0.4 to 0.6% of TAN applied) associated with another nitrification inhibitor (DCD) were reported by Mkhabela et al. (2006) from pig manure fertilisation in a laboratory study.

In year 1, after the 2nd cut at Site 1, DMPP (PS+DMPP treatment) significantly increased volatilisation (as a percentage of TAN applied), as well as after the 1st cut at Site 2. In year 2 at Site 2, the ENTEC® treatment significantly volatilised less (as a percentage of total N and TAN applied) than the PS treatments. Zhou et al. (2016) and Pan et al. (2016) reported that applying nitrification inhibitors often resulted in increased ammonia volatilisation. Menéndez et al. (2006) reported the same finding in perennial ryegrass fertilised with cattle slurry. They did not, however, observe any significant increase in ammonia associated with applying DMPP. The application of DMPP to PS probably delayed the transformation of NH_4^+ to NO_3^-, which would have facilitated ammonia volatilisation.

No satisfactory explanation could be found for the inconsistent effect of DMPP application on volatilisation through the study.

The amount of leached N should be taken into account in agricultural practice due to the vulnerability of agricultural land to nitrogen and water losses (Aschonitis et al., 2012). In this study, it was seen that nitrate leaching from mineral fertilisers could be minimised by applying the fertiliser when the crop starts growing and absorbing nitrogen, rather than at pre-sowing, provided the soil nitrogen content is sufficient for germination and initial growth.

3.3. Other factors affecting ammonia volatilisation

Sommer et al. (2003) reported that environmental factors such as temperature, moisture and wind speed, and also soil pH, have an influence on ammonia volatilisation. In the present study, a weak but mainly positive correlation was observed between air temperature and ammonia volatilisation at both Site 1 and Site 2 (Table 8, see Supplementary material). Moreover, an effect of the study location and an interaction between average daily temperature and rainfall plus irrigation were also observed at pre-sowing (year 1) at Site 1 (Table 9, see Supplementary material). This could be due to a higher soil water content at Site 1 due to irrigation. Wind speed did not significantly affect ammonia volatilisation in none year nor site.

Explanations for the differences in volatilisation between the sites (at Site 1, the pooled average of the cumulative volatilisation was significantly higher (18.16a) than at Site 2 (12.78b)) could be related to the higher dry matter (DM) content of the slurry of Site 1 than that of Site 2 (Table 5). The DM content of the PS tended to slow down infiltration, which would have permitted a longer PS exposure to the air. A low DM and a reduction in the viscosity of liquid manure are known to reduce ammonia emissions because they facilitate soil infiltration (Sommer et al., 2006). At Site 2, the DM content in the PS+DMPP treatment (5.9%) applied after 1st cut (year 1) was higher than that applied in year 2 after the 1st cut (2.6%), and lower level of ammonia volatilisation was observed from the slurry with lower DM. Häni et al. (2016) reported that applying slurry with a lower DM content could reduce ammonia losses. Bosch-Serra et al. (2014) observed reduced ammonia emissions from both low and high slurry DM. In the former case, this was because infiltration was facilitated; in the latter, it was due to the favouring of crust formation.

It is known that slurries with a low DM content are generally associated with lower ammonia emissions as they rapidly infiltrate into the soil (Sommer and Hutchings, 2001). According to Hafner et al. (2018), ammonia volatilisation was positively correlated with DM of PS for broadcast applications. In the present study, however, no correlation with DM was found (data not shown).

The difference between cumulative volatilisation per sites could also be associated with NH_4^+ and NO_3^- contents, which were higher at Site 1 than at Site 2 (Figs. 8 and 9, see Supplementary material). On the other hand, it could also be related to soil properties, though only a slightly positive correlation (0.17, $p< 0.05$) was found between cumulative volatilisation and soil organic matter content.

3.4. Yield

At Site 1, average yield ranged from 9133 to 15,759 kg DM ha^{-1} $crop^{-1}$; and at Site 2 from 7824 to 13,483 kg DM ha^{-1} $crop^{-1}$ (Table 10). Walsh et al. (2018) reported similar levels of production in a mixed pasture ley (ryegrass and clover) (~5000-12,000 kg DM ha^{-1} yr^{-1}). The average yield (two years) significantly differed between the control treatment and the fertilised ones, but no significant differences were found among the latter. The ENTEC® treatment tended to be the highest producer, though the yields were not significantly different from those of the rest of the fertilised treatments (Table 10). Only in year 2, at Site 2, was the yield from the ENTEC® treatment significantly higher than that from the PS+DMPP treatment. It seems that, at Site 1, the DMPP treatments (PS+DMPP and ENTEC®) tended to produce more than their counterparts without DMPP, although there were no significant differences between yields. This trend was not as clear at Site 2, where the PS treatment produced higher yields than the PS+DMPP treatment. Dougherty et al. (2016) also found no significant differences in yield between treatments for a mixed ryegrass and kikuyu system both with and without the application of DMPP.

4. Conclusions

The ammonia losses (as a percentage of total N and TAN applied) in the tested Mediterranean conditions were similar to those cited in the literature for other climates.

The hypothesis that PS could offer an alternative to mineral fertilisers for fertilising ryegrass without substantially increasing ammonia volatilisation can be partially accepted. It would be feasible to apply PS at pre-sowing combined with mineral N or processed PS fractions after ryegrass cuts. This could be a valid option to reuse this livestock by-product and replace mineral fertiliser.

DMPP application did not increase ammonia volatilisation at either of the studied sites. The effect of DMPP on ammonia volatilisation from fertilised ryegrass needs further study. ENTEC® tended to increase yield.

Mineral fertilisers should be supplied when there are N extractions (not at pre-sowing) to avoid nitrate leaching, provided there is enough N in the soil at germination and early growth.

It would be interesting to study the effect of treated PS or some fractions of it applied after the ryegrass cuts on ammonia volatilisation, and to study nitrate leaching at the irrigated site (where the lysimeters had not worked properly) in the future. If possible, it would also be interesting to repeat the experiment in a growth chamber (less dependence on meteorological conditions).

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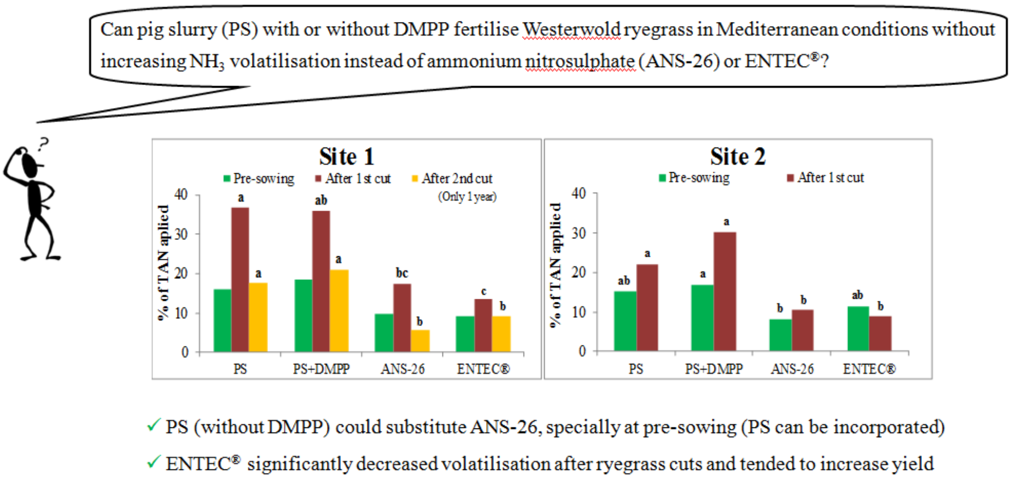
Graphical abstract



Can pig slurry (PS) with or without DMPP fertilise Westerwold ryegrass in Mediterranean conditions without increasing NH_3 volatilisation instead of ammonium nitrosulphate (ANS-26) or ENTEC®?

- ✓ PS (without DMPP) could substitute ANS-26, specially at pre-sowing (PS can be incorporated)
- ✓ ENTEC® significantly decreased volatilisation after ryegrass cuts and tended to increase yield

Tables

Table 1. Mean values of chemical soil properties at the beginning of the study, granulometric analysis and textural class in the upper soil layer at Site 1 and Site 2.

	Site 1*	Site 2
	Depth 0-30 cm	
Chemical properties		
$CaCO_3$ equivalent (%)	11	14 ± 2
Organic matter (Walkley-Black; %)	2.44	1.80 ± 0.11
pH (1:2.5 water extract)	8.1	8.2 ± 0.1
EC (1:5 water extract; dS m^{-1})	0.6	0.2 ± 0.0
NO_3^--N (colorimetry; mg kg^{-1})	19	13 ± 2
P (Olsen; ppm)	23	25 ± 5
K (Ammonium acetate extract; ppm)	156	102 ± 24
Na (Ammonium acetate extract; ppm)	103	-
Mg (Ammonium acetate extract; ppm)	310	-
Ca (Ammonium acetate extract; ppm)	6424	-
Granulometric analysis		
Clay (%)	14.8	11.1 ± 0.5
Silt (%)	32.1	40.4 ± 1.7
Sand (%)	53.1	48.5 ± 2.1
USDA textural class	Sandy loam	Loam

*At Site 1, the soil analysis was done with one soil sample composite of nine subsamples, thus there is no standard deviation.

Table 2. Nitrogen (N) leached per ryegrass cut and total N leached during the three cuts of the cropping year 2006-07 quantified in the drainage water sampled from the lysimeters.

Treatment	N leached (kg N ha^{-1})			
	1st cut	2nd cut	3rd cut	Cumulative
C	1.01	0.00	0.01	1.02
PS	0.02	0.00	0.00	0.02
PS+DMPP	0.04	0.01	0.06	0.11
ANS-26	12.08	0.00	0.00	12.08
ENTEC®	34.75	0.00	0.00	34.75

Table 3. Timing of sowing, ammonia sampling period after each fertilisation, and harvest per study site.

Study site	(dd/mm/yy)						
	Sowing	Ammonia sampling starting on the day of fertilisation			Cuts		
		After pre-sowing	After 1st cut	After 2nd cut	1st	2nd	3rd
Site 1	27/09/2005	26/09 to 17/10/2005 (22 days)	12/03 to 25/04/2006 (45 days)	02/05 to 02/06/2006 (32 days)	28/02/2006	26/04/2006	01/06/2006
	03/10/2006	29/09 to 29/11/2006 (62 days)	23/12 to 30/01/2007 (39 days)	-	18/12/2006	27/04/2007	15/06/2007
Site 2	30/09/2005	29/09 to 20/10/2005 (22 days)	13/01 to 10/02/2006 (29 days)	-	04/01/2006	27/03/2006	11/05/2006
	07/09/2006	06/09 to 11/10/2006 (36 days)	05/12 to 02/01/2007 (29 days)	-	01/12/2006	30/03/2007	14/05/2007

Table 4. Doses of pig slurry (PS) and mineral fertiliser supplied in each application at Site 1 and at Site 2.

Treatment	Site 1										Site 2							
	Year 1						Year 2				Year 1				Year 2			
	At pre-sowing		After 1st cut		After 2nd cut		At pre-sowing		After 1st cut		At pre-sowing		After 1st cut		At pre-sowing		After 1st cut	
	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}	kg NH_4^+-N ha^{-1}	kg total N ha^{-1}
PS	151.0	219.4	223.8	264.6	209.1	285.6	82.6	99.1	97.9	147.2	86.8	98.0	110.2	173.9	69.0	123.2	120.6	155.7
PS+DMPP	194.5	246.6	127.9	151.6	160.8	232.0	73.4	97.4	112.2	170.8	86.8	98.0	110.2	173.9	69.0	123.2	120.6	155.7
ANS-26	93.8	125.0	93.8	125.0	93.8	125.0	93.8	125.0	93.8	125.0	75.0	100.0	112.5	150.0	75.0	100.0	112.5	150.0
ENTEC®	88.9	125.0	88.9	125.0	88.9	125.0	88.9	125.0	88.9	125.0	71.1	100.0	106.7	150.0	71.1	100.0	106.7	150.0

Table 5. Analysis of each pig slurry (PS) applied at Site 1 and Site 2 in both years.

	Site 1										Site 2							
	Year 1						Year 2				Year 1				Year 2			
	At pre-sowing		After 1st cut		After 2nd cut		At pre-sowing		After 1st cut		At pre-sowing		After 1st cut		At pre-sowing		After 1st cut	
	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP	PS	PS+DMPP
Dose (m^3 ha^{-1})	42.9	57.1	40.6	46.3	49.7	38.2	25.0	25.0	28.8	32.9	50.0	50.0	40.0	40.0	32.5	32.5	50.0	50.0
pH	9.0	8.9	-	-	8.0	7.9	8.6	8.5	8.6	8.5	8.5	8.5	9.2	9.3	8.7	8.7	8.4	8.4
EC (25ºC, dS m^{-1})	20.39	19.57	-	-	6.13	6.76	5.30	5.52	5.29	5.82	16.09	16.10	14.44	12.33	2.92	2.92	2.8	2.8
Dry matter (%, f.m.)	5.7	6.2	6.2	2.4	4.5	5.3	2.4	3.4	6.4	6.2	0.8	0.8	6.2	5.9	7.5	7.5	2.6	2.6
Organic matter (%, d.m.)	74.0	73.6	-	-	72.2	71.7	57.2	61.3	67.6	66.3	42.4	43.6	58.5	60.0	55.9	55.9	57.6	57.6
N Kjeldahl (%, d.m.)	2.80	1.47	10.51	13.64	3.42	3.52	2.75	2.82	2.68	2.87	2.66	2.95	2.51	2.57	2.47	2.47	2.75	2.75
NH_4^+-N (%, d.m.)	6.18	5.49	8.89	11.51	9.35	7.94	13.76	8.64	5.32	5.50	21.01	22.41	4.37	4.54	2.6	2.6	9.46	9.46
C/N	13.2	25.0	-	-	10.6	10.2	10.4	10.9	12.6	11.6	8.0	7.4	11.7	11.7	11.3	11.3	10.5	10.5
P (%, d.m.)	1.73	1.73	1.81	1.61	2.21	2.53	0.68	1.28	3.18	3.23	1.15	1.25	2.48	2.42	3.13	3.13	2.03	2.03
K (%, d.m.)	4.72	4.40	6.73	9.29	5.75	5.25	13.41	9.93	5.12	5.57	19.60	18.89	3.46	3.77	2.32	2.32	7.98	7.98
Cu (ppm, d.m.)	417	411	-	-	608	742	392	549	1804	1876	168	181	507	527	332	332	308	308
Zn (ppm, d.m.)	2872	2807	-	-	3411	4206	1347	2013	1484	1464	571	564	3031	3133	1798	1798	1486	1486
Ca (%, d.m.)	2.25	2.31	-	-	2.91	2.72	1.61	2.71	4.74	4.26	-	-	-	-	-	-	-	-
Mg (%, d.m.)	0.97	1.01	-	-	1.10	1.33	0.42	0.99	1.87	1.90	-	-	-	-	-	-	-	-

EC: electrical conductivity; f.m.: fresh matter basis; d.m.: dry matter basis.

Table 6. Cumulative ammonia volatilisation expressed as a percentage of the total ammonium nitrogen (TAN) applied, per factor studied (N type and DMPP addition), and the effect of both factors and its interaction in years 1 and 2, at Site 1 and Site 2.

Factors studied	% TAN applied								
	Site 1					Site 2			
	Year 1			Year 2		Year 1		Year 2	
	Pre-sowing	After 1st cut	After 2nd cut	Pre-sowing	After 1st cut	Pre-sowing	After 1st cut	Pre-sowing	After 1st cut
Pig slurry	9.11 ± 2.16	21.99 ± 4.04	19.39 ± 2.06a	25.46 ± 5.71a	50.73 ± 21.04a	17.51 ± 4.21	29.88 ± 11.65a	14.46 ± 2.26a	25.25 ± 4.26a
Mineral N	7.09 ± 2.16	16.55 ± 5.42	7.48 ± 3.11b	12.01 ± 7.65b	14.36 ± 6.20b	14.05 ± 5.09	7.45 ± 1.63b	5.29 ± 0.36b	12.04 ± 7.21b
+DMPP	7.53 ± 2.12	19.80 ± 2.88	15.17 ± 6.90a	20.25 ± 10.77	29.63 ± 22.87	18.50 ± 4.80a	21.80 ± 16.10a	9.71 ± 4.69	17.04 ± 9.22
-DMPP	8.67 ± 2.55	18.74 ± 7.38	11.70 ± 6.65b	17.21 ± 8.84	34.46 ± 27.05	13.06 ± 3.16b	12.53 ± 7.37b	10.04 ± 5.79	20.25 ± 9.14
N type	ns	ns	*	*	*	ns	*	*	*
+DMPP/-DMPP	ns	ns	*	ns	ns	*	*	ns	ns
N type x +DMPP/-DMPP	ns	ns	ns	ns	ns	ns	ns	ns	ns

Different letters per column indicate significant differences between treatments ($p < 0.05$, Student's t-test). The statistical analysis was only carried out until the 22nd day of sampling in order to make comparisons between years and fertilisations.

***:** Significant ($p < 0.05$); ns: not significant.

Table 7. Cumulative ammonia volatilisation, and expressed as a percentage of total N and of total ammonium nitrogen (TAN) applied per fertilisation and the effects of different factors at Site 1 and Site 2 over both years of the study (except for the fertilisation after the 2nd cut, for which only one year was available).

Fertilisation	Site 1			Site 2		
	kg NH_3-N ha^{-1}	% of total N	% of TAN applied	kg NH_3-N ha^{-1}	% of total N	% of TAN applied
Pre-sowing	11.61 ± 6.96b	10.52 ± 6.84b	13.88 ± 8.69b	8.35 ± 4.99b	9.13 ± 4.94b	12.54 ± 5.68b
After 1st cut	27.15 ± 21.25a	17.82 ± 11.82a	27.85 ± 18.36a	18.68 ± 13.13a	13.79 ± 7.47a	19.42 ± 10.86a
After 2nd cut	20.28 ± 14.98a	14.78 ± 4.66ab	19.72 ± 6.71ab	-	-	-
Factors studied						
N type	*	*	*	*	*	*
+DMPP/-DMPP	ns	ns	ns	ns	ns	ns
Year	ns	*	*	ns	ns	ns
Fertilisation period	*	*	*	*	*	*
Site	*					

Different letters per column indicate significant differences between treatments ($p< 0.05$, Tukey's test and Student's t-test). The statistical analysis was only carried out until the 22nd day of sampling in order to make comparisons between years and fertilisations.

***:** Significant ($p< 0.05$); ns: not significant.

Table 10. Yield (expressed as dry matter) per treatment and year at Site 1 and Site 2.

Treatment	Yield (kg ha^{-1})					
	Site 1			Site 2		
	Year 1	Year 2	Average of both years	Year 1	Year 2	Average of both years
C	10,115 ± 1942b	8151 ± 1642	9133 ± 1777b	8551 ± 1623b	7098 ± 688c	7824 ± 1235b
PS	15,087 ± 1167a	13,356 ± 2726	14,222 ± 2056a	12,424 ± 1645a	13,043 ± 1167ab	12,734 ± 1387a
PS+DMPP	16,868 ± 1034a	13,323 ± 1645	15,096 ± 1465a	11,935 ± 1555ab	11,306 ± 1149b	11,620 ± 1331a
ANS-26	15,696 ± 1403a	11,790 ± 1720	13,743 ± 1664a	12,049 ± 1849ab	14,076 ± 1202ab	13,062 ± 1552a
ENTEC®	17,580 ± 1243a	13,939 ± 1778	15,759 ± 1614a	12,164 ± 1959ab	14,801 ± 1260a	13,483 ± 1661a

Different letters within columns indicate significant differences between treatments ($p< 0.05$, Tukey's test).

Figures

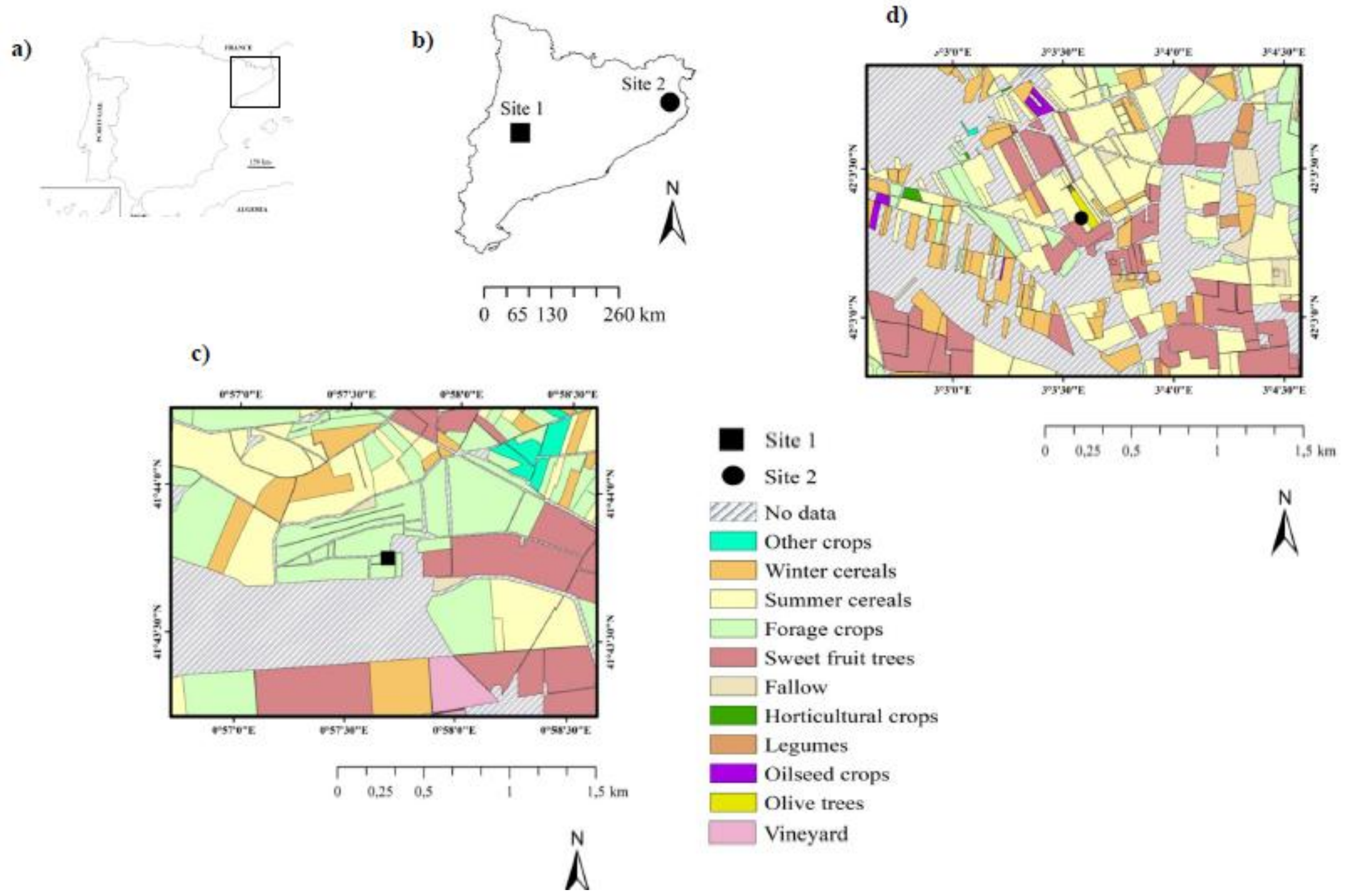


Figure 1. Location maps (a and b), and land uses (DARP, n.d.) at Site 1 (c) and at Site 2 (d). Cartographic projection UTM 31 N.

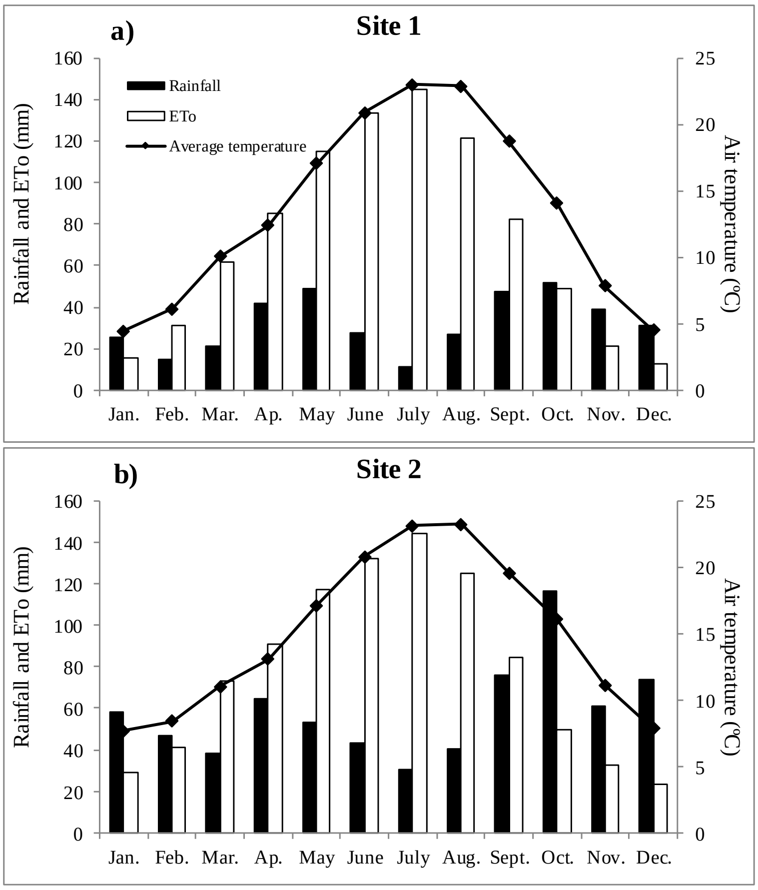
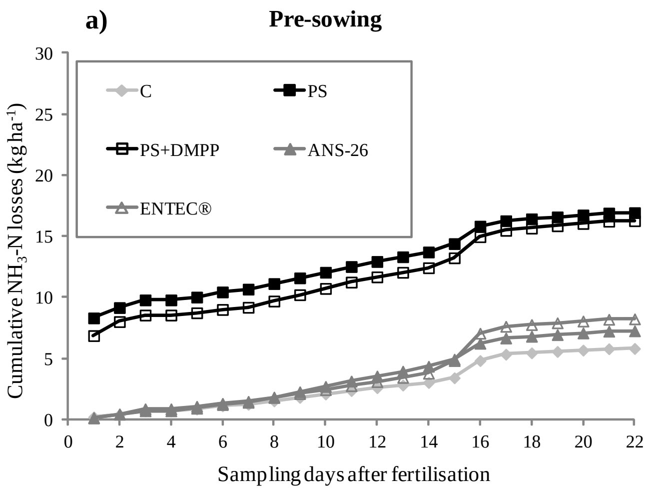
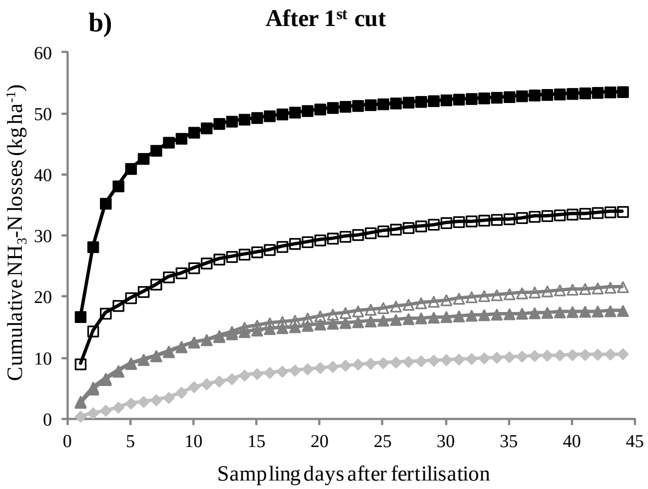


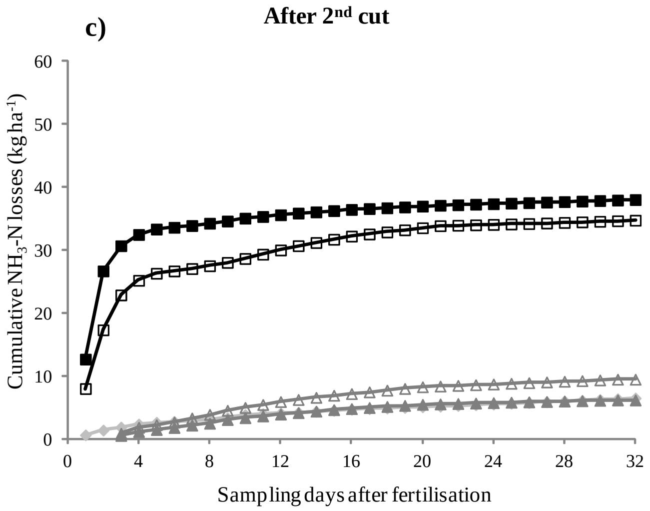
Figure 2. Average monthly rainfall, air temperature and reference crop evapotranspiration (ETo, FAO Penman-Monteith equation) (a) from the El Poal and Tornabous meteorological stations (period 2000-2010), and (b) from an automatic meteorological station located at the experimental Site 2 (period 1990-2007).



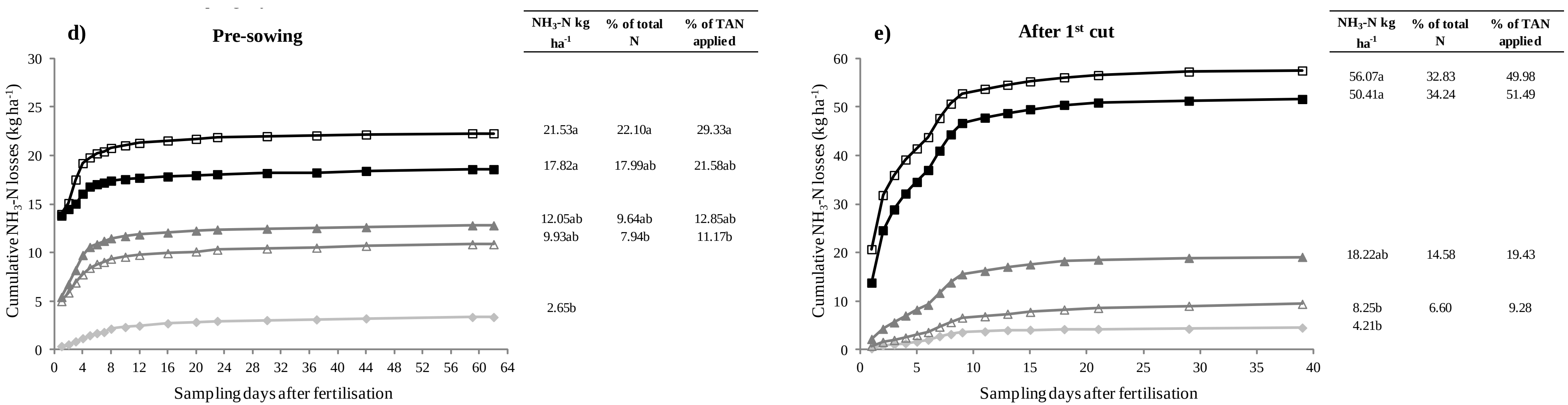
NH_3-N kg ha^{-1}	% of total N	% of TAN applied
15.97a	7.28	10.58
14.86a	6.02	7.64
6.60b	5.28	7.42
6.34b	5.07	6.76
4.56b		



NH_3-N kg ha^{-1}	% of total N	% of TAN applied
49.16a	18.58	21.97
28.16b	18.57	22.02
15.64bc	12.51	17.59
14.54bc	11.64	15.51
7.42c		



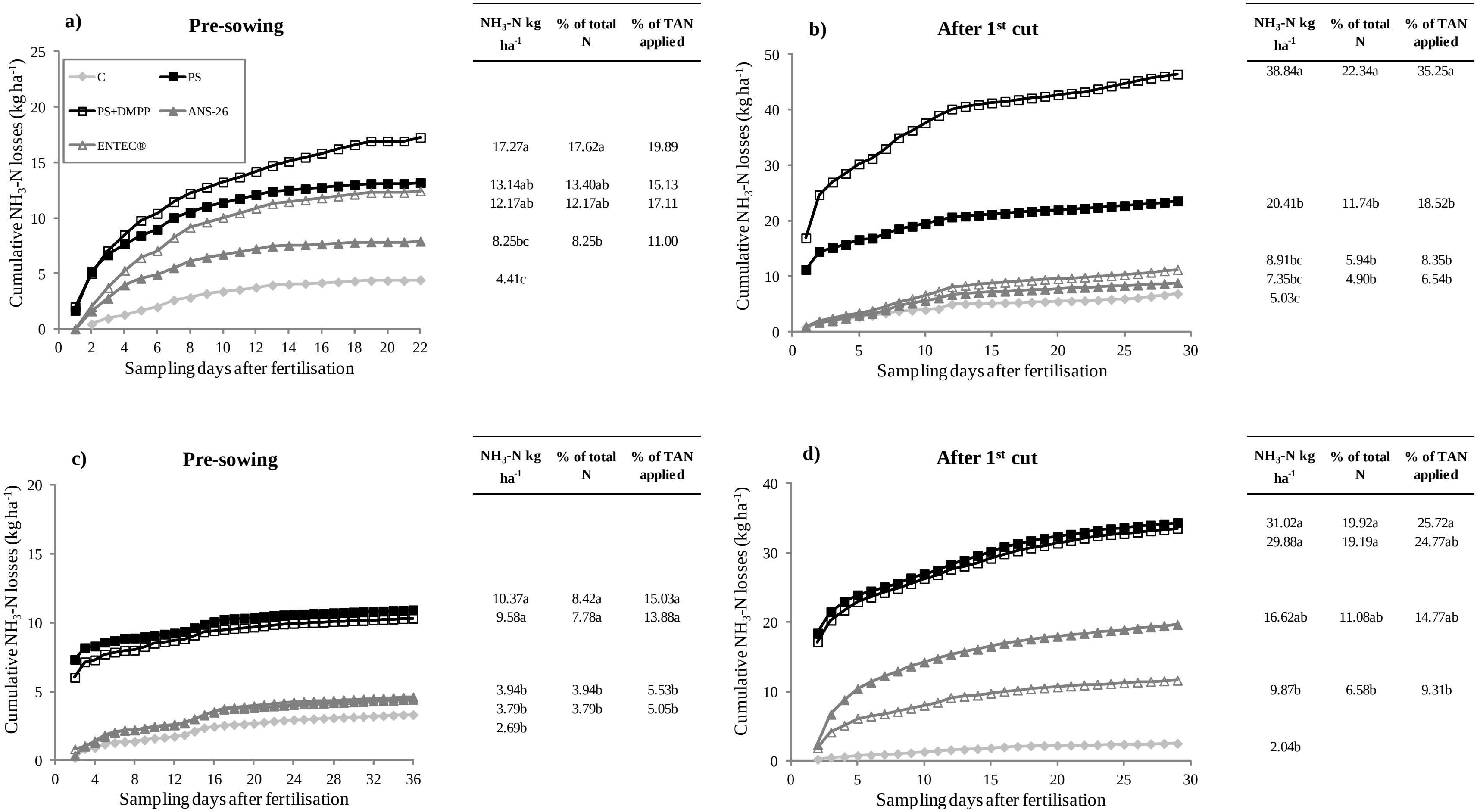
NH_3-N kg ha^{-1}	% of total N	% of TAN applied
37.08a	12.98a	17.73a
33.84a	14.59a	21.05a
8.26b	6.61b	9.30b
5.32b	4.25b	5.67b
5.18b		



NH_3-N kg ha^{-1}	% of total N	% of TAN applied
21.53a	22.10a	29.33a
17.82a	17.99ab	21.58ab
12.05ab	9.64ab	12.85ab
9.93ab	7.94b	11.17b
2.65b		

NH_3-N kg ha^{-1}	% of total N	% of TAN applied
56.07a	32.83	49.98
50.41a	34.24	51.49
18.22ab	14.58	19.43
8.25b	6.60	9.28
4.21b		

Figure 6. Cumulative ammonia volatilisation, and expressed as a percentage of the total N and of the total ammonium nitrogen (TAN) applied per fertilisation in year 1 (a, b, c) and year 2 (d, e) at Site 1. C: control; PS: pig slurry; PS+DMPP: pig slurry with DMPP; ANS-26: ammonium nitrosulphate; ENTEC®: ANS-26 with DMPP. The statistical analysis was only carried out until the 22nd day of the sampling period in order to compare the years and fertilisations. Different letters within columns indicate significant differences between treatments ($p < 0.05$, Tukey's test).



a) Pre-sowing

NH_3-N kg ha^{-1}	% of total N	% of TAN applied
17.27a	17.62a	19.89
13.14ab	13.40ab	15.13
12.17ab	12.17ab	17.11
8.25bc	8.25b	11.00
4.41c		

b) After 1st cut

NH_3-N kg ha^{-1}	% of total N	% of TAN applied
38.84a	22.34a	35.25a
20.41b	11.74b	18.52b
8.91bc	5.94b	8.35b
7.35bc	4.90b	6.54b
5.03c		

c) Pre-sowing

NH_3-N kg ha^{-1}	% of total N	% of TAN applied
10.37a	8.42a	15.03a
9.58a	7.78a	13.88a
3.94b	3.94b	5.53b
3.79b	3.79b	5.05b
2.69b		

d) After 1st cut

NH_3-N kg ha^{-1}	% of total N	% of TAN applied
31.02a	19.92a	25.72a
29.88a	19.19a	24.77ab
16.62ab	11.08ab	14.77ab
9.87b	6.58b	9.31b
2.04b		

Figure 7. Cumulative ammonia volatilisation, and expressed as a percentage of the total N and of the total ammonium nitrogen (TAN) applied per fertilisation in year 1 (a, b) and year 2 (c, d) at Site 2. C: control; PS: pig slurry; PS+DMPP: pig slurry with DMPP addition; ANS-26: ammonium nitrosulphate; ENTEC®: ANS-26 with DMPP addition. The statistical analysis was only carried out until the 22nd day of sampling in order to make comparisons between years and fertilisations. Different letters within columns indicate significant differences between treatments ($p < 0.05$, Tukey's test).

Supplementary material

Tables

Table 8. Relationship between daily ammonia volatilisation and driving factors (average daily temperature and daily rainfall).

Fertilisation	NH_3-N (kg ha^{-1})							
	Site 1				Site 2			
	Year 1		Year 2		Year 1		Year 2	
	T (ºC)	Rainfall (mm)	T (ºC)	Rainfall (mm)	T (ºC)	Rainfall (mm)	T (ºC)	Rainfall (mm)
Pre-sowing	-0.06 (11)	0.26 (11)*	0.54 (17)*	0.02 (17)	0.33 (12)*	-0.07 (12)	0.40 (16)*	-0.31 (16)*
After 1st cut	-0.37 (20)*	-0.12 (20)*	-0.26 (15)*	-0.13 (15)*	0.09 (16)	-0.09 (16)	0.14 (15)*	-0.00 (15)
After 2nd cut	0.18 (11)*	-0.11 (11)	-	-	-	-	-	-

Numbers in brackets indicate the total data used in each correlation and per each treatment.

***:** Significant ($p < 0.05$); ns: not significant.

Table 9. ANOVA of the effect of different variables (average daily temperature and average maximum daily temperature, rainfall plus irrigation and wind speed) and their interactions with statistical significance ($p < 0.05$) on ammonia volatilisation.

Driving factor	NH_3-N (kg ha^{-1})								
	Site 1					Site 2			
	Year 1			Year 2		Year 1		Year 2	
	Pre-sowing	After 1st	After 2nd	Pre-sowing	After 1st	Pre-sowing	After 1st	Pre-sowing	After 1st
Average T (ºC)	*	ns	ns	ns	ns	ns	ns	ns	ns
Average maximum T (ºC)	*	ns	ns	ns	ns	ns	ns	ns	ns
Rainfall+Irrigation (mm)	*	ns	ns	ns	ns	-	-	-	-
Wind speed (m s^{-1})	ns	ns	ns	ns	ns	ns	ns	ns	ns
Aver. T x Rainfall+Irrigation	*	ns	ns	ns	ns	-	-	-	-
Average max. T x Rainfall+Irrigation	*	ns	ns	ns	ns	-	-	-	-

Figures

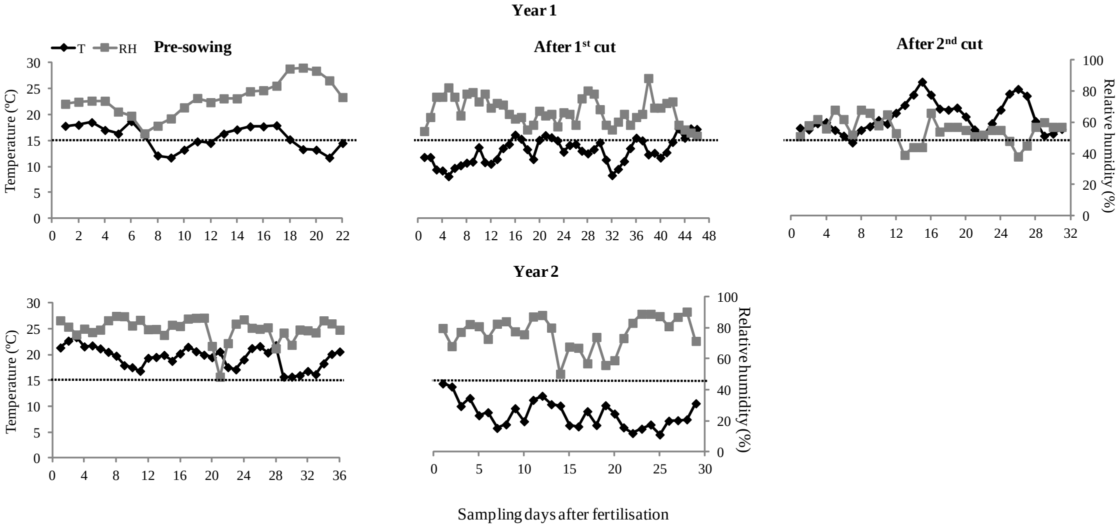


Figure 3. Daily temperature and relative humidity during sampling in years 1 and 2 at Site 1. T: temperature; RH: relative humidity. The dotted line is the average air temperature during the period represented.

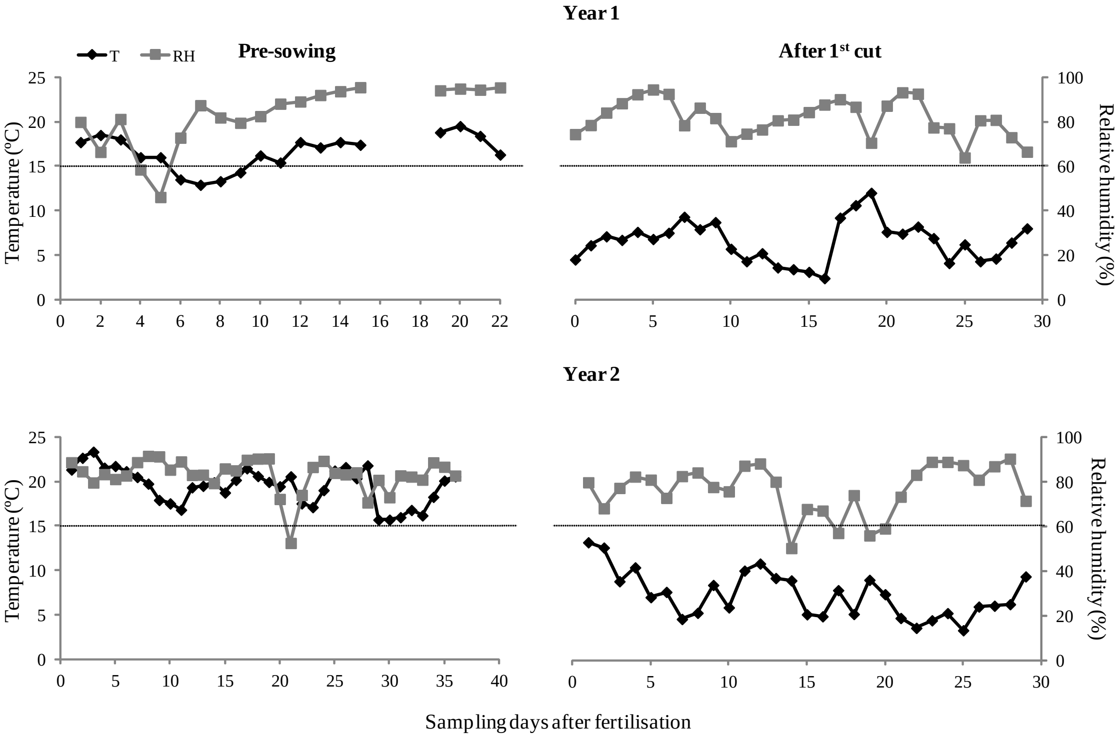


Figure 4. Daily temperature and relative humidity during sampling in years 1 and 2 at Site 2. T: temperature; RH: relative humidity. The dotted line is the average air temperature during the period represented.

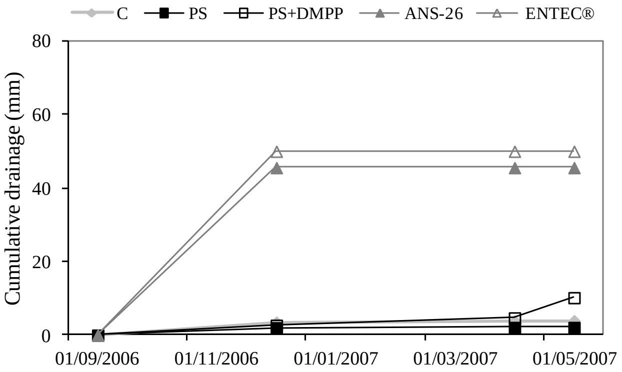


Figure 5. Cumulative drainage collected by the lysimeters below 90 cm depth.

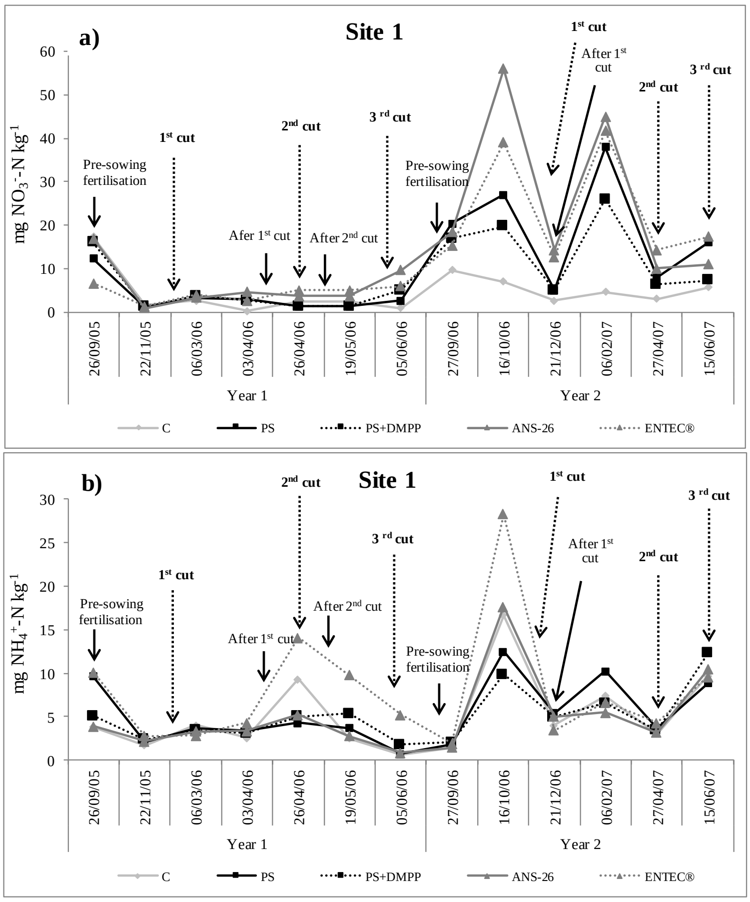


Figure 8. Soil nitrate (a) and ammonium (b) nitrogen contents per treatment before the pre-sowing fertilisation and before the 1st cut and after each cut, in year 1 and year 2, at Site 1.

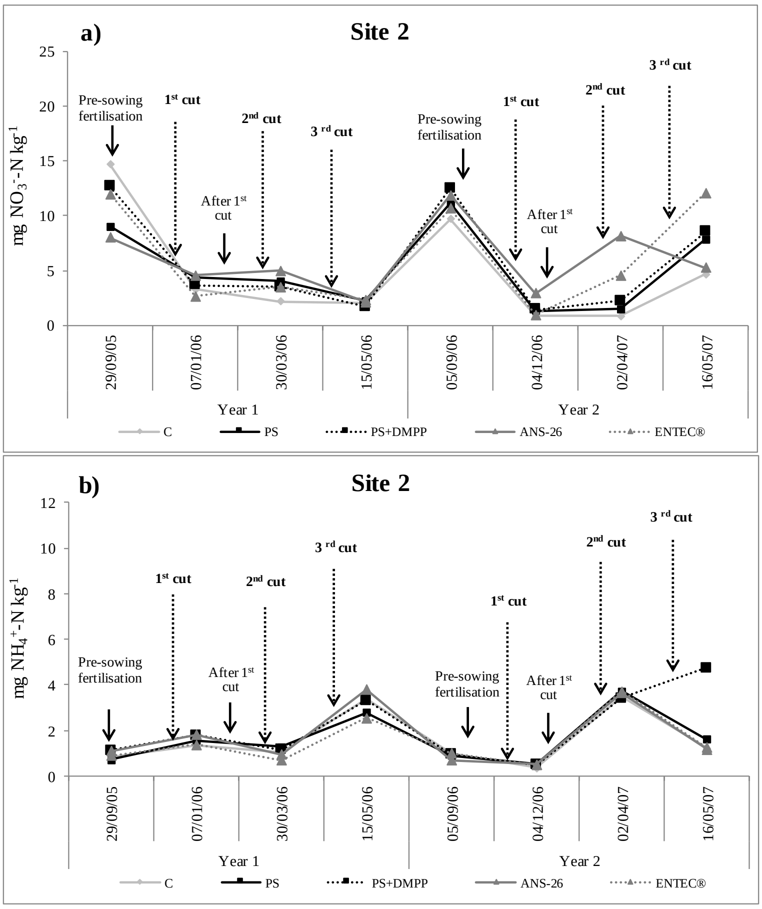


Figure 9. Soil nitrate (a) and ammonium (b) nitrogen contents per treatment before pre-sowing fertilisation and after each cut in year 1 and year 2, at Site 2.