

Laboratory assessment of ammonia emission after soil application of treated and untreated manures

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SUMMARY

Ammonia (NH₃) volatilization from soil-applied manure not only causes environmental pollution but also reduces the fertilization value of the manure. Anaerobic digestion and solid/liquid separation alter the physical and chemical characteristics of slurry, which affect NH₃ emissions after application. The present study measured potential laboratory NH₃ losses from different manures, untreated pig slurry and the liquid fractions of each untreated and digested slurry, after their application to two different soil types. The experiment was carried out in dynamic chambers using a photoacoustic infrared gas analyser to determine the NH₃ concentration in the air stream directly.

The estimated values of nitrogen (N) emitted for surface-applied, untreated pig slurry were 0.26 ± 0.064 mg per mg of applied total ammoniacal nitrogen (TAN). For the liquid fractions of pig slurry and digested pig slurry, results were 0.13 ± 0.064 and 0.16 ± 0.064 mg/mg, respectively. Initial NH₃-N emission rates from surface-applied, untreated pig slurry were higher than those measured for either liquid fraction; in the case of the untreated pig slurry, half was emitted in the first 4.9 h of measurement. Silty-loam soil showed a higher N emission than loam soil with surface-applied slurries. This result was probably due to the higher infiltration rate of loam soil, even offsetting the effect of its high soil pH. Immediate manure incorporation into the soil was shown to reduce NH₃ emissions by 82%. Results demonstrated that a method combining dynamic chambers with a photoacoustic gas analyser was as reliable as the widely used acid traps method. Moreover, direct measurement with the gas analyser permits an increasing temporal resolution that gave a high-quality description of the NH₃ emission dynamic.

INTRODUCTION

Animal husbandry and manure management account for more than 0.80 of total ammonia (NH₃) emissions from European agriculture, estimated at 2.9 Mt/year; land application of manure, especially slurry, contributes a large proportion of these losses, and represents between 0.30 and 0.40 of total emissions from livestock production (Hutchings *et al.* 2009).

Ammonia volatilization reduces the manure nitrogen (N) use efficiency in crop production and increases the uncertainty of crop N balance and optimal fertilizer N rate calculations (Sommer *et al.* 2004b).

From an environmental perspective, the loss of NH₃ to the atmosphere causes negative effects through acidification and eutrophication of natural ecosystems, as well as via secondary particulate matter formation (Sutton *et al.* 1998; Goebes *et al.* 2003). For these reasons, international and European agreements have established regulations to limit this pollutant, which requires calculating national inventory levels of the contribution from agriculture to total emissions (Erisman *et al.* 1998). These inventories are based on emission factors that, in the advanced method of calculation, are also specific for the emissions produced during manure spreading.

Ammonia emissions from manure applied to the soil are shaped primarily by physical and chemical

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processes, and secondarily by biological ones. Environmental conditions (i.e. wind speed, temperature, precipitation and humidity), manure properties (i.e. dry matter content, viscosity, pH and TAN content), soil properties (i.e. pH, cation exchange capacity, porosity and infiltration rate) and the interaction between slurry and soil, as well as the slurry distribution method and the rate of application have all been shown to cause variations in the amount of NH_3 lost, from approximately nil to 0.60 of the total ammoniacal-N (TAN) applied (Sommer *et al.* 2003). Under constant environmental conditions, the pattern of NH_3 volatilization shows high loss rates immediately after slurry application due to the high initial concentration of TAN and the pH increase in the manured-soil surface (Søgaard *et al.* 2002). The volatilization rate then decreases rapidly as a result of a reduction in the concentration of ammonium (NH_4^+) in the fertilized soil surface from volatilization, infiltration and nitrification. The pattern of NH_3 volatilization after soil application of slurry is usually described in the literature by the Michaelis–Menten type equation (Sommer & Ersbøll 1994; Søgaard *et al.* 2002; Misselbrook *et al.* 2005).

Anaerobic digestion, solid/liquid separation of liquid manure and other manure treatments may also influence gaseous losses because they change the physical and chemical characteristics of manure, and affect the extent of emissions after field spreading (Amon *et al.* 2006; Sommer *et al.* 2006; Petersen & Sørensen 2008). Solid/liquid separation reduces the dry matter (DM) in the liquid portion of the slurry, which boosts slurry infiltration. Anaerobic digestion increases TAN content and manure pH, which raises the NH_3 volatilization potential, and decreases DM content which increases slurry infiltration rates and decreases NH_3 emissions.

The effect of manure and soil properties on NH_3 emissions has been extensively investigated in field (Thompson & Meisinger 2002; Sommer *et al.* 2004a; Misselbrook *et al.* 2005) and laboratory studies (Amon *et al.* 2006; Sommer *et al.* 2006). For laboratory assessment, the dynamic chamber is the most utilized method, in which NH_3 emitted from manure is collected in acid solutions (sulphuric, boric or orthophosphoric) and determined via titration (Le Cadre *et al.* 2005). Recently, slurry infiltration (Sommer & Jacobsen 1999), slurry type (Sommer *et al.* 2006), urease inhibitor (Watson *et al.* 2008) and no-till (Rochette *et al.* 2009) effects on potential NH_3 emissions were investigated 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.

Infrared photoacoustic spectroscopy has recently become popular for analysing several gases (e.g. CO_2 , N_2O , CH_4 and NH_3) in air samples, due to its high accuracy and selectivity (Hoy 1995; Berg *et al.* 2006; Dinuccio *et al.* 2008). Commercial applications of this technology allow automated air sampling, high temporal resolution of measurements and direct data recording and display of electronic datasets. Mathematical descriptions of the emission dynamics can consequently be performed more effectively. However, to date, few studies exist on the accuracy and reliability of using photoacoustic spectroscopy with dynamic chambers for NH_3 emission assessment.

The present work reports the results of a laboratory assessment of NH_3 emission after soil application of different treated and untreated pig slurry (i.e. no treatment, solid/liquid separation, anaerobic digestion followed by solid/liquid separation) using a system composed of dynamic chambers and a photoacoustic infrared gas analyser. The reliability of this method was evaluated via comparison to the most commonly used acid traps method.

MATERIALS AND METHODS

Treatments and soils

Three manure treatments were compared: untreated pig slurry (Untreated S), the liquid fraction of untreated pig slurry (Untreated S liquid) and the liquid fraction of anaerobically digested pig slurry (Digested S liquid). The pig slurry samples were sourced from a house for grower–finisher heavy pigs (160–180 kg/head), raised on a fully slatted-floor. Anaerobic digestion was performed in continuously fed laboratory reactors under mesophilic conditions (38–39 °C), with a hydraulic retention time of 26 days and an organic loading rate of 1.35 kg volatile solids/ m^3/d . The solid and liquid fractions of untreated and digested slurries were separated using a laboratory centrifuge at 7000 rpm (Mantovi *et al.* 2009). All manure types were stored for a total of 120 days in 50-litre covered tanks at ambient temperatures to simulate farm storage before

Table 1. *Main characteristics of the manures utilized in the experiment*

Type of slurry	pH	DM* (g/kg)	TOC [†]	Total N	TAN [‡]	Total P	Total K
Untreated slurry	8.3	28	8.0	3.8	2.9	1.06	2.93
Untreated slurry liquid	8.5	13	2.0	2.2	2.0	0.17	3.33
Digested slurry liquid	8.7	13	2.0	2.0	1.8	0.13	3.35

* DM, dry matter.

† TOC, total organic carbon.

‡ TAN, total ammoniacal nitrogen.

Table 2. *Main characteristics of the two soils utilized in the experiment*

Soil	Sand (mg/g)	Silt (mg/g)	Clay (mg/g)	pH	TOC* (mg/g)	Total N (mg/g)	C/N	CEC meq/100 g
Silty-loam	158	756	86	6.1	8.4	0.81	10.4	12.5
Loam	484	431	85	8.2	8.3	0.83	10.0	8.2

* TOC, total organic carbon.

spreading. The different manures were then analysed for their main physical and chemical characteristics (Table 1) and stored at 4 °C until the start of each trial.

The soil used in the experiment was collected during June 2008 from the tilled top 200 mm layer of two arable soil types in the western Po River Plain (Northern Italy). The two soils were labelled loam and silty-loam, based on their texture classification (Soil Survey Staff 1998) (Table 2). The soils were air dried and sieved at 5 mm to remove plant debris. A sample of each soil was ground (2 mm) prior to analysis for soil characterization. Loam and silty-loam soils have a sub-alkaline and a sub-acid reaction, respectively. While both of these soils were poor in organic matter and total N content, each had a different cation-exchange capacity (loam soil: low; silty-loam: medium) (Sposito 1989; Bourlot *et al.* 2007). The bulk density of the dry soil, rewetted at 0.6 m³/m³ of water filled pore space (WFPS), was measured by drying 100 ml of soil (four replicates) at 105 °C for 3 days; it equalled 1.34 and 1.45 g of dry soil/ml for loam and silty-loam soils, respectively.

Measurement system

The measurement system (Fig. 1) was prepared in a climatic chamber and was composed of six measurement lines connected to a vacuum pump that provided airflow to the system. Each line was equipped with a cylindrical glass jar (3200 ml) for soil sample

placement, a glass jar collector (1100 ml) for air sampling with a photoacoustic trace gas analyser (P-TGA) system (LumaSense Technologies, INNOVA 1412), a flow meter with needle valve for system air flow regulation and a volumetric air meter to measure total air passed through the line. One line was also equipped with a humidity and temperature data recorder to measure environmental conditions in the volatilization chamber. All parts had air-tight connections through input and output ports, using Teflon or nylon tubes with internal diameter of 4 mm. Teflon tubes were utilized to avoid NH₃ adsorption in the system.

The air stream NH₃ concentration was determined using the P-TGA. At each measurement, an air sample was collected by the P-TGA's internal pump by manually connecting it to the jar collector with a Teflon tube. The air suction/measurement cycle totalled 79 s, of which 38 s were for suctioning. The cycle was repeated three to four times until a steady-state NH₃ concentration was attained (Dinuccio *et al.* 2008), which represented the actual concentration of the air stream in the measurement system. The internal pump of the P-TGA had an average air flow rate of 24.7 ml/s so that the total amount of air subtracted from the system for one measurement was 2.82 litres. At the end of each measurement, the NH₃ air entering the volatilization chambers (background) was monitored with the P-TGA and the amount was subtracted from the measured concentration of NH₃. Under the

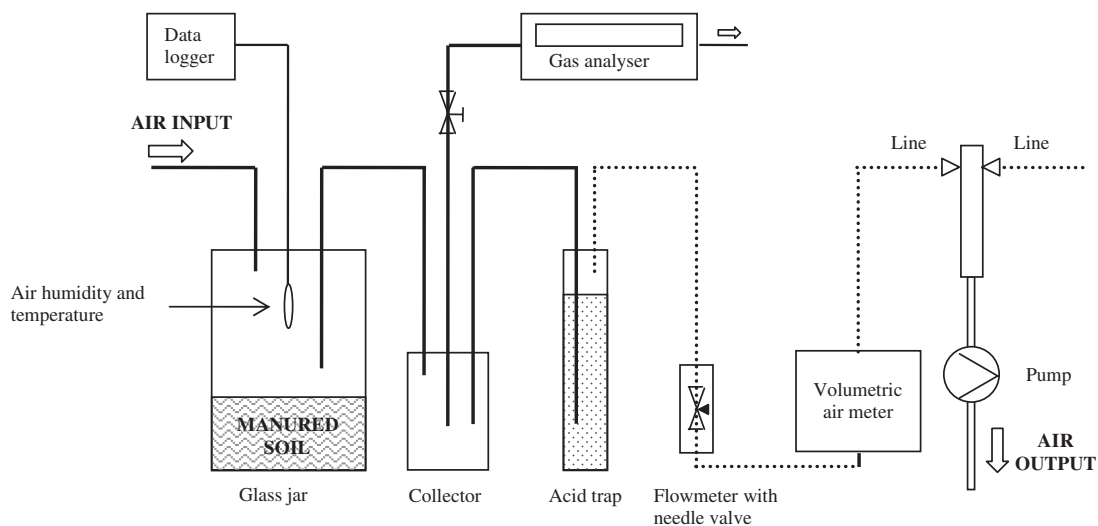


Fig. 1. Measurement system used in the experiment. Unbroken and dotted lines refer to Teflon and nylon tubes, respectively.

operating conditions of this experiment, the limit of detection of the P-TGA was 0.2 mg/kg of NH_3 .

Ammonia volatilization experiment

The experiment was organized on a three randomized complete block design. Six cylindrical glass jars per block (three treatments per two soils) were filled with 1343 and 1451 g of dry soil for loam and silty-loam, respectively, to standardize to a final headspace volume of 2000 ml per jar. WFPS of $0.6 \text{ m}^3/\text{m}^3$ was attained by moistening the samples with 295 and 270 g of deionized water to loam and silty-loam, respectively. A pin-holed plastic film was used to cover each glass jar and then was placed in a climate-controlled room at 20 °C for 6 days to stabilize the samples prior to experimental start. During this period, the jars were weighed each day; the water content was adjusted when necessary.

The experiment began with jar fertilization. Soil samples were fertilized manually on the jar's surface (0.0154 m^2) at a rate of 90.7 kg N/ha. The fertilization time was different for every treatment and was recorded as a t_0 value for each jar. The measurement was then made in the same order as for fertilizer application. Immediately after fertilization, the six jars were closed, randomly connected to the lines of the measurement system and the pump was switched on. The flow rate settled at 2 litres/min, which corresponds to an air renewal rate of one headspace volume per minute in each volatilization chamber (Dinuccio *et al.* 2008).

The experiment was conducted at 20 °C for three different measurement sessions, representing the three blocks, which lasted about 50 h after fertilization.

A parallel experiment was carried out to evaluate the effect of incorporation on NH_3 volatilization from untreated slurry. In this parallel experiment, untreated slurry was manually incorporated into the two soils at the same fertilization rate of the main treatments.

Measurement system evaluation

In order to evaluate the reliability of the method with P-TGA, a simultaneous comparison with the acid trap method was carried out. A Drechsel bottle (250 ml), for the containment of the acid trap solution (80 ml of 0.1 Normal sulphuric acid ($\text{N H}_2\text{SO}_4$) solution), was inserted into each line as showed in Fig. 1. The comparison between methods was carried out during five intervals of c. 3 h each during which the concentration of NH_3 was measured three non-consecutive times for each treatment with P-TGA. At the end of each interval, the acid solutions contained in the acid traps were collected and the Drechsel bottles accurately washed with deionized water in order to remove all the trapped $\text{NH}_4^+\text{-N}$. The Drechsel bottles were then filled with acid trap solution if another interval of measurement was scheduled; otherwise, they were filled with deionized water. The acid traps samples were transferred to volumetric flasks, filled to 250 ml with deionized water and stored at 4 °C until the time for $\text{NH}_4^+\text{-N}$ content analysis with an ionometer. The amount of NH_3 trapped in acid

solution during the measurement run was determined by a multi-meter equipped with a pH/Ion module and an ammonium selective electrode (ionometer, certified detection limit of 0.1 ppm of NH₄⁺), and followed the method described by ISO TC 147/6778 (ISO 1984).

Data processing and analysis

Data measured by P-TGA were converted to an actual NH₃-N emission surface rate by calculating the air flux rate using the elapsed time between the start and end of each measurement interval and their corresponding air volumes.

The emission rates measured for each replication were fitted using a least squares minimization procedure with the derivative of the Michaelis–Menten type equation (Eqn 1) presented by Sommer & Ersbøll (1994):

$$\frac{dN}{dt} = N_{\max} \frac{k_m}{(t + k_m)^2} \quad (1)$$

where t is the time from the fertilization event (h), dN/dt is the emission rate expressed in mg NH₃-N/m²/h, N_{\max} is the total loss of NH₃ (mg NH₃-N/m²) as time approaches infinity, and parameter k_m is the time t when $N = 1/2 N_{\max}$. Moreover, parameter N_{\max}/k_m represents the value of the emission rate (mg NH₃-N/m²/h) when $t=0$.

The above mentioned parameters were analysed using an ANOVA procedure considering manure and soil as fixed effects, and their interaction and block as random effects.

Emission data have been reported throughout the text as mean value ± standard error of the mean (S.E.M.). In order to compare the P-TGA data with the NH₃ trapped in the acid solution, total NH₃-N emitted during each 3 h measurement interval was calculated by integrating the P-TGA data with the fitted equations for the time intervals. A linear regression analysis was carried out to compare these results with the total amount of measured NH₃-N emitted using acid traps. The analysis was performed using the five measurement interval results, excluding the data below the detection limits of the P-TGA and ionometer (final $n=133$).

RESULTS

Measured NH₃-N emission rates shown in Fig. 2 were significantly fitted with the derivative of the Michaelis–Menten equation (average $R^2=0.99$). As expected,

surface-applied treatments showed very high emission rates immediately after fertilization, followed by a strong reduction in a few hours thereafter. In the Michaelis–Menten equation, these results correspond to a high initial emission rate (216.0 mg NH₃-N/m²/h, on average) (Table 3). The low k_m values also confirmed the quick reduction in emission rates during the first hours (6.3 h, on average).

Manure had no significant effect on total N losses, but the ratio total N losses/TAN was significantly affected. Untreated S showed a higher value (0.26 mg/mg) than Untreated S liquid (0.13 mg/mg), while the difference between the Untreated S liquid and the Digested S liquid treatment was not significant. Soil effect was highly significant both for total N losses and total N losses/TAN, showing higher values in silty-loam than in loam.

The initial emission rate was significantly influenced by the manure × soil interaction. Specifically, Untreated S showed higher emission rates in both soils (334.4 mg/NH₃-N/m²/h, on average) v. the value for the liquid fraction of untreated and digested slurry (160.2 and 153.4 mg/NH₃-N/m²/h, respectively), with larger differences in silty-loam than loam soil.

Incorporating untreated slurry caused very low and constant emission rates throughout the experiment (Fig. 2), with a low initial emission rate (7.6 mg NH₃-N/m²/h) and a high k_m value (39.0 h). Incorporated untreated slurry showed low total N losses (308.0 mg NH₃-N/m², averaged across the two soils) (Fig. 3), which represents a reduction of 81.7% compared with surface-applied slurry. Moreover, when slurry is incorporated, there is a tendency for higher emissions in loam than in silty-loam soil, as evidenced by the values of total N losses (537.1 and 79.0 mg NH₃-N/m², respectively).

The linear regression between the P-TGA and the acid traps methods for NH₃-N emission assessment (Fig. 4) was highly significant ($R^2=0.95$). However, when using P-TGA, a small underestimation was indicated by the coefficients (0.925 ± 0.037) and intercept (-4.21 ± 5.72 mg NH₃/m²) of the linear equation.

DISCUSSION

Treatments comparison

For surface-applied untreated pig slurry, total N losses averaged 0.26 ± 0.064 mg/mg of applied TAN across the two soils. These values were similar to those

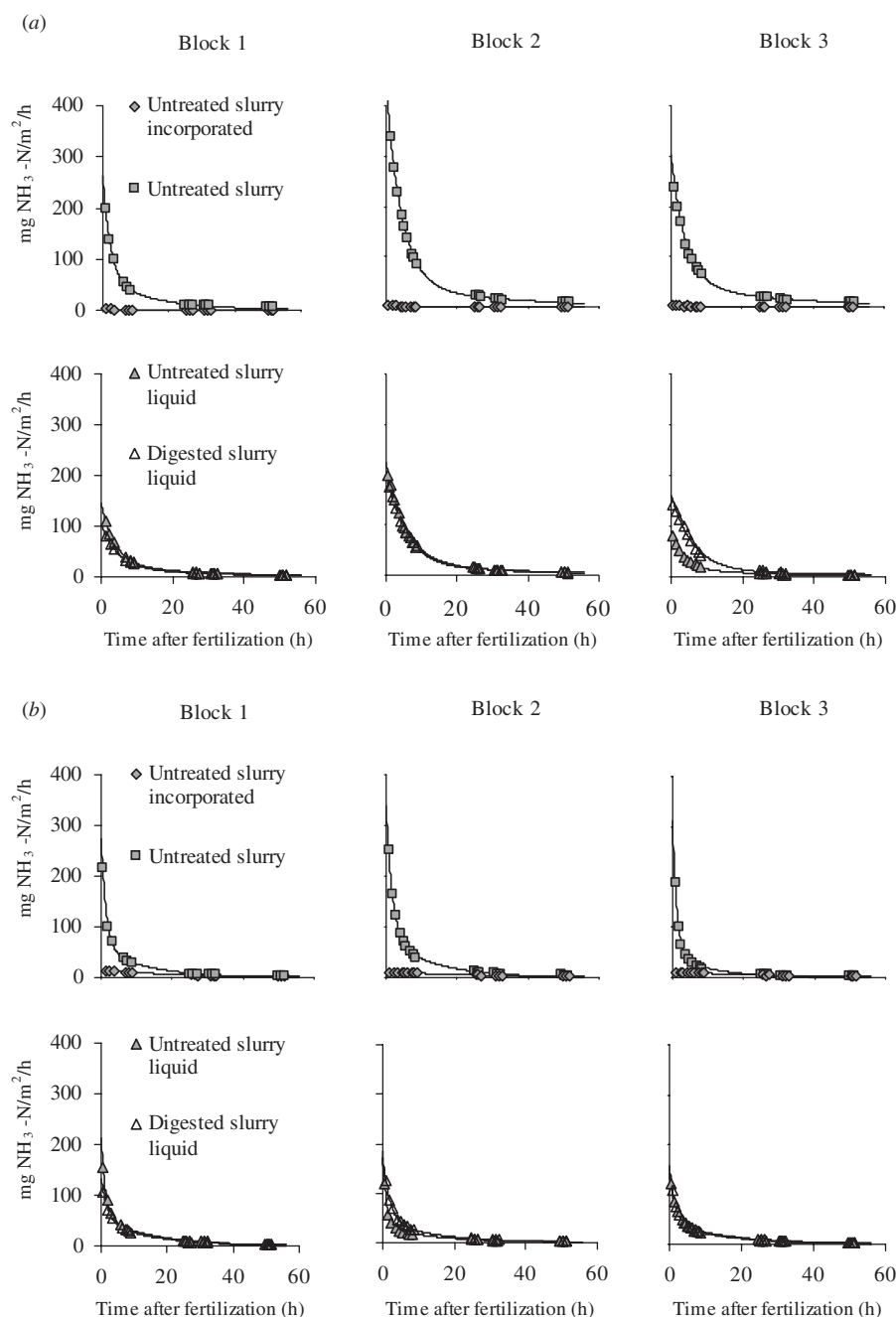


Fig. 2. $\text{NH}_3\text{-N}$ emission rates measured with the dynamic chambers combined with photoacoustic infrared gas analyser for the different fertilizer treatments in (a) silty-loam and (b) loam soils. Continuous lines represent the Michaelis–Menten fitting equation.

reported by Sommer & Ersbøll (1994) for field application of pig slurry on harrowed sandy-loam soil. Moreover, the values of total N losses expressed as a percent of TAN are within the range reported by Misselbrook *et al.* (2005) for field application of pig slurry with low DM content. The emission inventory guidebook (Hutchings *et al.* 2009) indicated an emission factor for spread swine slurry of 0.40 of TAN

within a range of 0.20–0.80; Reidy *et al.* (2008) reported factors between 0.25 and 0.68.

Total N losses/TAN values were 0.13 ± 0.064 for the liquid fraction of pig slurry and 0.16 ± 0.064 for the liquid fraction of digested pig slurry, which suggests that NH_3 emission from land application can be reduced when swine slurry is treated. Generally, it is assumed that a strong, positive relationship between

Table 3. Estimated values of the parameters of the Michaelis–Menten equation ($dAE/dt = N_{max} (k_m / (t + k_m)^2)$) and the results of ANOVA for the surface-applied manure treatments (D.F. of residual = 10)

	Total N losses (N_{max})	k_m	Initial emission rate (N_{max}/k_m)	N_{max}/TAN^*
	mg NH ₃ -N/m ²	h	mg NH ₃ -N/m ²	mg/mg
Manure effect				
Untreated slurry	1683	4.9	334	0.26
Untreated slurry liquid	989	6.1	160	0.13
Digested slurry liquid	1212	7.8	153	0.16
S.E.M.†	200.4	0.53	19.4	0.029
<i>P</i>	ns	0.010	<0.001	0.027
Soil effect				
Silty-loam	1074	20	139	0.15
Loam	750	22	126	0.12
S.E.M.	163.6	0.4	15.9	0.024
<i>P</i>	0.004	<0.001	ns	0.004
Manure × Soil				
S.E.M.	188.5	0.4	0.2	0.03
<i>P</i>	ns	ns	0.031	ns
Block				
<i>P</i>	ns	ns	ns	ns

* TAN, total ammoniacal nitrogen.

† S.E.M., standard error of the mean.

ns, not significant.

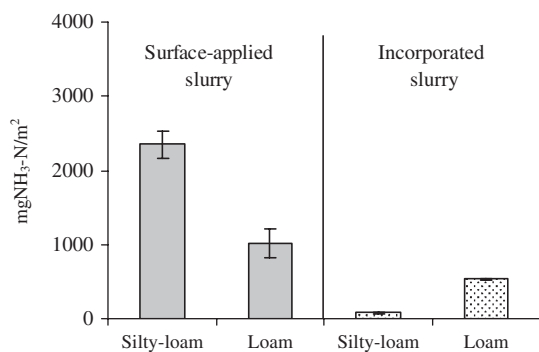


Fig. 3. Total NH₃-N losses from untreated slurry surface-applied and incorporated in the two soils utilized in the experiment. Error bars represent the S.E.M.

slurry-DM content and volatilization exists (Søgaard *et al.* 2002; Sommer *et al.* 2003; Misselbrook *et al.* 2005) due to the rapid infiltration of diluted slurries into the soil. Solid/liquid separation of both untreated and digested slurry reduces the DM content of the liquid fraction, thereby causing a NH₃ emission reduction after application. The intermediate value of emission for the liquid fraction of digested slurry was probably due to the higher pH of this treated manure that counterbalanced the increased infiltration rate. Previous field studies on NH₃ emissions from land application of anaerobic digested slurries have yielded

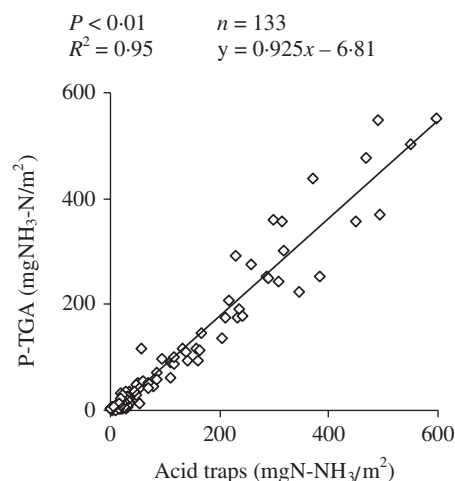


Fig. 4. Linear regression between NH₃ emission estimated by dynamic chambers using acid traps (x) and photoacoustic infrared gas analyser (P-TGA; y) methods.

contrasting results. Rubáek *et al.* (1996) reported lower emissions with digested rather than with untreated cattle slurry after ryegrass surface application. While not significant, Pain *et al.* (1990) found a tendency of digested pig slurry to reduce NH₃ emissions compared with undigested slurry on grassland. Specifically, the authors reported a total N loss/TAN ratio of 0.21 and 0.13 for undigested and digested slurry, respectively,

48 h after land application. In contrast, others (Wulf *et al.* 2002; Chantigny *et al.* 2004; Amon *et al.* 2006) have reported NH₃ emission values in a similar range for different digested slurries as opposed to untreated slurries. Chantigny *et al.* (2007) concluded that the discrepancy among studies might be explained by the higher pH of anaerobic digested slurry which could offset the benefit of improved infiltration.

Loam soil showed a lower total N loss than silty-loam soil for surface-applied slurries. This was probably due to its higher sand content that increased the slurry infiltration rate. Whatever process lay behind this difference, it was able to overcome the effect of the higher pH and lower cation-exchange capacity of the loam *v.* that of the silty-loam soil. However, in the instance of the incorporated untreated slurry, a higher volatilization was recorded in loam than in silty-loam soil, which might have been caused by the different pH of the two soils. These results underscored the importance of considering soil characteristics and different soil types when evaluating NH₃ emissions from fertilizers.

Many cost-effective abatement techniques have been proposed to reduce NH₃ volatilization. Broadcast application and immediate incorporation by plough reduced emissions by 80–90% on arable land (Hutchings *et al.* 2009). The average value found in the present experiment (82%) is within the expected range, even though the amount of NH₃ emitted was estimated in laboratory and the incorporation was performed manually. Moreover, the low values of k_m estimated for surface-applied manure treatments showed that half of the NH₃ is emitted within 6 h after manure spreading; this result points to the importance of implementing regulations on immediate incorporation of manure.

The Michaelis–Menten equation described the pattern of NH₃–N emission rates after manure application well. Several authors (Sommer & Ersbøll 1994; Søgaard *et al.* 2002; Misselbrook *et al.* 2005) have used this equation to model accumulated NH₃ losses from field-applied manure, which relates equation parameters to measured variables, such as soil properties, manure properties and environmental conditions. In contrast, Sommer *et al.* (2006) found that the Michaelis–Menten equation could not be fitted to NH₃ emissions from slurry measured in dynamic chambers, and concluded that this model was not suitable for laboratory result interpretation. The present experiment gives an operational outcome for the analysis of potential NH₃–N emissions under constant laboratory

conditions and demonstrates the possibility of fitting the results to the Michaelis–Menten equation if NH₃ emission rates are directly measured.

Measurement system evaluation

The present investigations highlighted several benefits associated with the P-TGA method to measure NH₃ volatilization. First, the experiment showed that the combination of a photoacoustic infrared gas analyser with dynamic chambers could make reliable assessments of potential NH₃ emissions when compared with the more frequently used acid traps. Second and distinct from the latter, direct NH₃ concentration measurement with a P-TGA allows temporal resolution to improve the assessment of NH₃ emission from manures. Specifically, the measurement system allowed the production of high-quality analyses of good fit, which permitted a good evaluation of the process dynamics. Third, the P-TGA system is less time-consuming both during dynamic chamber operations and for sample analyses compared with the acid-traps method. Although there is the initial cost of the instrument and scheduled maintenance (once or twice a year), it allows for a drastic reduction in the time and costs of NH₄⁺ determination. Moreover, the measurement time schedule can be easily tested and modified to better match the NH₃ volatilization dynamics, so that NH₃ emissions can be determined for a wide range of fertilizer and soil types.

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