

INFLUENCE OF WAITING TIME AFTER INSERTION OF BASE CHAMBER INTO SOIL ON PRODUCED GREENHOUSE GAS FLUXES

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The soil chamber technique is most commonly used for measuring gas exchange between soil surfaces and the atmosphere, to understand regulatory processes relevant to determine the greenhouse gas (GHG) emissions from soils and to improve the emissions inventory of agricultural systems. The chambers are inserted into the soil to avoid the lateral diffusion of the gases. However, soil disturbance caused by chamber insertion causes in degassing and can result in erroneous flux data from measurements made immediately following chamber insertion. Here we assess the effect of soil disturbance associated with the insertion of the chambers on nitrous oxide (N₂O) and methane (CH₄) fluxes with and without N fertilization, from a New Zealand pasture soil. We collected gas samples from the chambers at 0, 2, 4, 24, 96 and 168 h after chambers insertion. Our results show elevated levels of N₂O inside the chambers (1.7 fold those of atmospheric concentrations outside the chambers) and greater fluxes within the first 4 h of installation compared to the values observed at 24 h and later; and negative CH₄ flux in the same time period, but near to zero after 24 h. Our results suggest that for accurate measurement of gas fluxes, measurements should be taken after 24 h of chambers installation to avoid the degassing effect.

Key words: Nitrous oxide, methane, agricultural soil, soil biological processes, gases monitoring.

In situ measurements of greenhouse gas (GHG) emissions from soils are crucial to improve the GHG inventories of the countries that are required to develop their independent emission factors (EFs). Diverse tools and methods are used to determine carbon dioxide (CO₂), nitrous oxide (N₂O), and methane (CH₄) fluxes from a specific site or farm-scale. These include the use of default EFs recommended by the Intergovernmental Panel on Climate Change (IPCC, 2007), measurements using chambers and micrometeorological approaches and modeling emissions (Saggarr *et al.*, 2009). The soil chambers are the most commonly used techniques for measuring gas exchange between soil surfaces and the atmosphere in diverse ecosystems and agricultural systems in countries all over the world (Rochette and Eriksen-Hamel, 2008). This approach is essential to capture the highly variable - in space and time - emissions resulting from uneven excretal-N inputs and the episodic nature of the emissions, and to understand regulatory processes (Saggarr *et al.*, 2009). Chambers are containers (cylinders or boxes) randomly inserted into the soil to form an air-

tight soil enclosure. The insertion of the chamber into the soil avoids the leakage or gas contamination by lateral diffusion. Gas fluxes are monitored after temporarily sealing the chambers with a gas-tight seal using self-locking lids and greased O-ring. Chamber technique is based on an increase in gas concentration within the enclosed headspace and the concentration in the headspace is precisely determined by gas chromatography (Hedley *et al.*, 2006). Gas fluxes are calculated using a linear or non linear regression with the increase of the gas concentration with time. A minimum of three discrete air samples are required to check the linear or non-linear increase in gas concentration within the chamber headspace (Rochette and Eriksen-Hamel, 2008). This technique contributes to the understanding of the magnitude and spatiotemporal variability of GHG fluxes (Saggarr *et al.*, 2008).

Despite this technique being used extensively little research has been conducted on the effect of the soil disturbance caused by the insertion of the chamber, except that reported by Matthias *et al.* (1980). They indicated the soil disturbance resulting from chamber insertion caused an increase in the N₂O flux and this increase in flux was indirectly proportional to the diameter of the chamber. Also a minimum set of criteria established for chamber technique by Rochette and Eriksen-Hamel (2008), from the review of chamber studies conducted in different parts of the world, does not include the wait time in gas collecting after the insertion of the chamber base. Furthermore, among other factors, the gaseous

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concentrations in the soil and their fluxes can be strongly influenced by N inputs (Saggar *et al.*, 2004; Gregorich *et al.*, 2005; Jones *et al.*, 2005; Chu *et al.*, 2007). It is necessary to include the N fertilization influence on the GHG fluxes caused by soil disturbance during the insertion of the chamber. The objectives of this study were to determine: i) the effect of the influence of chamber insertion and the equilibration time for collection of gas samples following the disturbance, and ii) their effect on N₂O and CH₄ emission from a pasture soil with and without N fertilizer input.

MATERIALS AND METHODS

Site

The experiment was conducted under the temperate climate in Manawatu-Wanganui Region, in the North Island of New Zealand. The soil at this site is an Argillic-fragric Perch-gley Pallic, Tokomaru silt loam (Hewitt, 1992) (Dairy 4 Farm), Turitea Campus of Massey University, Palmerston North (40°21', 175°39'). The average rainfall is 969 mm (from 1970 to 2005) and the mean annual air temperature is 12.8 °C. The productive system in this soil is a commercial dairy farm of legume-based pastures.

Experimental method

Two treatments with 12 replicates represented two levels of N (control and 50 kg N ha⁻¹ applied as potassium nitrate). Twenty-four plots (1 m × 1 m) were established. Calculated amount of KNO₃ was dissolved in water and applied in N treatments.

Static chambers (Ø 250 mm, 300 mm high) (Saggar *et al.*, 2004), were inserted into the soil. A slot was cut into the soil with a sharp knife along the outer diameter of the chamber and the chamber was then pushed into the soil to a depth of 10 cm by hammering.

To evaluate the time of the influence of soil disturbance on GHG fluxes following chamber installation gas samples were collected immediately and after 2, 4, 24, 96, and 168 h using the methodology proposed by Hedley *et al.* (2006). Each time chambers were closed with the lid. After sealing, the headspace air was mixed by a 60 mL polypropylene syringe fitted with 3-way stopcock and then three gas samples were taken at times t_0 , t_{30} and t_{60} (30-min intervals) from the headspace of each chamber. Following the last sampling (t_{60}) the chambers were opened to allow them to equilibrate with the atmospheric concentration for the next sampling. Additionally, two ambient air samples were collected from the field at each gas sampling day and used as reference to determine the atmospheric gas concentration. The air temperature within the chamber was also measured using a digital thermometer. Once in the laboratory, 25 mL of the collected gas samples were transferred to a 12-mL- evacuated vial (Exetainer, Labco, England) that was held in over-pressure.

The gas samples were analyzed using a gas chromatograph (Shimadzu, GC 2010, Kyoto, Japan) equipped with a ⁶³Ni-electron capture detector (ECD) and a flame ionization detector (FID) with methanizer. Nitrogen (99.99% purity) was used as carrier gas and a makeup gas of 5% methane in argon for ECD. In the case of CH₄ analysis, N was used as carrier, while hydrogen (99.99% purity) and synthetic air (99.99% purity) were used for generating the combustion. A standard calibration curve was obtained using standards that covered the range of gas sample concentrations analyzed and that were run every 15 samples to check accuracy. The average rate of change in gas concentration was determined using either linear or non-linear regression, and gas-fluxes were then calculated from Equation [1] using the ideal gas law as described in Saggar *et al.* (2009):

$$F = p \frac{V}{A} \frac{\Delta c}{\Delta t} \frac{273}{(T + 273)} \quad [1]$$

where F is the gas-flux (μg m⁻² h⁻¹), p is the density of the gas (g m⁻³), V is the volume of the chamber (m³), A is the area of the chamber (m²), Δc/Δt is the average rate of change of concentration with time (mg L⁻¹ h⁻¹), and T is the temperature (°C) in the chamber.

The soil water content was measured at each time of sampling, and expressed as gravimetric water fraction relative to oven dried (at 105 °C) soil.

Statistical analysis

A complete randomized factorial design 2 (treatments) × 6 (times) was used to analyze the data. A non-parametric analysis of Kruskal Wallis (p ≤ 0.05) and its corresponding Conover post hoc test (Conover and Iman, 1981; Conover, 1999) with a 95% confidence interval (p ≤ 0.05). The Conover test allows conducting pairwise comparisons between the means of the ranks of the treatments and/or contrasts between them.

RESULTS AND DISCUSSION

The soil water content in the 0-10 cm depth in all plots during this field study was 23 ± 5%. Figures 1 and 2 show the time zero (t_0) concentrations of N₂O and CH₄ in gas samples collected immediately after closing the chambers at 0, 2, 4, 24, 96, and 168 h of inserting the chambers in the soil in both the control and N fertilized treatment plots. The concentrations of N₂O within the chambers during the initial period (0 to 4 h; 498 ± 18 μg L⁻¹) were almost twice to those measured (285 ± 18 μg L⁻¹) between 24 and 168 h of chamber insertion, while the atmospheric concentrations (outside the chambers) over the whole sampling period of this study were 292 ± 21 μg L⁻¹. These high initial chamber N₂O concentrations appear to be the result of the degassing of N₂O from the disturbed soil caused by chamber insertion into the soil.

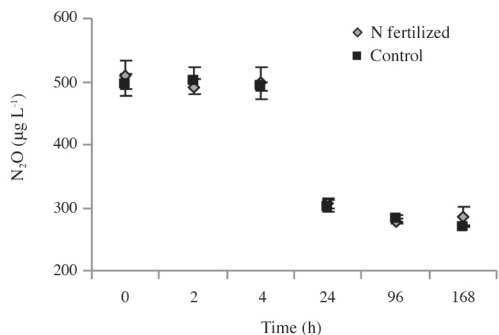


Figure 1. Initial concentration of N₂O inside the chamber at different times after the base chamber insertion in a field experiment (Conover, $p \leq 0.05$).

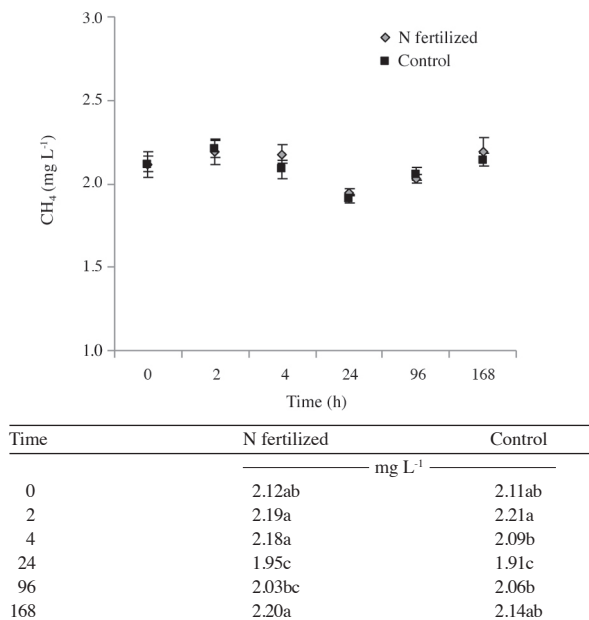
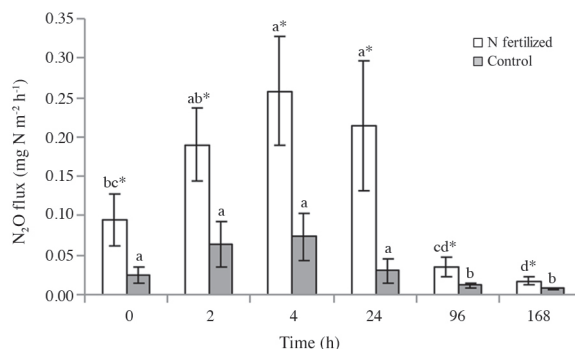


Figure 2. Initial concentration of CH₄ inside the chamber at different times after the base chamber insertion in a field experiment (Conover, $p \leq 0.05$).

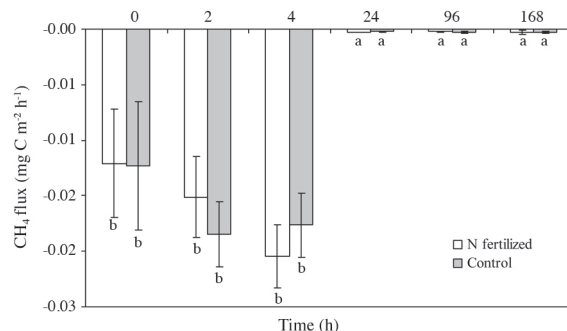
The concentration of CH₄ within the chambers at t_0 (2.10 ± 0.13 mg L⁻¹) was slightly higher than the atmospheric concentrations (outside the chambers, 1.81 ± 0.05 mg L⁻¹). The chamber concentrations measured at t_0 for the remaining period also did not differ from the atmospheric concentrations. The similarity in CH₄ concentrations between the chambers and the atmosphere seems to be due to aerobic soil conditions resulting in little CH₄ accumulation and little degassing during chamber insertion.

Fluxes of N₂O and CH₄ are shown in Figures 3 and 4. The N₂O fluxes from the control were generally low N₂O fluxes ($0-0.05$ mg N m⁻² h⁻¹). These fluxes were, however, significantly higher during the initial 0-24 h measurement period than those obtained after 24 h (0.01 ± 0.002 mg N m⁻² h⁻¹) of chamber insertion. In the N fertilized treatment the fluxes were much higher ($0-0.30$ mg N m⁻² h⁻¹) than the control. However, the pattern of fluxes between the 0-24 h (0.19 ± 0.08 mg N m⁻² h⁻¹) and after 24 h periods (0.03 ± 0.01 mg N m⁻² h⁻¹) was similar to that observed in the control. The differences in fluxes in both the treatments appear to be resulting from a relevant impact of disturbance caused by inserting the chambers. CH₄ fluxes slightly fluctuated between negative (methane consumption) and zero (neither consumption nor production) during the measurement period under these aerobic soil conditions (Figure 4). Negative CH₄ flux values were obtained only between 0 to 4 h after chamber insertion when the gas fluxes were in the range of -0.01 to -0.02 mg CH₄-C m⁻² h⁻¹ for both the control and N fertilized treatments, which could be the result of a stimulation of the methanotrophs microorganisms due to soil disturbance. After 24 h, the CH₄ flux was close to zero, with no statistical differences ($p \leq 0.05$) between the treatments. However, Saggari *et al.* (2008) indicated different levels of CH₄ consume (or negative fluxes) in different land management systems



*Statistical difference between the pair of data (N fertilized and control) at each gas sampling. Letters indicate statistical difference between each gas sampling.

Figure 3. N₂O fluxes in a pasture soil with N fertilization and a control soil (Conover, $p \leq 0.05$).



Letters show statistical differences between each gas sampling (Conover, $p \leq 0.05$).

Figure 4. CH₄ fluxes in a pasture soil with N fertilization and a control soil.

CONCLUSIONS

The results of this study show that efflux of N_2O and CH_4 from the soil is influenced by the soil disturbance caused during the insertion of chambers into the soil. The differences in N_2O concentration in gas samples collected within the first 4 h of inserting the chamber into the soil was significantly higher than both the atmospheric concentrations and those measured after 24 h of chamber installation in fertilized and not fertilizer soil. These high initial chamber concentrations also resulted in very high and erroneous flux values. These results suggest that an equilibration period of 24 h is needed before the measurements are made to avoid erroneous results.

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Influencia del tiempo de espera después de la inserción de la base de la cámara en el suelo en los flujos de gases de efecto invernadero producidos.

La técnica de la cámara es la más comúnmente usada para medir el intercambio de gases entre la superficie del suelo y la atmósfera, entender los procesos regulatorios relevantes para determinar las emisiones de gases de efecto invernadero (GHG) desde los suelos, y mejorar los inventarios de emisiones de sistemas agrícolas. Las cámaras se insertan en el suelo para evitar la difusión lateral de los gases. Sin embargo, la alteración en el suelo causada por la inserción de las cámaras causa pérdida de gases y puede resultar en datos de flujos erróneos desde las mediciones realizadas inmediatamente posteriores a la inserción de las cámaras. Determinamos el efecto de la alteración del suelo asociada con la inserción de las cámaras sobre los flujos de óxido nitroso (N_2O) y metano (CH_4) con y sin fertilización nitrogenada, desde un suelo con pradera en Nueva Zelanda. Colectamos muestras gaseosas desde las cámaras a 0, 2, 4, 24, 96 y 168 h después de la inserción de las cámaras. Nuestros resultados muestran elevados niveles de N_2O al interior de las cámaras (1,7 veces las concentraciones atmosféricas del exterior de las cámaras) y mayores flujos dentro de las primeras 4 h de instalación comparada a los valores observados a 24 h y posteriores; y flujos negativos de CH_4 en el mismo período de tiempo, pero cercanos a cero después de 24 h. Nuestros resultados sugieren que para la medida exacta de los flujos de gases, la medición debe ser tomada después de 24 h de la inserción de las cámaras para evitar el efecto de la liberación de gases.

Palabras clave: Óxido nitroso, metano, suelo agrícola, procesos biológicos del suelo, monitoreo de gases.

in New Zealand, determining a seasonal pattern of soil methane sink in pastoral soils. In other agricultural systems, negative fluxes were obtained by Gregorich *et al.* (2005), but they indicate that the CH_4 uptake from cultivated soils plays a minor role in atmospheric loading of GHG in relation to other sources/sinks. The CH_4 fluxes do not seem to be affected by fertilization practice, since similar CH_4 flux values were obtained in both treatments (N fertilized and control treatments), same as the results obtained by Metay *et al.* (2007) in an clayey Oxisol in Brazil.

The results indicate that chamber insertion causes soil disturbance resulting in immediate degassing of N_2O stored within the soil. Higher initial t_0 N_2O concentrations in the chambers than the atmospheric concentrations in the field obtained during the initial 24 h sampling period produce artificially high N_2O fluxes, obtaining in the first 24 h 5 fold higher N_2O fluxes than after 96 h after chamber insertion in non-N fertilized soils, and 7 fold higher the N_2O fluxes in N fertilized soils, supporting our hypothesis of higher efflux being the result of soil disturbance. These concentrations were almost twice the atmospheric values but declined after 24 h. These results suggest that 24 h were required for the disturbed soil to reach equilibrium in soil-atmosphere exchange of gases. Our results also show that these initial higher chamber concentrations also resulted in higher fluxes in the first 24 h after inserting the base chamber (Figure 3). Similarly, Matthias *et al.* (1980) indicate that the insertion of a metal cylinder from chambers into the soil (5 cm of depth) produce an increase 250% (in average) in the N_2O emission rate from different sites of a Webster soil.

In the N fertilized plots, the higher initial levels of N_2O flux could result from the combined effect of the soil disturbance and the nitrification/denitrification processes that occur fast after a fertilization event. These produce an increase of the N_2O flux in the first time with a decrease afterwards. In this sense, Abbasi and Adams (2000) indicate that between 60% to 100% of the added N (applied as KNO_3) disappeared from the soil mineral N pool over a 7-d period, with losses of 5-22 kg N ha^{-1} as N_2O in a compacted grassland soil.

The higher N_2O fluxes in fertilizer plots compare to control soil, indicates the relevant impact of N input on N_2O fluxes. Saggat *et al.* (2004) evaluated the N_2O flux in a grazing system in the same soil (Diary 1 Farm), obtained an annual net emission of 2.5 kg N_2O -N ha^{-1} (gas sample collection 24 h after the insertion of the chamber) in an ungrazed area, obtaining an increase to 10.4 kg N_2O -N ha^{-1} in a grazed area.

It is seen that the higher initial fluxes of N_2O and negative CH_4 fluxes caused by soil disturbance reported in this study are not representative of the normal soil-atmosphere exchange of gases and are biased by the chamber insertion. Thus these initial fluxes are erroneous and flawed. It is therefore suggested that for more accurate flux estimates the gas samples should be collected after 24-h chamber insertion.

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