

Application of Passive Sampler for Ammonia Gas in Soil

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Abstract The emissions of ammonia (NH_3) from soil have accelerated rapidly and have affected both vegetation and the atmosphere. It is thus necessary to investigate not only the amounts of NH_3 gas released from the soil surface but also the dynamics of NH_3 gas in the soil. Active sampling and diffusive sampling have been adopted to measure the components of soil air. However, gas aspiration for active sampling inevitably collects a wide range of soil gases. We examined the application of passive sampling to NH_3 gas measurements in soil and compared the outcome to active sampling results. In laboratory experiments, the performance of the present passive sampler in moist soil was investigated. When immersed in solution, the passive sampler collected gas released from the solution, depending on the vapor pressure of the volatile substance. In laboratory experiments measuring NH_3 gas in soil, there were no significant differences among the values measured by passive sampler at each measurement point. Thus, we concluded that the passive sampler can accurately measure NH_3 gas in soil. In field experiments, the average NH_3 gas concentrations were 43 ppb in urea-added soil and 1 ppb in control soil. The relative standard deviation of NH_3 concentrations in urea-added soil was large. This result

is expected because soil characteristics can change under the influence of ambient environmental factors such as wind, rain, and temperature. In other words, the spatial differences in NH_3 emissions were reflected in the passive sampler measurements.

Keywords Passive sampler · Ammonia gas · Soil air · Ammonia · Conversion coefficient · Filter pack

1 Introduction

Ammonia (NH_3) originates mainly from agricultural activities and animal feedlot operations (Todd et al. 2008; Beusen et al. 2008). NH_3 emissions to the atmosphere have accelerated since the middle of the twentieth century due to agricultural development (Ni 1999; Li et al. 2016). It was estimated that global atmospheric emissions of NH_3 increased from 20.6 T g N year⁻¹ in 1860 to 58.2 T g N year⁻¹ in 1993; by 2050, it was considered to be 118 T g N year⁻¹ (Galloway et al. 2004). In the USA, NH_3 emissions were increased by 11% from 1990 to 2010 due to the growth of livestock activities, agriculture, and application of nitrogen oxide control technologies using NH_3 -rich material as reducing agent (Xing et al. 2013). In China, NH_3 emissions have doubled from 1980 to 2010, indicating the expansion of agricultural production along with the doubled of nitrogen fertilizer usage and the number of livestock (Liu et al. 2013). Increase of NH_3 emission has become important in agricultural production and an air pollution problem.

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Excess amounts of NH_3 can be toxic to vegetation, cause soil acidification, and inhibit the growth of plants (De Vries 1988; Wilson and Skeffington 1994). The acute exposure of NH_3 gas to broad-leaved plants resulted in death of leaf tissue between intravenous (Krupa 2003). In addition, an increased ammonium (NH_4^+) concentration caused a decrease in hairy root formation (Bensaddek et al. 2001). In the atmosphere, NH_3 is the most abundant alkaline gas and is important as a neutralizing agent for acidic gases. It forms particles by reacting with sulfuric acid, nitric acid, and hydrochloric acid (Baek et al. 2004). Agriculture as the main source of NH_3 has significant impact on $\text{PM}_{2.5}$, which is the primary source on mortality associated with outdoor air pollution in many countries (Lelieveld et al. 2015). The observed increase in atmospheric NH_3 concentrations is strongly related to human health and changes in atmospheric composition. Therefore, the dynamics of NH_3 are important for agricultural production, ecosystem maintenance, and atmospheric environment.

Many researchers have measured the amounts of NH_3 released from the soil surface (Yamulki et al. 1996; Sommer et al. 2004; Pacholski et al. 2006; Viguria et al. 2015). Although the NH_3 release involved in the production and diffusion of NH_3 in soil (Ni 1999), few studies have examined NH_3 gas in soil. Thus, it is necessary to investigate not only the amount of NH_3 released from the soil surface but also the amount of NH_3 gas produced in the soil to understand the dynamics of NH_3 . Active sampling and diffusive sampling have been adopted to measure soil air composition. Active sampling using a pump or syringe is a widely accepted method for soil air observations (Hamada and Tanaka 2001; Wu et al. 2010; Moore et al. 2011). However, gas aspiration by active sampling disturbs the air in the soil, influencing the pressure in the soil. A wide range of soil air samples is also collected when the active method is used. As a consequence, active sampling cannot measure soil air under real environmental conditions.

The diffusive method is based on mass transport due to gas concentration gradients (Kot-Wasik et al. 2007). This method is less susceptible to changes in ambient and soil conditions and does not disturb the soil air because air is collected without suctioning gas (Hoekstra et al. 2001). Two types of diffusive methods may be employed. In the first method, a sampler is placed in the soil. Gas diffuses into the sampler and is collected for analysis (Wang et al. 2013; Yu et al. 2017).

When using this method, the target gas should be present at a high concentration or the gas concentration should be constant over time. However, this method is not suitable for measuring NH_3 gas in soil: the amount of NH_3 gas released depends greatly on the soil environment, and NH_3 concentrations can increase or decrease rapidly (Ni 1999). In the second diffusive method, the collection medium used to capture the target compound from the air is set inside the sampler (passive method). Passive samplers have been used for atmospheric NH_3 gas measurements and can collect low-concentration compounds by prolonged exposure (Kirchner et al. 2005; Drewer et al. 2015). In addition, no power supply is required, which enables measurements at multiple points.

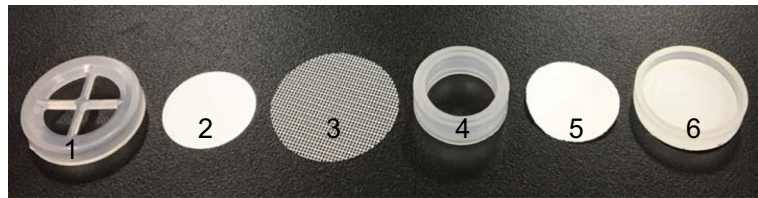
In this study, we examined the application of passive sampling for NH_3 gas in soil. Although passive sampling techniques have been used to monitor air and water quality for many years, their application to solid matrices has a relatively short history. The concentrations of soil gases are dependent on many factors (e.g., soil particle size, mineralogy, moisture content, temperature), leading to difficulties in converting the amount of analyte collected by the sampler to its concentration in the sampled air. As a result, most passive sampling applications are restricted to qualitative or screening purposes only (Seethapathy et al. 2008). We determined the concentration of NH_3 gas in soils using the same method as that applied for passive sampler measurements of atmospheric gas.

2 Materials and Methods

2.1 Passive Sampler

Figure 1 shows the passive sampler for NH_3 gas used in this study (Warashina et al. 2001). A membrane filter (PTFE, T100A025A, 25 mm diameter, ADVANTEC, Japan) was placed on the inlet to prevent the entry of particles and water droplets. A mesh filter (polyester mesh sheet, 28 mesh, ADVANTEC, Japan) was placed under the membrane filter to prevent the intrusion of soil into the sampler when placing and retrieving the sampler in the soil. Collection filters (Cellulose filter, No. 51A, 26 mm diameter, ADVANTEC, Japan) were washed with ultra-pure water (Millipore SAS, resistivity $\geq 18.2 \text{ M}\Omega \text{ cm}$, 67120 Molsheim, France) at least three times and dried. The collection filters were impregnated with 1% glycerin and

Fig. 1 Passive diffusion sampler for NH₃ gas. 1, cap; 2, membrane filter; 3, mesh filter; 4, spacer; 5, collection filter; 6, bottom case



2% phosphoric acid in 50/50 methanol/water and dried for 1 h at room temperature in the collection container (6 L), which has an inflow that allows free NH₃ gas to pass through a phosphoric acid-impregnated filter. After sampling, the collection filter was extracted with 10 mL ultra-pure water. The extract was analyzed by ion chromatography (IC7000, Yokogawa, Japan).

2.2 Determination of the NH₃ Concentration by Fick's Law

The principle of passive sampling is based upon mass transport, described by Fick's first law of diffusion (Kot-Wasik et al. 2007). The ambient NH₃ concentration, C' (ng cm⁻³), measured by the passive sampler is expressed by Eq. (1).

$$C' = \frac{M \cdot L}{D \cdot A \cdot t} \quad (1)$$

where D is the diffusion coefficient (cm² h⁻¹), A is the cross section of the diffusion zone (cm²), L is the length of the diffusion zone (cm), M is the amount collected by the passive sampler (ng), and t is the sampling time (h).

2.3 Determination of the NH₃ Concentration by Conversion Coefficient

The ambient concentration can be obtained by Fick's first law. However, the amount of analyte collected by the passive sampler depends on the sampler's shape and properties, and some researchers have used a conversion coefficient for each passive sampler and substance to calculate the ambient concentration (Lan et al. 2004). The concentration of NH₃ measured by a passive sampler is expressed by Eq. (2).

$$C' = \frac{K' \cdot M}{A \cdot t} \quad (2)$$

where K' is the conversion coefficient (ng cm⁻³ h cm² ng⁻¹) of the passive sampler. The conversion coefficient

was estimated from the amount of NH₃ collected by the passive sampler and the average concentration of NH₃ measured in the same exposure period by active sampling. In the present study, we used a denuder method (242 mm length, URG, USA) as the active sampler, operated in parallel with the passive sampler, which was operated as described above; therefore, it is possible to ignore the cross section of the diffusion zone. The NH₃ concentration in the air is expressed in parts per billion by volume. Therefore, we simplified Eq. (2) to Eq. (3).

$$C = \frac{K \cdot M}{t} \quad (3)$$

where K is the conversion coefficient (ppbv h ng⁻¹) and C is the NH₃ concentration in ppbv.

NH₃ gas volatilized from ammonia solution was used as the air applied to the samplers. Five hundred milliliters of ammonium chloride solution (1, 2, 3, or 6 mM), adjusted to pH 10 with sodium hydroxide, was transferred to a 42-L container. The container has an air inlet with a filter impregnated with phosphoric acid to prevent the NH₃ from influencing the ambient inflow air. Four passive samplers in the 6 L container were exposed to NH₃ gas (1 L min⁻¹) for 3 to 16 days using a mass flow controller (Kofloc, model 3660, Japan) that had been calibrated with a soap film flow meter and an air pump with denuder monitoring. All measurements were conducted at a temperature of 25 ± 2 °C.

2.4 Gas Collection in Aqueous Ammonia Solution

Because soil usually has a high moisture content, unlike the atmosphere, it was necessary to confirm how soil moisture affects the collection of NH₃ gas by passive sampling. Additionally, in soil, heavy rain may permeate inside the sampler. The performance of the present passive sampler in water was investigated. Passive samplers were immersed into a 1 mM ammonium chloride

solution, adjusted to pH 10 with sodium hydroxide, at 2 °C; samplers were also placed above the solution.

2.5 Measurement of NH₃ Gas in Soil

Laboratory measurements of NH₃ gas in soil were carried out using a soil modified to release NH₃ gas. A total of 5 kg Akadama soil mixed with 500 mL of 300 mM ammonium chloride solution and sodium hydroxide solution in a 12.5-L container was used as the laboratory soil sample. An air inlet with a filter impregnated with phosphoric acid was placed at the top of the container. Eight passive samplers were placed at 5 and 10 cm depth below the soil surface (expressed as -5 and -10 cm, respectively), and the collection surface was oriented sideways. Four passive samplers were set at 10 cm above the soil surface on the container wall. Additionally, eight passive samplers were placed on the soil surface with the collection surface facing the soil. The measurement period was 6 h, and the soil temperature was 25.0 ± 0.2 °C at -10 cm.

In addition, NH₃ gas in the soil was measured using the filter pack method. For filter pack measurements, a membrane filter (PTFE, T100A047A, 47 mm diameter, ADVANTEC, Japan) was used in the first stage, and collection filters (No. 51A, 47 mm diameter, ADVANTEC, Japan) impregnated with the same reagent used in the passive sampler collection filters were used in the second to fourth stages. The soil air was drawn at -10 cm at a flow rate of 100 mL min^{-1} through a Teflon tube (4 mm inner diameter) by air pump for 6 h.

Field experiments were conducted at logging sites (2×2 m) of orange tree over the course of 7 days, from 2 to 9 August, 2017, in Osaka prefecture, Japan ($34^\circ 51' \text{ N}$, $135^\circ 56' \text{ E}$). The ambient temperature was 30 ± 3 °C at 1.5 m above the soil surface, and the soil temperature was 28 ± 1 °C at -10 cm during the measurement period.

The sampling site was plowed to a depth of 20 cm before the experiment. Twenty passive samplers were placed at intervals of 30 cm at -10 cm in the soil, where solid urea fertilizer (RAKUYO, Japan) was added (10 g m^{-2}). The amount of fertilizer application in this experiment is about three times of the recommended amount by fertilizer sales company to make the difference by adding fertilizer considerable. NH₃ gas was measured both in control soil without added urea and in urea-amended soil. Three passive samplers were placed at 150 cm above the soil under a rain shelter to

measure NH₃ gas in the atmosphere. The passive samplers were pushed directly into the soil without additional digging.

2.6 Soil Sample Collection and Analysis

The chemical characterization of the soils used Akadama and field soil is shown in Table 1. In the laboratory experiment, soil samples were collected before adding ammonium chloride solution. In the field experiment, four soil samples (10 cm depth) were collected before adding urea fertilizer and after measurement of soil air. Soil samples (10 g dry equivalent) were collected from each soil and extracted with ultra-pure water by shaking for 1 h (1:5 soil/water ratio). The extracts were filtered through a filter (Millex-LG, 0.20 μm, Merck KGaA, Germany) and analyzed by ion chromatography (cation IC: IC7000, Yokogawa, Japan, and anion IC: IC7000, Yokogawa, Japan) for NH₄⁺, NO₂⁻ and NO₃⁻. To measure soil pH, the soils were extracted using the same method as for ion analysis (1:2.5 soil/water ratio), and the extract was measured by a pH meter (F-52, HORIBA, Japan).

3 Results and Discussion

3.1 Determination of NH₃ Concentration

Figure 2 shows the results of NH₃ ambient air measurements by the passive sampler and the denuder method. A strong linear relationship was observed between the two methods ($y = 0.655 \pm 0.0563x$, $r^2 = 0.9296$). A conversion coefficient K ($1.53 \pm 0.13 \text{ ppbv h ng}^{-1}$) was obtained from the slope of the straight line. The dotted line (Fig. 2; $y = 1.347x$, $K = 0.74 \text{ ppbv h ng}^{-1}$) is the theoretical line according to Fick's law (Eq. (1)) and the diffusion coefficient of NH₃ in air (Massman 1998). Feigley and Lee (1987) reported that the theoretical value obtained by (the amount of analyte collected by the passive sampler) / (the ambient analyte concentration) is about 1.5 times higher than the measured value. The similar trend was observed in our experiment. Nevertheless, it is still unknown exactly which factors affect the results. Therefore, it is necessary to calculate a conversion coefficient for each sampling event at one to several points.

Table 1 Water-soluble ion concentrations in soil ($\mu\text{g g}^{-1}$ dry soil)^a and soil pH (H_2O)^b ($n = 4$) (Average \pm 1SD)

Soil	Sampling time	NH_4^+	NO_2^-	NO_3^-	pH
Akadama soil	Before adding solution	2 ± 0	N.D.	10 ± 0	5.5 ± 0.0
Urea-added soil	Before fertilization	8 ± 9	3 ± 1	129 ± 93	6.6 ± 0.2
	After measurement	77 ± 37	98 ± 46	227 ± 31	6.6 ± 0.2
Control soil	Before fertilization	8 ± 2	5 ± 2	156 ± 11	5.7 ± 0.0
	After measurement	7 ± 2	4 ± 2	38 ± 7	6.0 ± 0.1

^a The soil:water extraction ratio is 1:5 (dry equivalent). ^b The soil:water extraction ratio is 1:2.5 (dry equivalent)

N.D.: not detected

3.2 Effect of Moisture on NH_3 Collection by the Passive Sampler

It was necessary to confirm that no liquid could enter the passive sampler. The passive samplers were immersed in solution containing NH_4OH . The amount of NH_3 gas collected increased with exposure time for both the passive sampler in solution and the sampler in the headspace (Fig. 3). It was concluded that no solution entered the sampler. A strong linear relation was obtained between the amount collected and the exposure time at both positions (Solution; $r^2 = 1$, Headspace; $r^2 = 0.9986$). It was confirmed that for passive sampler, there was a proportional relationship between exposure time and collection amounts up to 250 μg .

Applying a t test to the collection efficiencies for the passive samplers in solution and in the headspace, no significant difference at the 95% confidence level was observed between the measurements. This means that only gas volatilizing from the solution surface is

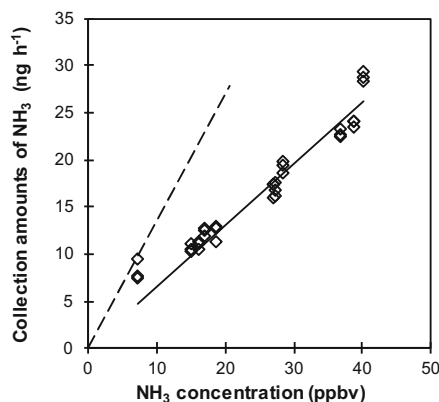


Fig. 2 Relationship between NH_3 concentration measured by denuder (ppbv) and the amount of NH_3 collected by passive sampler (ng h^{-1}). The black line shows the line of best fit ($y = 0.655 \pm 0.0563 x$, $r^2 = 0.9296$). The dotted line shows the theoretical line obtained by Fick's law ($y = 1.347 x$)

collected when the passive sampler is immersed in the solution. In other words, depending on the vapor pressure of the volatile substance, gas released from the solution can be collected by the passive sampler even if a liquid is present on the collection filter surface.

3.3 Measurement of NH_3 Gas in Soil (Laboratory Experiment)

Figure 4 shows the measurement of NH_3 gas in soil. The average measurements and the errors ($P = 0.05$) of the passive sampler were 1410 ± 330 , 1360 ± 240 , 950 ± 330 , and 640 ± 30 ppbv at -10 , -5 , 0 , and 10 cm height, respectively. A t test was applied to the measurements at -10 and -5 cm, and there was no significant difference at the 95% confidence level. In addition, the difference in NH_3 concentration for each passive sampler at the same depth was small (relative standard deviation = 0.10 to 0.14). Thus, we concluded that the accuracy of the passive sampler in the soil was suitably high.

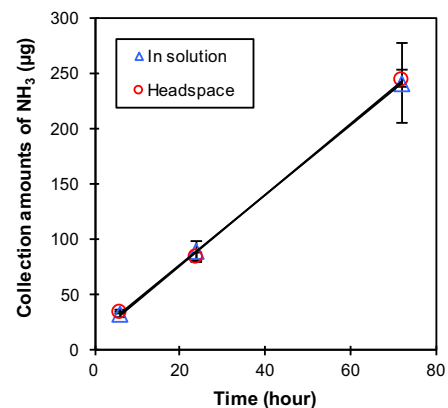


Fig. 3 The amount of NH_3 gas collected increased with exposure time in the passive samplers both in solution and in headspace. $n = 3$. The errors are 95% confidence intervals

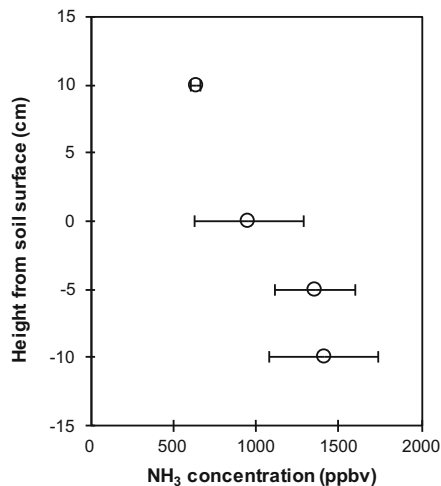


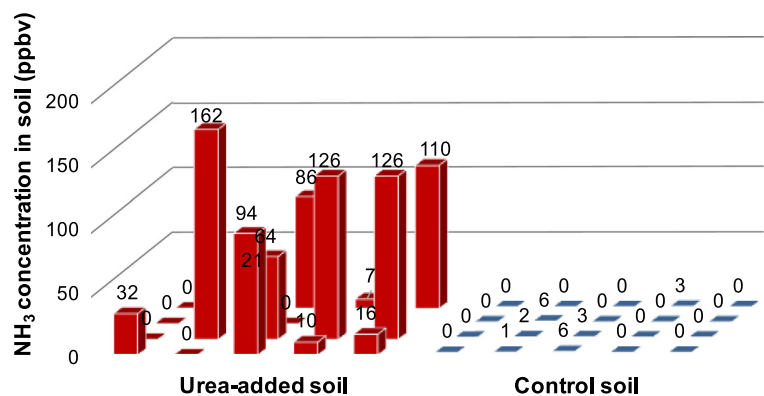
Fig. 4 NH₃ gas concentration at each height. $n = 8$ at -10 , -5 , and 0 cm. $n = 4$ at 10 cm. The errors are 95% confidence intervals

NH₃ as measured by filter pack (370 ppbv) was significantly lower than that measured by passive sampler in the soil (1410 ppbv). This observation can be explained by the collection of atmospheric gas during active sampling and by the NH₃ that could have adsorbed to water droplets formed from water vapor in the Teflon tube (Schmohl et al. 2001). Passive sampling seems to compensate for most of the disadvantages of active sampling in soil air.

3.4 Field Experiment

Figure 5 shows the NH₃ gas concentration at each measurement location in the urea-added soil and control soil. Extreme deviations from the average were omitted by Grubbs' test for outliers. The

Fig. 5 NH₃ gas concentrations at each measurement location in urea-added soil and control soil



average concentrations of NH₃ gas measured by the passive sampler and their errors ($P = 0.05$) were 47.0 ± 28.5 ppbv for urea-added soil, 1.1 ± 0.9 ppbv for control soil, and 8.9 ± 2.3 ppbv in the atmosphere. Concentrations of NH₄⁺, NO₂⁻, and NO₃⁻ increased in the urea-added soil after sampling compared with those before sampling. However, NH₄⁺ and NO₂⁻ did not change much in the control soil, and the NO₃⁻ content decreased (Table 1). The urea was decomposed by enzymes (Singh et al. 2013). From these results, we concluded that NH₃ gas generated in the urea-added soil was collected by the passive sampler.

Singh et al. (2013) reported that the application of urea (10 g N m^{-2}) or urine (47.6 g N m^{-2}) to soil core significantly escalated NH₃ emission and soil pH within 1–2 days; then, the emission of NH₃ and soil pH decreased after 1 week. In the field's experiment as well, the soil pH was not changed in urea-added soil before and after the measurement (Table 1), so it was assumed that the release of NH₃ by adding urea had almost ceased after 1 week. However, further research will be needed to yield any findings about NH₃ gas generation and diffusion in the soil by subdividing the measurement period with various types of fertilizers.

The relative standard deviation of NH₃ concentrations in the urea-added soil was large (1.18). This result is probably because soil characteristics can change under the influence of ambient environment factors, such as wind, rain, and temperature (Bristow et al. 1986), and furthermore that urea was not uniformly distributed in the soil because solid urea was used. Therefore, the generation of NH₃ gas was scattered. We concluded that

the site-to-site differences in NH₃ emissions were reflected by passive sampling measurements.

4 Conclusion

In conclusion, passive samplers were applied to measure NH₃ gas in soil. The passive sampler used in the present study can collect gas released from solution depending on the vapor pressure of the volatile substance. The results showed that it is possible to use this passive sampler in moist soil. The passive sampler can also accurately measure the NH₃ gas present at the soil collection surface. The present method improved our understanding of the dynamics of NH₃ both in the soil and released from the soil surface. In this experiment, we used a slightly larger passive sampler, and we showed here the possibility to use passive samplers to measure more accurate NH₃ concentrations in the soil. Using a smaller passive sampler, it would be possible to measure NH₃ gas without disturbing the soil environment.

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