PAB
Pesquisa Agropecuária Brasileira

ISSN 1678-3921

Journal homepage: www.embrapa.br/pab

For manuscript submission and journal contents, access: www.scielo.br/pab

Soil Science/ Scientific Notes

Sampling frequency to estimate cumulative nitrous oxide emissions from the soil

Abstract – The objective of this work was to assess the influence of gas sampling frequency on the cumulative emissions of nitrous oxide (N_2O) from the soil. Gas emissions were assessed over a period of two years (2014–2016), in four systems: eucalyptus forestry, crops, pasture, and native forest. The cumulative emissions of N_2O were calculated at sampling intervals of 7, 14, and 21 days. The sampling intervals did not influence the final results of cumulative N_2O emissions from the soil in the assessed systems.

Index terms: agriculture, emission factor, forestry, greenhouse effect, native forest, N_2O.

Frequência de amostragem para estimativa das emissões acumuladas de óxido nitroso do solo

Resumo – O objetivo deste trabalho foi avaliar a influência da frequência de amostragem de gases na estimativa das emissões acumuladas de óxido nitroso (N_2O) do solo. Foram avaliadas emissões de gases durante dois anos (2014–2016), em quatro sistemas: plantio de eucalipto, lavoura, pastagem e fragmento florestal. As estimativas acumuladas de N_2O foram calculadas para intervalos de 7, 14 e 21 dias. Os intervalos de amostragem não influenciaram os resultados finais de emissões acumuladas de N_2O do solo nos sistemas avaliados.

Termos para indexação: agricultura, fator de emissão, floresta plantada, efeito estufa, floresta nativa, N_2O.

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Received
October 5, 2017

Accepted
April 29, 2019

How to cite
NASCIMENTO, A.F. do; RODRIGUES, R. de A.R. Sampling frequency to estimate cumulative nitrous oxide emissions from the soil. **Pesquisa Agropecuária Brasileira**, v.54, e00211, 2019. DOI: https://doi.org/10.1590/S1678-3921.pab2019.v54.00211.

According to the guidelines for assessing greenhouse gas emissions from the soil using manual static chambers, the adopted sampling frequency depends on the system being evaluated. For natural or agricultural systems, as well as for long-term experiments that do not aim to assess the influence of fertilization, irrigation, sowing, or rainfall on soil N_2O emissions, it is recommended that gas sampling intervals range from 7 to 21 days (Parkin, 2008; Parkin & Venterea, 2010; Klein & Harvey, 2015; Butterbach-Bahl et al., 2016).

For soils with fewer perturbations or when low fluxes are expected, the sampling frequency could be lower, at least every 2 or 3 weeks (Parkin & Venterea, 2010; Butterbach-Bahl et al., 2016); however, this increases error when measuring the cumulative N_2O flux (Parkin, 2008). According to Rochette et al. (2015), gas sampling should be performed twice every week when gas peak fluxes are expected and once during the period of low fluxes. Parkin & Venterea (2010) recommend that sampling should be carried out daily after events that lead to higher fluxes, such as sowing, fertilization, irrigation, and rainfall, and weekly in other events during the crop cycle. For Reeves & Wang (2015), in agricultural systems, sampling should be done at least one time a week, but two times after rain events.

Increasing gas sampling frequency ensures a greater accuracy and representativeness in the estimation of gas emissions from soils (Parkin, 2008). However, an increase in the interval that does not change the final estimates of cumulative emissions can lead to a reduction in research costs: team, field, and laboratory costs. Another difficulty is related to the distance of the area to be evaluated, which makes weekly visits practically impossible due to high costs and difficult sampling logistics, hindering some research on soil gas emission.

The objective of this work was to evaluate the influence of gas sampling frequency on the cumulative emissions of N_2O from the soil.

The research was conducted at the experimental farm of Embrapa Agrossilvipastoril, located in the municipality of Sinop, in the state of Mato Grosso, Brazil (11°51'38"S, 55°36'3"W). From November 2014 to October 2016, soil N_2O emissions were assessed in four systems: 1 ha eucalyptus, 1 ha crops, 2 ha pasture, and native forest fragment. The H13 eucalyptus (*Eucalyptus urograndis*) clone was planted in November 2011, at a density of 952 plants per hectare, with a spacing between plants of 3.0×3.5 m. Since November 2011, the crop system has been cultivated with soybean [*Glycine max* (L.) Merr.] and, after its harvest, with corn (*Zea mays* L.) intercropped with 'Marandu' grass [*Urochloa brizantha* (A.Rich.) R.D.Webster (Syn. *Brachiaria brizantha*) (A.Rich.) Stapf], which works as soil cover after corn harvest. The pasture was formed in November 2011 with 'Marandu' grass. The forest fragment is close to the other systems, approximately 500 m away, and is composed of initial secondary species. The areas with eucalyptus, crops, and pasture were evaluated with six replicates, and the forest, with three. All these systems are on a Latossolo Vermelho-Amarelo distrófico típico of clayey texture (Santos et al., 2018), which corresponds to a Hapludox of clayey texture, in flat relief (Soil Survey Staff, 1999). The main attributes that characterize the 0–10-cm soil layer of the studied areas are presented in Table 1.

Soil N_2O emissions were assessed using the method of vented static chambers, which were rectangular-shaped, with a base of metal and a top of polyethylene. A three-way gas sampling faucet was attached to the center of the top of the chamber, and a tube for internal ventilation was installed on the side of the chamber (Parkin & Venterea, 2010). Gas collections were performed every 7 days, always in the morning, between 8:00 and 11:00 a.m., using a 20-cm^3 syringe. For each chamber, four gas samples were collected at 0, 20, 40, and 60 min after chamber deployment (Parkin & Venterea, 2010). In addition, at the time of gas sampling, the internal temperature of the chamber was also monitored using a digital thermometer.

Gas samples in the syringes were transferred to 20-cm^3 glass vials, which were duly sealed and vacuumed. N_2O concentrations were determined in the GC-2014 gas chromatograph (Shimadzu, Tokyo, Japan), equipped with an electron capture detector, an auto-sampler, and a column system composed of HayeSep 80/100 mesh (1/8" × 2.1 mm) series columns held at 75°C throughout the analysis. Ultrapure nitrogen was used as the entrainment gas at a flux rate of 25 mL min^{-1}, and the injector pressure was maintained at 300 kPa. The injection volume was 1 mL, and the total analysis time was 5 min. The analytical curve used for the estimates of the gas concentrations in the samples was obtained through three known concentrations of N_2O standards – 383, 808, and 2,027 nmol mol^{-1} –, purchased from White Martins (Rio de Janeiro, RJ, Brazil).

Using the analytical results, a linear model was adjusted by the relationship between the variations in N_2O concentrations within the chamber and time, i.e., 0, 20, 40, and 60 min. These data were used to calculate the N_2O flux from the soil to the atmosphere, according to the equation proposed by Hutchinson & Livingston (1993): Flux (μg N m^{-2} h^{-1}) = ($dCdt^{-1}$) × V/A × (mVm^{-1}), where $dCdt^{-1}$ is the change in gas concentration (mol L^{-1}) inside the chamber as a function of time (h), V is the volume of the chamber (L), A is the chamber area (m^2), m is the molar mass (g mol^{-1}), and Vm is the molar volume of the gas (L mol^{-1}).

Flux results were used to estimate the cumulative emissions of the gas during the evaluated period, by the Newton-Cotes (trapezoidal integration) method of numerical integration (Rochette et al., 2015). Sampling intervals of 7, 14, and 21 days were used for integration, which, together with the flux data, represent one of the factors in this calculation. The cumulative N_2O emissions were estimated for two years, i.e., 2014–2016, specifically for the dry and rainy seasons, which contributed to determine the most adequate sampling frequency for each season.

Even after their transformation, the data for two years of soil N_2O emissions did not follow a normal distribution according to the Shapiro-Wilk test; therefore, the standard error of the mean was used to compare the sampling frequency within and between systems.

The cumulative emissions of N_2O from the soil did not differ for the two experimental years, for all gas sampling intervals in all systems (Figure 1). This result may be related, in part, to the great variability of data, leading to a high standard error of the mean, common in gas emission studies (Parkin, 2008; Venterea et al., 2009; Barton et al., 2015).

The sampling interval also did not change the final estimates for cumulative N_2O obtained just for the rainy or dry period (Figure 1). This is indicative that, if greater sampling intervals of 14 or 21 days were used, the cumulative emission would not differ for forest and agricultural systems, even during the period of high soil moisture, when fluxes are higher (Kachenchart et al., 2012; Teh et al., 2017). It should be noted that these results refer only to the final estimates of gas emission and may not be useful for understanding its temporal dynamics, which would include observing the evolution of emissions over time (Rochette et al., 2015). When the goal is to determine flux dynamics rather than cumulative emissions, Barton et al. (2015) pointed out that the sampling frequency should be higher than once every week due to the high variability of soil N_2O data, especially when the intention is to evaluate the effect of agricultural practices on N_2O emissions from the soil.

Because the cumulative emissions were the same for each system evaluated at the sampling intervals recommended by international protocols (Parkin, 2008; Parkin & Venterea, 2010; Klein & Harvey, 2015; Butterbach-Bahl et al., 2016), it may be questioned whether the indicated sampling frequency is adequate for the studied conditions and systems. In this sense, it is necessary to assess if increases in sampling frequency to more than once a week would result in different values of cumulative emissions of N_2O from the soil in different systems, mainly the agricultural ones, which are characterized by greater amounts of soil and more cultural management practices.

The availability of and accessibility to an apparatus with automatic chambers make it feasible to sample gases daily, hourly, or more than once a week in long-term experiments, which enhances accuracy and decreases sampling errors (Fassbinder et al., 2013; Reeves & Wang, 2015). Only in this way, will it be possible to follow recommendations to increase sampling frequency after important events that alter gas emissions, such as rainfall, fertilization, and sowing, among other soil and cultural management practices (Parkin & Venterea, 2010; Reeves & Wang, 2015). However, tests performed by Smith & Dobbie (2001) indicate that there is no significant difference in the cumulative estimate of N_2O by increasing sampling frequency from 7 or 3 days to 8 hours. Likewise, Reeves & Wang (2015) observed that gas sampling thrice or once a week in agricultural systems had the same level of accuracy and did not represent significant losses in the annual estimates of soil N_2O emissions.

Therefore, the obtained data show that cumulative N_2O emissions from the soil for the general evaluation of agricultural and forest systems can be estimated at sampling intervals of 7 to 21 days, without significantly hindering final results. However, it is also important to assess other soil and climatic conditions.

Table 1. Main attributes of the 0–10-cm layer of the Hapludox of the evaluated systems[(1)].

System	pH_{H2O}	C (%)	N (%)	S ($cmol_c\ kg^{-1}$)	V (%)	Clay	Silt	Sand
						-------------------	(g kg^{-1})	-------------------
Eucalyptus (*Eucalyptus urograndis*)	5.5	2.4	0.2	1.4	37	520	110	370
Crops	5.8	2.3	0.2	2.3	43	500	120	380
Pasture	5.4	2.6	0.2	1.6	42	490	160	350
Forest	4.6	4.6	0.3	0.5	10	480	110	410

[(1)]C and N, carbon and nitrogen, determined by dry combustion; S, sum of bases, determined by the sum of $Ca^{2+} + Mg^{2+} + K^{+}$; V, base saturation; and clay, silt, and sand determined by the pipette method.

DOI: 10.1590/S1678-3921.pab2019.v54.00211

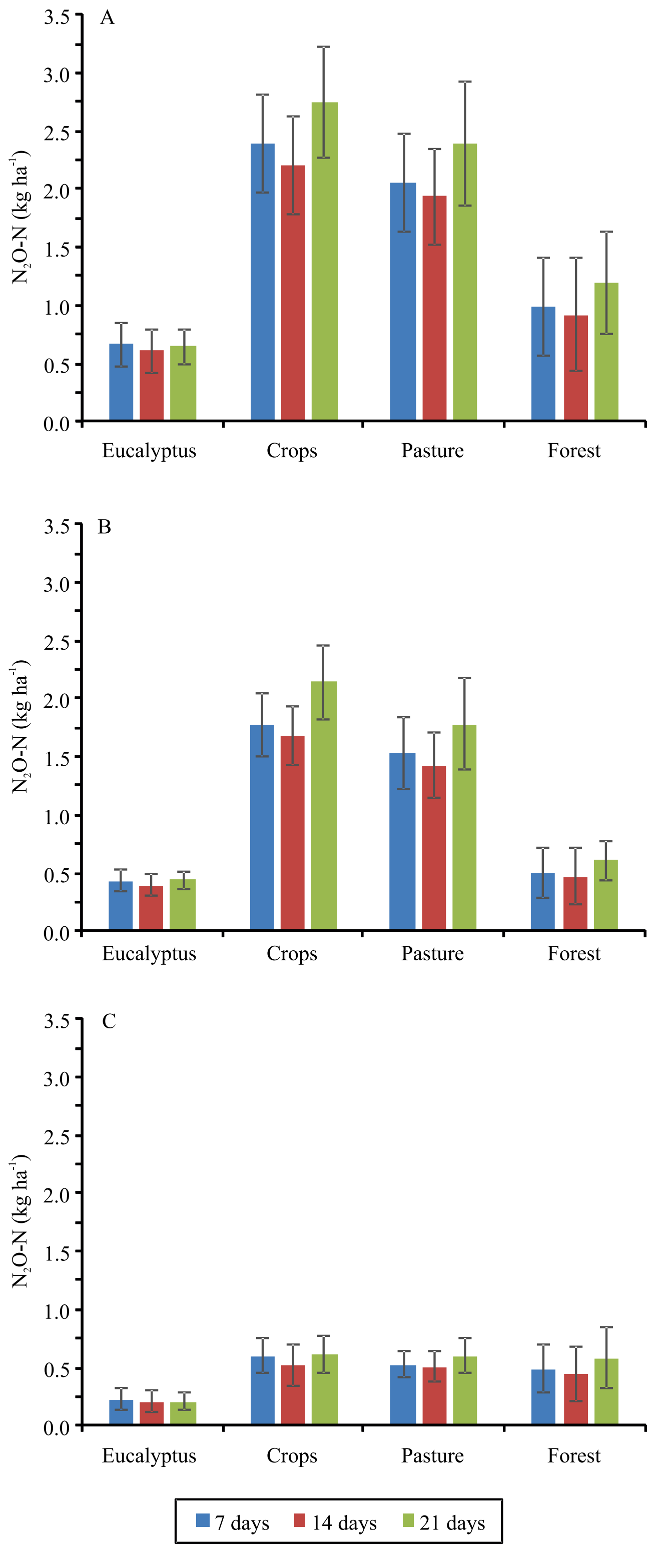

Figure 1. Cumulative N_2O-N emissions from soil under eucalyptus (*Eucalyptus urograndis*), crops, pasture, and native forest, estimated at gas sampling frequencies of 7, 14, and 21 days during two years, i.e., 2014–2016 (A), in the rainy (B) and dry (C) seasons.

Acknowledgments

To Inter-American Development Bank (IDB) and to Rede ILPF, for financial support to carry out the research.

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