



N₂O flux measurements over an irrigated maize crop: A comparison of three methods

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ABSTRACT

This paper presents the NitroCOSMES campaign, aimed at testing and evaluating the performance of three methods for monitoring N₂O fluxes over an agricultural field. The experiment was conducted from May to August 2012 at a site located in the south-west of France. N₂O fluxes from a 24 ha irrigated maize field were measured using eddy covariance (EC), automated chamber (AC) and static chamber (SC) methodologies. Uncertainties were calculated according to the specificities of each set-up. Measurements were performed over a large range of water-filled pore spaces (WFPS), soil temperatures, and mineral nitrogen availability, and offered the opportunity to compare methodologies over a wide range of N₂O emission intensities. The average N₂O fluxes were compared among the three methodologies during the same periods of measurement and for different intensities of emissions (low, moderate and high). Periods of comparison were determined according to the AC results. On average, the three methods gave comparable results for the low (SC: 14.7 ± 2.2 , EC: 15.7 ± 10.1 , AC: 17.5 ± 1.6 ng N₂O-N m⁻² s⁻¹) and the high (SC: 131.7 ± 22.1 , EC: 125.3 ± 8 , AC: 125.1 ± 8.9 ng N₂O-N m⁻² s⁻¹) N₂O emission ranges. For the moderate N₂O emission range, AC measurements gave higher emissions (57.2 ± 3.9 ng N₂O-N m⁻² s⁻¹) on average than both the SC (41.6 ± 6.6 ng N₂O-N m⁻² s⁻¹) and EC (33.8 ± 3.9 ng N₂O-N m⁻² s⁻¹) methods, which agreed better with each other. The relative standard deviation coefficient (RSD) indicated that EC methodology gave highly variable values during periods of low N₂O emissions, from -52.2 ± 88.1 to 62.2 ± 50.7 ng N₂O-N m⁻² s⁻¹, with a mean RSD of 151%. Water vapour effects (dilution and spectroscopic cross-sensitivity) were discussed in an attempt to explain the high variability in low N₂O emission measurements. Even after applying the Webb term correction, there could still be a spectroscopic cross-sensitivity effect of water vapour on the N₂O trace gas signal because of the layout of the analysers, which was not determined during the experiment. This study underlined that EC methodology is a promising way to estimate and refine N₂O budgets at the field scale and to analyse the effects of different agricultural practices more finely with continuous flux monitoring. It also highlighted the need to continue the effort to assess and develop chambers and EC methodologies, especially for the low N₂O emission measurement range, for which values and systematic uncertainties remain high and highly variable.

1. Introduction

The need to assess the dynamics of greenhouse gas exchanges between land surface and atmosphere more accurately is of high priority. While carbon dioxide fluxes have been widely measured using the eddy-covariance method for many years (Baldocchi, 2014), continuous

measurements of nitrous oxide (N₂O) fluxes remain scarce at the ecosystem scale (Nicolini et al., 2013). Since N₂O is estimated to account for 6% of the global greenhouse effect (Ciais et al., 2013), and the application of nitrogen fertilizers in agriculture is estimated to be responsible for more than half of the anthropogenic N₂O emissions (IPCC, 2006), the accurate evaluation N₂O emissions from croplands is critical.

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In a Europe-wide synthesis study performed on 17 different crop sites (51 years of CO₂ flux monitoring), [Ceschia et al. \(2010\)](#) pointed out that N₂O emissions (estimations based upon IPCC 2006 emission factor) had the potential to attenuate the CO₂ sink activity of croplands by 16%. [Zenone et al. \(2016\)](#) demonstrated that N₂O flux would offset 50% of the sink activity in a short rotation coppice used for bioenergy production and that accurate monitoring of the N₂O emission events was critical for deriving correct estimates of the GHG budget. Moreover, [Smith et al. \(2014\)](#) showed that strong potential levers exist for attenuating N₂O emissions from cropland. Nitrogen (N) fertilization modalities, plant (for N use efficiency) and water management appear as key levers in cropland. Although [Lesschen et al. \(2011\)](#) show that the emission factor can vary considerably according to the soil, climate, crop and management, the IPCC emission factor for estimating N₂O emissions remains widely used when N₂O fluxes cannot be monitored continuously. In most studies, N₂O flux measurements are performed using manual or automated chambers combined with a gas chromatograph or infrared analyser ([Eugster and Merbold, 2015](#)). Both chamber methodologies have the advantages of being cost effective and of addressing the issue of spatial variability on reported fluxes within the studied plot ([Cowan et al., 2015](#)). In addition, automated chambers have the advantage of monitoring N₂O fluxes more frequently with less dependence on manpower. They require less gap-filling than manual chambers, which are very demanding in manpower and introduce considerable uncertainty on calculations of the total annual N₂O budget when used at low sampling frequency ([Crill et al., 2000](#); [Smith and Dobbie, 2001](#); [Barton et al., 2015](#)). For both methodologies, one disadvantage is the uncertainty related to spatial and/or temporal sampling rates being too low ([Barton et al., 2015](#)), which may lead to skewed sampling of emissions over the whole range of spatial and temporal variation (under sampling of hot moments).

N₂O emissions from soils are known to vary rapidly in both space and time ([Cowan et al., 2015](#)). The exchanges of N₂O between agroecosystems and the atmosphere depend on complex interactions with the available substrate (nitrogen and carbon), as the feeding process on one side and the availability of oxygen on the other side determine the pathway that is taken in the nitrification or denitrification processes ([Butterbach-Bahl et al., 2013](#)). Hot spots of N₂O production in a plot are often due to high variability of the spatial distribution of organic matter and of texture components (clay particularly), heterogeneous residual crop incorporation, soil compaction, manure or slurry spreading and the area of waterlogged spots ([Cowan et al., 2015](#)). So far, measuring soil-atmosphere trace gas exchanges with high accuracy and adequate spatial representativeness of the whole field remains a challenge. In order to assess the effects of management and climate variability on net GHG budgets, methodologies are required that are more suitable for measuring GHG fluxes at the scale at which agroecosystems are managed, i.e. at the field scale. Micrometeorological methods are the most appropriate at such a scale. During the last decade, micrometeorological greenhouse gas measurements have become more common as an alternative to the traditional chamber ones ([Pattey et al., 2007](#)). With the availability of a new generation of fast analysers ([Hensen et al., 2013](#); [Rannik et al., 2015](#); [Shurpali et al., 2016](#)), an increasing number of investigations are being conducted on the use of the eddy covariance method to measure N₂O fluxes at the ecosystem and landscape scales ([Bureau, 2017](#)), although they still remain too scarce ([Eugster and Merbold, 2015](#)). The majority have been carried out on pasture sites and bio-energy plantations ([Eugster et al., 2007](#); [Neffel et al., 2010](#); [Zona et al., 2013](#); [Merbold et al., 2014](#); [Rannik et al., 2015](#)). The eddy covariance method has the advantage of continuously measuring and directly integrating flux data across a large area (> 100 m²) without disturbing the soil or the interface between the surface and the atmosphere. However, the measurement of small N₂O flux events with the EC method is still very challenging because the N₂O gas analyser requires a much higher resolution to detect N₂O atmospheric fluctuations than is needed for CO₂ fluctuations, since the ratio between

the concentrations of the two gases in the atmosphere is about 1000:1. To our knowledge, only a few studies assessing EC accuracy on N₂O flux measurements have been conducted on crops ([Skiba et al., 1996](#); [Molodovskaya et al., 2011](#); [Wang et al., 2013](#); [Huang et al., 2014](#)). Moreover, [Nicolini et al. \(2013\)](#) have reported that few studies directly compare N₂O flux dynamics using chambers and EC methods over a long period of experimentation at crop plot scale. Most of them have been based on manual chambers, which are subject to large errors due to low frequency of measurement. According to the available studies, [Nicolini et al.](#) drew contrasted conclusions on the issue. Some case studies led to good agreement ([Laville et al., 1999](#); [Jones et al., 2011](#); [Molodovskaya et al., 2011](#); [Hargreaves et al., 1996](#); [Wienhold et al., 1995](#)) while a study carried out in Scotland resulted in poor agreement (differences of up to 200%) between the two methods ([Galle et al., 1994](#); [Hargreaves et al., 1994](#); [Smith et al., 1994](#)). Discrepancies between manual chambers and micrometeorological techniques were mostly due to the differences in the sampled area or spot sources generated by a drainage system within the crop plot, which manual chambers could not measure ([Denmead et al., 2010](#)). It is thus indisputable that eddy-covariance flux systems for N₂O measurement still require evaluation against reference methods with higher frequencies of measurement and longer periods of comparison. A longer period of comparison allows methods to be tested over a large range of variations in key environmental factors.

In this paper, we present the results of the NitroCOSMES project, which was conducted to compare four methods for measuring N₂O fluxes during a growing season over an irrigated maize field: automated chambers, manual chambers, eddy covariance and relaxed eddy accumulation (REA). Unfortunately, the REA method failed rapidly and we did not obtain relevant measurements from it for comparison, so it will not be presented in the following. In this paper, we describe and critically assess the three methods effectively used to measure N₂O fluxes and report results from 100 days of campaign. We postulated that both sets of chambers would capture the spatial heterogeneity of N₂O fluxes along with the area integrated by the EC method. We tested whether the EC method was sensitive enough to capture background N₂O fluxes and, above all, the temporal N₂O flux variability that the chamber methods are not able to monitor. We also suspected and analysed a possible effect of the automated chamber system on soil microclimate, compared to the non-intrusive EC system, and found that it probably created some artefacts in the measurement, inducing over- or under-estimation of the calculated N₂O fluxes.

2. Material and methods

2.1. The experimental site

The campaign to compare methodologies was conducted from 10 May to 18 August 2012 (100 days), on a flat agricultural field site of 24 ha located in the south-west of France, 30 km from the city of Toulouse (43°49'65"N, 01°23'79"E) at an altitude of 180 m above sea level. Located near the village of Lamasquère, the experimental site belongs to a dairy farm which is the property of the Purpan Engineering School ([Beziat et al., 2009](#)). The Lamasquère site (FR-Lam) is also part of the regional spatial observatory (OSR) and the European Research Infrastructure Consortium ICOS (Integrated Carbon Observation System). The soil is classified as clayey (54.3% clay, 33.7% loam, 12% sand). The mean organic carbon and total nitrogen soil contents of the 0–30 cm layer were 80 ton ha⁻¹ and 8.8 ton ha⁻¹, respectively, during the campaign. Winter wheat had been sown in the previous year's rotation. Maize seeds were sown on April 27. The maize was irrigated 5 times during the growing season, fertilized with solid manure (145 kg N eq. per ha) in September 2011 and with mineral nitrogen (urea) once, on 20 May 2012 (110 kg N eq. per ha). Herbicide was applied on 15 May. N₂O flux measurements started on 10 May and ended on 18 August, thus covering the majority of the maize-growing season.

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