



Laboratory assessment of ammonia volatilization from pig slurries applied on intact soil cores from till and no-till plots

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Abstract

Aim of study: Agricultural activities are the main source of volatilized ammonia (NH₃). Maximum rates are reached within a few hours after slurry application. This study aimed to evaluate the influence of soil texture, tillage and slurry dry matter (DM) on NH₃ volatilization.

Area of study: Mediterranean semiarid environments (NE Spain).

Material and methods: Ammonia volatilization from pig slurry directly applied on the soil surface was quantified in the laboratory, in soil samples from two experimental sites with different soil textures: silty loam and sandy loam. Field treatments consisted of two tillage management practices: till by disc-harrowing or no-till. At topdressing (cereal tillering), tillage treatments were combined with slurries of different DM contents applied onto the silty loam soil. Measurements were done for two cereal cropping seasons and during the period of maximum NH₃ flux (12 h after slurry application). A photoacoustic analyzer was used.

Main results: Slurry spreading at sowing resulted in low volatilization (0.7-9% of NH₄⁺-N applied) as it also did at topdressing (0.3-1.4% of NH₄⁺-N applied). At sowing, ammonia volatilization from high DM slurry (>7.5%) was significantly enhanced by no-till in both soils. At topdressing, this result was also found in records on silty loam soil. No differences were found between tillage systems when slurry of low DM content was applied, whatever the soil texture and application moment. Although NH₃ volatilization was probably affected by the laboratory conditions, the comparisons between treatments were still valuable.

Research highlights: Ammonia volatilization abatement can be improved (<1 kg NH₃-N ha⁻¹) if fertilization is done after crop establishment using low DM slurries (<3.5%).

Additional keywords: calcareous soils; fertilization; Mediterranean agricultural systems; NH₃; no-till; soil texture; winter cereals.

Abbreviations used: DM (dry matter); HDM (high dry matter); LDM (low dry matter); NT (no-tillage); T (tillage); TAN (total ammonium-N).

Authors' contributions: Field maintenance was done by ADBS and EGL; ADBS, FDO and MRTE contributed to the design of the experiment; SCM and EGL were in charge of soil sampling; SCM and MRTE contributed to measurements; SCM wrote the initial version of the manuscript and performed data analysis with ADBS; ADBS, MRTE and SCM improved the final manuscript according to the suggestions and comments of the reviewers. All authors read and approved the final version of the manuscript.

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Introduction

Agricultural activities are the main origin of ammonia (NH_3) volatilization (Metz *et al.*, 2007). Mineral N fertilizers and the application of manures lead to approximately 60% of the volatilized NH_3 (Cameron *et al.*, 2013). Ammonia emissions from nitrogen fertilizer application negatively impact both the quality of the environment and human health; therefore, there is a need to develop methods to reduce NH_3 volatilization, to reduce the wastage of fertilizer resources, and to improve nitrogen use efficiency (Lam *et al.*, 2019).

Ammonia volatilization is influenced by several factors such as soil type and humidity, fertilizer characteristics and tillage systems (Sommer *et al.*, 2003). Calcareous soils are prone to volatilization (Iqbal *et al.*, 2013). Ammonia volatilization is also related to the dry matter (DM) content of the applied fertilizer as it can affect soil pore sealing and infiltration, causing ponding at the soil surface which, in turn, might increase NH_3 volatilisation (Sommer *et al.*, 2003). Fine textured soils with relatively high clay content have smaller pores and when developed under the influence of similar vegetation and climatic conditions generally show higher cation exchange capacity, organic matter content and water holding capacity than coarse textured soils. Consequently, fine textured soils are less conducive to volatile loss of ammonia than sandy soils (Sommer *et al.*, 2003). However, the higher tortuosity of fine textured soils can lead to a reduced rate of infiltration of surface-applied mineral fertilizer or pig slurry and its reaction products, which may result in significant NH_3 loss from the surface (Stevens *et al.*, 1989).

As a conservation tillage practice, no-tillage (NT) has been adopted worldwide as a way to control soil erosion or to increase soil organic carbon sequestration, which usually leads to a yield increase and, subsequently, to higher economic benefits for farmers. However, some reports have drawn attention to a volatilization increase with NT compared to conventional tillage (Mkhabela *et al.*, 2008), through the improvement of soil urease activity and the greater presence of crop residues on the surface that prevent the infiltration of the N fertilizer (dissolved in water or as animal effluent) into soil (Mkhabela *et al.*, 2008; Rochette *et al.*, 2009).

These factors are of special relevance in European NH_3 volatilization. Europe produces 25% of the world's pigs (*Sus scrofa domesticus*), and nowadays Spain is the biggest producer (MAGRAMA, 2017). In Spain pig slurries are mainly directly applied as fertilizers to cropped fields, in which no-tillage is implemented over $700 \cdot 10^3$ ha (MAGRAMA, 2017).

The first 20 hours after slurry application are the most crucial timespan in terms of NH_3 -N losses. Thompson *et al.* (1990) and Bittman *et al.* (2005) found that 57-85% of total NH_3 -N volatilization occurred in the first 24 hours

after applying mineral fertilizer or surface applied manure. In a recent study Yagüe *et al.* (2019) found that the maximum NH_3 flux volatilization was always observed during the earliest period of measurements (3.5-5 hours) after slurry spreading.

Estimated NH_3 volatilization in Spanish soils has been found to range from 7% to 78% of the total ammonium N applied with mineral fertilizers or pig slurries (Bosch-Serra *et al.*, 2014). Surface applications promoted by legislators at different administrative levels (EU, 2016; DOGC, 2019) can reduce these figures close to 1% of total N applied, mainly at cereal tillering applications (Yagüe *et al.*, 2019). However, if all plant residues are left on the surface, and slurry infiltration is constrained, volatilization might rise.

Bosch-Serra *et al.* (2014) and Yagüe *et al.* (2019) are the only papers we could find that measured NH_3 volatilization in semiarid areas under different fertilization strategies (pig slurry and/or mineral fertilizers) applied at sowing and at tillering to winter cereals. In both studies, the NH_3 volatilization was measured by using dynamic chambers coupled with acid traps. Nevertheless, using acid traps is time-consuming (*e.g.* acid solution collection, separate laboratory analysis for NH_4^+ determination), with variable and, in general, low temporal resolution, especially when only small amounts of NH_3 are emitted. This second drawback leads to loss of information, especially during the first hours after fertilizer application when emission rates vary rapidly. In addition, to date, none of these studies performed in Mediterranean areas used the system made up of the dynamic chamber method and a photoacoustic analyser. Worldwide there are a few studies on the accuracy and reliability of the photoacoustic analyser with dynamic chambers for NH_3 emission assessment (Monaco *et al.*, 2012).

We hypothesized that tillage (T) might abate volatilization when compared with NT. This abatement of NH_3 volatilization could be further improved when combining T with the use of slurries of low DM content, although the results could be affected by soil texture due to its influence on slurry infiltration. Hence, the objectives of this study were: i) to compare NH_3 volatilization under different tillage systems (no-tillage *vs.* disc-harrowing tillage) when pig slurry with high DM content (>7%) was applied at sowing over two different textured soils: silty loam and sandy loam; and ii) to evaluate the influence of the DM content of pig slurries applied at the cereal tillering stage (topdressing) on volatilization under different tillage systems (no-tillage *vs.* disc-harrowing tillage) in the silty loam soil and the sandy loam soil.

The results of this study might help decision makers to develop environmental and agronomic NH_3 abatement management practices (tillage systems, slurry rates and their application periods).

Material and methods

Site description and experimental design

The framework of this work was within long-term pig slurry fertilization experiments in which comparison of the tillage systems (NT and T) was implemented two years before the present study started.

The experimental sites (two) are located in the Ebro river basin (NE Spain) on calcareous soils (Table S1 [suppl.]). The soil of the first site (Oliola, 41°51'29"N, 1°05'10"E at 443 m a.s.l.) has a silty loam texture and it is classified as a Typic Xerofluvent (Soil Survey Staff, 2014) (Table S1 [suppl.]). The soil of the second site (Torroella de Montgrí, 42°02'30"N, 3°07'35"E at 31 m a.s.l.) has a sandy loam texture and it is classified as a Xerocept calcixerollic (Soil Survey Staff, 2014). At both experimental sites, the climate is semiarid Mediterranean, with high summer average temperatures (>20° C), low annual precipitation (<650 mm yr⁻¹) and high average reference crop evapotranspiration (>1000 mm yr⁻¹).

In Oliola, two experiments were set up. In both experiments, the tillage systems (NT and T by disc-harrowing) was the main treatment. In the first experiment (randomized block design, three replicates), all plots received at sowing (without topdressing) pig slurry with a high DM content (~10%, HDM, Table S2 [suppl.]). In the second experiment, at the cereal tillering stage, two types of slurries (high, HDM, and low, LDM (<3.5%) dry matter content, Table S2 [suppl.]) were applied to NT and T plots (topdressing, unique application) following a split-block design with three replicates.

In Torroella de Montgrí, two experiments were set up. In both experiments, the same two tillage systems (NT and T) were established (randomized block design, three replicates). In the first experiment, pig slurry was applied before cereal winter sowing (Table S2 [suppl.]); in the second experiment LMD slurry was applied at the cereal tillering stage (Table S2 [suppl.]).

The experiments and treatments defined for each site and year are summarised in Table 1. Under field

conditions, disc-harrowing between 0.20 and 0.25 m was used to incorporate the stubble before sowing (October or November) and later on to bury the slurry. Tillage was performed not later than 12h following slurry application. At tillering (February or March) the fertilizer was not buried. Barley (*Hordeum vulgare* L.) was sown under rain-fed conditions. Every cropping season, straw was removed from the fields according to farmers' practice.

Ammonia volatilization: gas sampling and analysis

The volatilization study was done in the laboratory during two cropping seasons (2011-2012 and 2012-2013) in the silty loam soil, and during one crop season (2011-2012) in the sandy loam soil. The day before slurry application in the field, undisturbed soil cores were taken, using PVC tubes, which were 15 cm long and 7 cm in diameter, from two replicates (blocks). Each pig slurry applied on the field was analyzed for its main physical and chemical characteristics (Table S2 [suppl.]). A representative aliquot of the slurries was brought to the laboratory and stored at 4°C until the following day, when each type of slurry was applied to each soil at the same dose as it had been applied in the field (Table 1) and NH₃ volatilization monitoring started in the laboratory. The plant residue coverage in the undisturbed soil of NT cores was estimated to be close to 65% on the silty loam soil and to 55% on the sandy loam soil. Residues were maintained on the surface of the NT soil cores. No residues were left on the T soil surface.

Each soil core was placed in a chamber system coupled to a photoacoustic analyzer, in order to monitor NH₃ volatilization (Monaco *et al.*, 2012). The chamber system for NH₃ volatilization consisted of a sealed glass jar (1.5 L) in which the undisturbed soil cores were placed. Each glass jar was equipped with a two-way key that allowed direct connection between soil cores and the photoacoustic analyzer (Innova 1412 Photoacoustic Multigas Monitor) via a Teflon® tube. The temperature in the laboratory followed the open-air fluctuations. Air samples from inside

Table 1. Annual doses of pig slurry applied to the experimental sites with different textures: silty loam (SIL, site 1) and sandy loam (SL, site 2). The monitored treatments during two cropping seasons (2011-2012, 2012-2013) were defined according to the studied variables: moment of slurry application and tillage system (NT: no-tillage; T: tillage by disc-harrowing).

Framework	Slurry applied before sowing			Slurry applied at topdressing				
	SIL	SIL	SL	SIL	SIL	SIL	SIL	SL
Soil texture	SIL	SIL	SL	SIL	SIL	SIL	SIL	SL
Date	Oct-11	Oct-12	Nov-11	Feb-12	Feb-12	Feb-13	Feb-13	March-12
Slurry type	HDM	HDM	HDM	HDM	LDM	HDM	LDM	LDM
Dose (kg N ha ⁻¹)	185	130	245	113	123	99	80	158
Tillage system	T/NT	T/NT	T/NT	T/NT	T/NT	T/NT	T/NT	T/NT

the glass jar were taken immediately after pig slurry was directly applied on the soil surface (simulating a trail hose field application). The air stream NH_3 concentration was measured semi-continuously during 12 h after application. The detection limit of the photoacoustic analyzer was 0.2 ppm of NH_3 . The obtained data were converted to the actual $\text{NH}_3\text{-N}$ volatilization surface rate by calculating the air flux rate using the time elapsed between measurements and the sampled air volume. The cumulative $\text{NH}_3\text{-N}$ volatilization throughout the study period was calculated by integrating the volatilization curves over time.

In the laboratory, the minimum and maximum temperatures recorded during the 12 h after slurry application (sowing time) on the silty loam soil were 15.5 and 19 °C in 2011, while in 2012 they were 12 and 17°C. In 2013, at topdressing, the recorded temperatures for the same period of time were 8 and 12°C. When measuring volatilization in soil samples from the sandy loam soil, the recorded minimum and maximum temperatures were 18.5 and 21°C at sowing time (2011), while at topdressing (2012) they were 11.5 and 17°C. Laboratory conditions (*i.e.* absence of direct solar radiation, wind) may have reduced volatilization, nevertheless the comparisons between treatments are still valuable.

Data analysis

The effect of the tillage systems was evaluated by analysis of variance. Separation of means was done by the Duncan multiple range test ($\alpha = 0.05$). In the silty loam

soil at cereal tillering, the effect of tillage systems combined with slurries of different DM content on cumulative NH_3 volatilization was analyzed according to the field split-block design in order to observe potential interactions between both variables. In the analysis, the interactions between replicates and tillage or between replicates and slurry DM were used as an error term. The statistical package JMP version 12 (SAS Institute), was used.

Results and discussion

Before barley sowing, the cumulative NH_3 volatilization for the 12 h period after slurry spreading followed a similar pattern for both soil textures, and it was significantly affected by the tillage system (Table 2). The highest NH_3 volatilization corresponded to the NT system (Table 2) where the recorded volatilization ranged from 1.74 in the sandy loam soil up to 7.14 kg $\text{NH}_3\text{-N ha}^{-1}$ in the silty loam soil which equaled 0.7% and 5.5% of the total N applied, respectively. In the silty loam soil, NH_3 volatilization was approx. 55%-104% higher from NT than from T. At cereal tillering stage (2012) in the sandy loam soil, when low DM slurry (3.2%) was surface applied (Table S2 [suppl.]), NH_3 volatilization average was also higher from NT than from T, but the difference was not significant (Table 2). In the silty loam soil, an interaction was found in 2012 between tillage system and slurry DM (Table 3) which indicated that NT only boosted NH_3 volatilization if HDM slurry was applied. In 2013, no interaction was found in the statistical analysis between

Table 2. Effect of tillage systems (T: tillage by disc-harrowing, NT: no-tillage) on cumulative NH_3 average volatilization during the 12 h (\pm standard deviation) following surface application of pig slurry. Slurry with high dry matter content ($>7\%$) was applied in different cereal cropping seasons before sowing (Oct-Nov) and over two soils (from two sites) with different texture: silty loam (SIL) and sandy loam (SL). Volatilization was also quantified when low dry matter slurry (3.2%) was surface applied at topdressing (March) in a SL soil.

Period (mm-yy)	Site/ Texture	Tillage system	Slurry application			Cumulative NH_3 volatilization	
			Dose (t ha ⁻¹)	Total N (kg ha ⁻¹)	$\text{NH}_4^+\text{-N}$ (kg ha ⁻¹)	Anova data ^[a] (df/ MS/ <i>p</i>)	$\text{NH}_3\text{-N}$ ^[b] (kg ha ⁻¹)
Oct-11	Site 1/ SIL	T	25	185	111	1/ 6844357.4/	2.52±0.02 b
		NT	25	185	111	0.009	5.14±0.34 a
Oct-12	Site1/ SIL	T	22	130	80	1/ 6431592.1/	4.60±0.27 b
		NT	22	130	80	0.01	7.14±0.23 a
Nov-11	Site 2/ SL	T	46	245	166	1/ 60940.6/	1.49±0.07 b
		NT	46	245	166	0.043	1.74±0.01 a
March-12	Site 2/ SL	T	34	158	127	1/ 27146.9/	0.91±0.07
		NT	34	158	127	0.43	1.08±0.23

^[a] Anova analysis; df: degrees of freedom; MS: mean square (data units were g ha⁻¹); *p*: significance. ^[b] Average values with different letters are significantly different according to the Duncan multiple range test for a $\alpha=0.05$ probability level.

Table 3. Average cumulative NH₃ volatilization during 12 h (\pm standard deviation) from slurries with different dry matter content (HDM: high dry matter, >7%; LDM: low slurry dry matter, <3.5%) applied on the soil surface with different tillage treatments (T: tillage by disc-harrowing, NT: no-tillage) at cereal tillering stage (topdressing) over the silty loam soil (site 1) in 2012.

Slurry type	Slurry application			Tillage system	Cumulative NH ₃ volatilization	
	Slurry dose (t ha ⁻¹)	Total N (kg ha ⁻¹)	NH ₄ ⁺ -N (kg ha ⁻¹)		Anova data ^[a] (df/ MS/ p)	NH ₃ -N ^[b] (kg ha ⁻¹)
HDM	16	113	75	T	1/ 207075/	0.376 \pm 0.06 b
HDM	16	113	75	NT	0.030	0.831 \pm 0.11 a
LDM	51	123	85	T	1/ 7834.3/	0.470 \pm 0.13
LDM	51	123	85	NT	0.093	0.381 \pm 0.10

^[a] Anova analysis by tillage system when a previous analysis showed a significant interaction between slurry dry matter and tillage system; df: degrees of freedom; MS: mean square (data units were g ha⁻¹); p: significance. ^[b] Average values followed by different letters are significantly different according to the Duncan multiple range test for a $\alpha=0.05$ probability level.

tillage and slurry DM. Also, no differences between treatments were found (Table S3 [suppl.]). However, the amounts of ammonia detected in both cases were low (<1.4 kg NH₃-N ha⁻¹).

The importance of soil surface roughness in volatilization reduction was demonstrated by Bacon *et al.* (1988). In the studied T plots, large pores enhanced slurry infiltration. As slurry infiltrates, it reduces the pool of total ammonium-N (TAN) at the soil surface, NH₃ concentration is also reduced and therefore, subsequent volatilization decreases (Thompson *et al.*, 1990), and NH₃-N is protected from volatilization by adsorption onto soil colloids (Sadeghpour *et al.*, 2015). As a result, NT showed higher volatilization potential, in agreement with Rochette *et al.* (2009). This fact also agrees with the abatement when LDM slurries were used (Table S3 [suppl.]), as pig slurry with a lower solid content infiltrates more readily. The concept of reducing NH₃ volatilization by facilitating better infiltration of slurry (low DM content) into soil (*i.e.* decanted slurry, mechanically assisted filtration) is supported by Bhandral *et al.* (2009) and Bosch-Serra *et al.* (2014).

When comparing volatilization at the two sites it was higher from the silty loam soil (2.52 to 7.14 kg NH₃-N ha⁻¹) than from the sandy loam soil (0.91 to 1.73 kg NH₃-N ha⁻¹) despite the higher ammonium N rate applied to this latter soil (Table 2). The results can be explained in terms of slurry infiltration enhancement in coarse textured soils.

It is important to point out that the cumulative NH₃ volatilization obtained in the present experiment was much lower than that described in some other studies with similar soils (pH >7) and dry climate characteristics, such as the one from Pacholski *et al.* (2006), who found that cumulative volatilization could be up to 48% of total applied N. However, the cumulative volatilization during the first 12 h following slurry application at tillering was <2% of the applied TAN in all the treatments, which was

close to the <3% of the applied TAN observed by Powell *et al.* (2011).

The results reinforce the importance of combining different agronomic practices to abate volatilization. The present study shows that the surface application of pig slurry at agronomic doses, combined with soil tillage, can be an effective strategy to reduce NH₃-N volatilization in rainfed Mediterranean semiarid environments. The success of abatement measures, whatever the tillage system, will be more evident with slurries of low DM content.

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