

Ammonia volatilization measured with the IHF method in a rainfed arable crop: Evaluation of tillage intensity and the number of experimental replicates

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ABSTRACT

Ammonia (NH₃) volatilization losses have severe impacts on human health, climate change and natural environments, but the effect of tillage intensity on these emissions has been barely evaluated in field conditions, particularly using micrometeorological methods, which require large surfaces. In this context, a field experiment in a barley (*Hordeum vulgare* L.) crop was set up in central Spain, in which NH₃ volatilization losses from non-tilled plots (NT) were compared with those from conventionally tilled (T) plots, using the integrated horizontal flux (IHF) technique with three replicates. Ancillary soil and meteorological measurements were taken to explain NH₃ fluxes, and the effect of the number of replicates on the results was statistically addressed. The highest NH₃ emissions were obtained in NT plots after both basal (40 kg N ha⁻¹ applied as urea) and top-dressing (120 kg N ha⁻¹ applied as urea) fertilization events, while NH₃ emission factors were higher after basal fertilization, in comparison with top-dressing application. Considering the sum of both periods, NT significantly increased NH₃ emissions by 63 % with respect to T. We observed a notable influence of the number of replicates, since only two of the nine combinations of two replicates led to rejecting the null hypotheses. No tillage requires the optimized management of N (timing, rate and particularly source) to abate the potential side effects regarding NH₃ volatilization, while the use of robust measurement methods (e.g. IHF) should be implemented with enough replicates to increase the precision in estimating differences.

1. Introduction

Global ammonia (NH₃) emissions from the agricultural sector have been estimated at 39.2 Tg N yr⁻¹ (average over the last six decades), with croplands accounting for approximately 42 % of these emissions (Yang et al., 2023). Ammonia is an alkaline gas that can react with acidic substances in the atmosphere, forming ammonium (NH₄⁺)-based salts involved in fine particular pollution and causing severe human health impacts (Olszyna et al., 2005). Moreover, deposition of these nitrogenous compounds leads to eutrophication, soil acidification, biodiversity loss and indirect nitrous oxide (N₂O) emissions, with a major impact on climate change (Sutton et al., 2022).

Among the potential strategies for NH₃ volatilization, global synthesis studies have demonstrated the relevance of fertilizer management, particularly N placement through irrigation, injection, incorporation or deep placement (Ti et al., 2019), but also the adjustment of the N rate, the replacement of urea with other synthetic N sources and the use of controlled-release fertilizers or urease inhibitors (Pan et al., 2016). However, less attention has been paid to tillage intensity. Conservation agriculture practices such as no tillage are of major relevance when seeking to mitigate the loss of soil organic matter through erosion and destruction of soil aggregates, decreasing carbon dioxide emissions and producing variable or neutral effects on N₂O and methane (CH₄) (Ruis et al., 2022) as well as other positive impacts on

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soil physical and chemical properties (Blanchy et al., 2023; Zhang et al., 2023). On the other hand, zero-tillage practices promote residue retention over the soil surface, which globally increases NH_3 volatilization by 26 % on average (Pan et al., 2016), and decreases the contact of NH_4^+ with the soil clays and organic matter, and therefore the chances for adsorption (Ma et al., 2021). Conventional and no-tillage management, however, has not been extensively compared at the field scale, particularly using micrometeorological methods such as integrated horizontal flux (IHF).

The IHF method has been considered a robust and meteorologically sensitive method to measure NH_3 fluxes (Herrero et al., 2021; Pacholski et al., 2008; Zhang et al., 2022). Therefore, a number of previous studies, e.g. Bai et al. (2017) or Di Perta et al. (2019), used IHF without replicates as a reference to test other NH_3 methods. Because of the high demand in surface and material, other studies such as Recio et al. (2018) and Recio et al. (2020) set up only two replicates of the IHF method to evaluate the differences between fertilization strategies. All of these authors argued that the robustness of the IHF method made the use of two replicates sufficiently reliable, but this has rarely been tested in croplands (Guardia et al., 2021). In this context, a field experiment was set up to quantify NH_3 volatilization losses in non-tilled and conventionally tilled plots using the micrometeorological IHF method with three replicates. We also aimed to discuss the meteorological and soil variables (moisture, mineral N) to explain NH_3 fluxes and to evaluate the statistical power of using two or three replicates. We hypothesized that: i) no tillage would increase cumulative NH_3 emissions in comparison to conventional tillage and ii) the use of two replicates would be sufficient to detect significant differences between the two tillage management practices.

2. Materials and methods

The experiment was carried out during the 2018–2019 cropping campaign at the “Centro Nacional de Tecnología de Regadíos-CENTER” (Madrid, Spain). Two different measurement periods were established: after seeding fertilization (Period I) and after dressing fertilization (Period II). The site has a typical Mediterranean climate with a mean air temperature of 14.2 °C and an annual rainfall of 384 mm (10-year average values). The soil is a *Typic xerofluvent* with a silty loam texture and basic pH (8.2 in water) and low organic matter (1.7 %, Walkley-Black) in the topsoil (0–20 cm). Meteorological data were obtained from a station located at the field site. The treatments were based on two different tillage practices: no tillage (NT) and tillage (T), which were arranged in a completely randomized design with three replicates. The plots had a surface area of 0.14 ha (36 m × 40 m), 50 m apart. The field has been traditionally managed with conventional tillage and the 2018–2019 campaign was the first one in which the two different tillage treatments were set up. Glyphosate (36 % p/v at) was applied to NT plots in the last week of October, while a disk harrow and a cultivator pass were performed in T plots prior to planting. Urea was applied in all plots at 40 kg N ha⁻¹ (at seeding, mid-November) and 120 kg N ha⁻¹ (at top-dressing, end of February), giving a total rate of 160 kg N ha⁻¹. Barley (*Hordeum vulgare* L.) was sown in mid-December at 200 kg seed ha⁻¹ and the field was kept free of pests following local practices. Ammonia fluxes were measured using the micrometeorological mass-balance IHF method. Briefly, each central mast was equipped with five passive flux samplers (shuttles) consisting of traps of oxalic acid, placed at different heights (0.25, 0.65, 1.25, 2.05 and 3.05 m above the crop canopy), as detailed in Recio et al. (2020) and Guardia et al. (2021). Two background masts with three passive samplers (0.25, 1.25, 3.05 m high) were placed to determine NH_3 background concentrations. Soil samples were taken on six and four occasions in Periods I and II, respectively, to analyse mineral N using flow injection analysis. Further detail on NH_3 volatilization and soil measurements can be found in [Supplementary Material](#).

Cumulative NH_3 fluxes in Periods I and II and the sum of the two periods were compared through an ANOVA analysis using Statgraphics

18 - X64. The sample size (number of replicates) to achieve a statistical power for a minimum effect size was calculated using non-central t distribution with “2 n–2” degrees of freedom and the non-central parameter (Harrison and Brady, 2004). Means comparisons tests for the nine possible combinations of two replicates were also performed for the cumulative emissions in Periods I and II.

3. Results and discussion

3.1. Ammonia volatilization after seeding and dressing fertilization

After seeding fertilization, maximum fluxes were observed at 3 days after fertilization (DAF), reaching 27.0 and 48.3 g N ha⁻¹ h⁻¹ in T and NT, respectively (Fig. 1a). The greatest proportion of NH_3 fluxes occurred during the first 21 days after basal fertilization, accounting for (on average) 89 % of cumulative emissions in Period I, in agreement with the findings of Recio et al. (2020) using the IHF method in an irrigated maize crop. This percentage was lower during Period II (70 %), thus showing that the peaking period was longer, in spite of the lower maximum fluxes reported. This longer peaking period in Period II was in agreement with high NH_4^+ availability even at 29 DAF (Fig. S1b). The proportion of the synthetic fertilizer lost as NH_3 during Period I was 24.0 % and 12.8 % in NT and T, respectively. The emission factors in Period II were 8.6 % and 5.9 % in NT and T, respectively, which were close to that found by Ma et al. (2021) for Europe (6 % on average) and in agreement with the ranges reported by Pan et al. (2016). The higher emissions in Period I than in Period II could be explained by the different meteorological conditions. The main differences between the two periods were the drier conditions after dressing fertilization, i.e. lower cumulative rainfall and water-filled pore space (WFPS) (Fig. S2), particularly during the 1st week after fertilizer addition. Even though the maximum wind speeds and temperatures were higher in Period II than in I (Table S1),

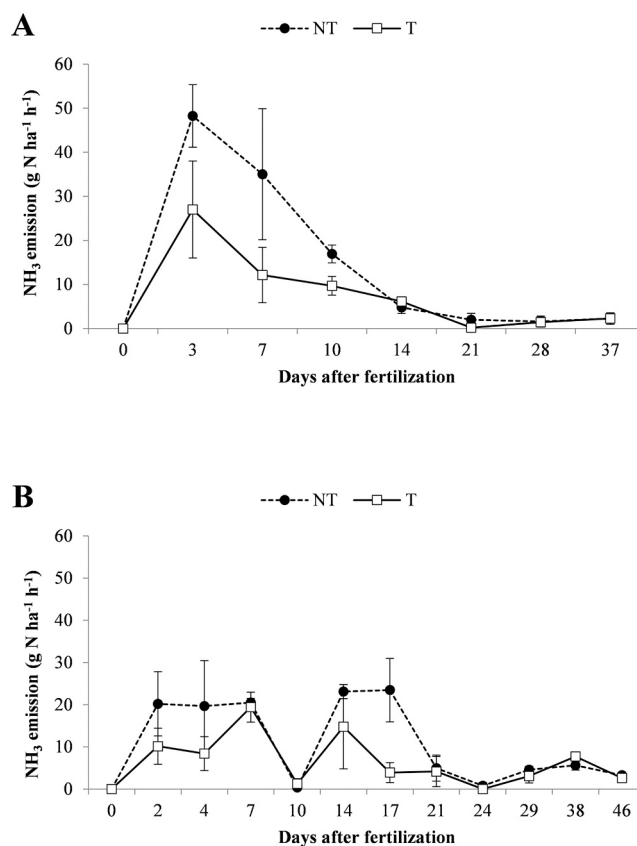


Fig. 1. NH_3 fluxes after seeding (Period I, A) and dressing fertilization (Period II, B). Vertical bars indicate standard errors.

the values in both periods were not limiting for NH_3 volatilization (Congreves et al., 2016). The results suggest, therefore, that rainfall (particularly the 1st week after fertilization) was possibly a key factor driving the amount of N which could be volatilized. Small amounts of rainfall stimulate volatilization losses while large precipitation events (above 7 mm) reduce NH_3 emissions due to the incorporation of the fertilizer (Sanz-Cobena et al., 2019). The scarce (particularly during the first 5 days after fertilization) precipitation during the 1st week in Period I could have contributed to dissolving the granules of fertilizer but was insufficient to incorporate the urea before 6–8 DAF (when two rainfall events > 6 mm took place). After dressing fertilization, no rainfall was recorded until 9 DAF (data not shown), and the granules of urea could have been partially dissolved only by dew drops, with daily minimum air temperatures ranging from -2.3 – 1.4 °C during the 1st week after fertilizer application. In addition, mean and minimum air temperatures in the 1st week after fertilization were warmer during Period I (Table S1), thus possibly contributing to higher volatilization rates.

Cumulative volatilization rates tended to be higher in the NT than in the T system, and this was statistically significant when considering the sum of Periods I and II (Table 1). The main hypothesis for the higher volatilization rates in tilled plots is that the mulch effect is caused by the retention of previous crop residues (oilseed rape) over the soil surface in the NT plots, as suggested by Pinheiro et al. (2018) and Liao et al. (2023), and consistent with those reported by Ma et al. (2021) in which higher background NH_3 emissions were obtained in no-till ($0.44 \text{ mg N m}^{-2} \text{ h}^{-1}$) than in till ($0.13 \text{ mg N m}^{-2} \text{ h}^{-1}$) cropping uplands. In tilled plots, however, the physical barrier of previous crop residues is substantially lower, thus stimulating the adsorption of NH_4^+ to the soil colloid and lessening the opportunities for volatilization (Grandy et al., 2006). This hypothesis was supported by the higher topsoil NH_4^+ concentrations in T than in NT at 3 DAF (Fig. S1a), when the maximum NH_3 flux during Period I was obtained (Fig. 1a). Even though additional studies should be conducted under different meteorological and agricultural conditions, the potential enhancement of NH_3 losses in non-tilled plots in the conditions reported suggests that adequate management of N (e.g. use of $\text{NO}_3\text{-N}$ based fertilizers, addition of urease inhibitors, injection) should be considered to offset this side effect of NT (Ti et al., 2019).

3.2. Relevance of the number of replicates with the IHF method

The probability of obtaining significant differences depending on the size of the effect (expressed as standard deviation) for two and three replicates is represented in Fig. S3. The power for two replicates was always lower than for three replicates, with noteworthy differences between 2 and 6 standard deviations. The effect size that would lead to test powers higher than 0.8 would be 3 and approximately 6 standard deviations for three and two replicates, respectively (Table S2). In addition, contrary to our second hypothesis, the results of the present experiment revealed that the hypothesized statistical power for means comparisons is substantially reduced when using two instead of three replicates, and nearly twice the effect size (in terms of standard deviations) is required to reach the same test power with two replicates, compared to three. The use of two instead of three replicates reduces the precision in the estimated means and reduces the probability of rejecting the null hypothesis when there are real differences between the treatments (in our case, NT versus T). In this sense, during Period I, three of the nine possible combinations of two replicates would reject the null hypothesis with a 95 % confidence level (Table S3), in contrast to the results obtained with three replicates (Table 1). In Period II, none of the combinations of two replicates would reject the null hypothesis (Table S4). For the sum of the two periods, the null hypothesis was rejected using three replicates (Table 2), but this would only occur for two of the nine combinations of two replicates at a 95 % probability level (Table 2). These results should be taken with caution because of the different meteorological conditions. Also, the factor tested (N

Table 1

Estimated cumulative NH_3 emission in Periods I and II and the sum of the two. S.E. indicates standard error of the mean.

Treatment	Cumulative volatilization		
	(kg N ha ⁻¹)		
	Period I	Period II	Period I + II
NT	9.6	10.3	19.9
T	5.1	7.1	12.2
S.E.	1.4	1.1	1.3
P-value	0.089	0.111	0.043

Table 2

Difference between estimated means, standard error (SE), degrees of freedom (df), confidence interval and hypothesis test for the nine possible combinations of replicates in tillage (T) and no tillage (NT) for the cumulative volatilization fluxes in the sum of Period I and Period II.

contrast	estimate	SE	df	lower.CL	upper.CL	t.ratio	p.value
NT - T	9.892	2.313	2	-0.061	19.845	4.276	0.051
NT - T	11.077	1.216	2	5.847	16.307	9.113	0.012
NT - T	8.851	1.341	2	3.080	14.622	6.599	0.022
NT - T	6.194	3.792	2	-10.122	22.511	1.634	0.244
NT - T	7.380	3.241	2	-6.567	21.327	2.277	0.151
NT - T	5.153	3.291	2	-9.005	19.312	1.566	0.258
NT - T	6.822	4.316	2	-11.748	25.392	1.581	0.255
NT - T	8.007	3.841	2	-8.519	24.534	2.085	0.172
NT - T	5.781	3.883	2	-10.925	22.487	1.489	0.275

placement, tillage, fertilizer source) influences the amount of NH_3 volatilized, the differences between mean values (e.g. the difference in cumulative NH_3 fluxes between incorporation versus no incorporation, or between urea with or without urease inhibitors, could be greater than those observed between NT and T) and the variance of the data. Therefore, our results suggest that the variance between IHF replicates is not negligible, and increasing the number of replicates is encouraged to increase precision in estimating differences.

4. Conclusions

Our results confirmed the risk of increasing NH_3 volatilization losses when adopting no tillage management. Therefore, we encourage the use of well-known improved N management practices such as urea incorporation by rainfall/irrigation water or the choice of N source (including the use of urease inhibitors) in non-tilled croplands to prevent these negative side-effects. Differences in cumulative emissions were only significant when considering the sum of Period I (after seeding fertilization, when the highest volatilization rates were obtained despite the lower urea-N rate) and Period II (after dressing fertilization). Even though the micrometeorological IHF method is considered as a reference technique for quantifying NH_3 volatilization losses, we demonstrated that its robustness and the significance of results were influenced by the number of replicates. The use of two replicates only led to significant differences between tillage intensities in 22 % of the possible combinations, while the implementation of non-replicated IHF experiments for NH_3 should be avoided.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.still.2023.105892](https://doi.org/10.1016/j.still.2023.105892).

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