Ammonia volatilization and yield of corn fertilized with different nitrogen sources in the Brazilian semiarid

Abstract – The objective of this work was to evaluate the effect of the use of different nitrogen fertilizers on N losses by NH₃ volatilization and on the grain yield of a corn (Zea mays) crop grown in the semiarid region of the state of Sergipe, Brazil. Corn was managed under rainfed conditions and conventional tillage. The fertilization treatments were: sulfur-coated urea (SU) and organo-mineral-coated urea (OMU). Regular urea and ammonium sulfate (AS) were also included as a reference of N sources of high and low NH₃-volatilization potential, as well as a control without N fertilization. The N sources were applied to the soil surface at a rate of 150 kg ha⁻¹ N as side-dressing at the V5 growth stage of corn. The experiment was carried out in 2015 and repeated in 2016. Losses of N through NH₃ volatilization differed among the N sources in the two study years, with AS presenting the lowest N losses and regular urea, the highest. In the area treated with OMU, losses by NH₃ volatilization were consistently controlled, showing a reduction of 74 and 67% in relation to that of regular urea in both years. SU, however, only mitigated NH₃-N losses in 2015, increasing them in 2016, compared with regular urea. Corn plant grain yield and N status, assessed by the index leaf, did not clearly reflect N losses by NH₃ volatilization. For the semiarid of Sergipe, OMU is the best urea-based fertilizer to mitigate N losses by NH₃ volatilization, and the use of AS is an alternative to increase corn yield and eliminate N losses as NH₃.

Index terms: Zea mays, ammonium sulfate, mitigation, urea-based fertilizers.
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Materials and Methods

The experiment was conducted during the 2015 and 2016 crop seasons in an area cropped with corn (Zea mays L.) under conventional tillage for several years, located at the experimental station of Embrapa Tabuleiros Costeiros, in the state of Sergipe, Brazil (10°27'S, 37°11'W, at 200 m above sea level). By the Brazilian soil classification system, the soil is an Argissolo Vermelho-Amarelo distrófico (Santos et al., 2013), i.e., a Haplic Acrisol (FAO, 2015). According to Köppen’s classification, the climate is of the AS type, being characterized as rainy tropical with a
dry summer. From 2001–2016, the mean annual temperature was 26°C and annual precipitation was 1,051 mm, of which 68% occurred between May and October (Figure 1).

The area was limed in 2014 at a rate of 1.2 Mg ha⁻¹ incorporated to a 20-cm depth. Soil samples were taken from the 0–20 and 20–40-cm layers two months before the experiment was installed for chemical and physical characterization (Table 1) using the methods described in Silva (1999). Prior to sowing, the area was ploughed and harrowed twice. Corn was mechanically sowed without N fertilizer application on 05/20/2015 and 05/16/2016 at a row spacing of 0.50 m, totaling 74,000 plants per hectare. The single-cross corn hybrid, AG 7088 VT PRO Max (Agroceres, Rio Claro, SP, Brazil), was used in both years. Fertilization with P and K was carried out at sowing, by applying 100 kg ha⁻¹ P₂O₅ as triple superphosphate and 80 kg ha⁻¹ K₂O as potassium chloride, due to the low P (< 7 mg dm⁻³) and medium K (30 to 45 mg dm⁻³) availabilities, both considered limiting for high grain yield (Sobral et al., 2007).

The N fertilization treatments consisted of: sulfur-coated urea (SU), containing 37% N and 16% S (FH Nitro Gold, Fertilizantes Heringer, Rosário do Catete, SE, Brazil); and organo-mineral-coated urea (OMU), containing 29% N, 9% S, 5% Ca, 2% Mg, and 0.3% B (Sulfammo MeTA, TIMAC Agro, Santa Luzia do Norte, AL, Brazil). Dry granular urea with 46% N (Petrobras, Fafen-SE, Laranjeiras, SE, Brazil) and ammonium sulfate (AS) with 20% N and 22% S (Petrobras, Fafen-SE, Laranjeiras, SE, Brazil) were also included as a reference of regular N sources, as well as a control without N fertilization. The experiments were arranged in a randomized complete block design, with five treatments and three replicates. The experimental units were 2x5-m plots with four plant rows. To prevent a bordering effect, only the two central rows in the plot, discarding 0.5 m from both ends of each row, were sampled. The N fertilizers were surface applied to the total area between rows at a rate of 150 kg ha⁻¹ N, at 36 and 31 days after sowing in 2015 and 2016, respectively, during the V5 growth stage of corn, when plants have a high demand for N (Sangoi et al., 2007). Even though the application of the whole N dose as side-dressing is unusual, its potential for use in treatments is likely improved as NH₃ losses by volatilization are expected to increase with urea-N rates (Cantarella et al., 2018).

Immediately after N fertilization, two semi-open chambers (Araújo et al., 2009; Jantalia et al., 2012) per plot were installed to measure NH₃ volatilization: one in a row between plants and another in the interrow space. The chamber was made of a transparent polyethylene terephthalate (PET) bottle, with a 2.0-L

Table 1. Soil chemical and physical characteristics in the experimental area.

| Depth       | Chemical characteristics | Physical characteristics |
|-------------|--------------------------|--------------------------|
|             | pH (H₂O) | OM (1) (g kg⁻¹) | P (mg dm⁻³) | K (mg dm⁻³) | Ca (mmol dm⁻³) | Mg (mmol dm⁻³) | H⁺+Al (mmol dm⁻³) | Al (mmol dm⁻³) | Na (mmol dm⁻³) | V (2) (%) | Sand (g kg⁻¹) | Silt (g kg⁻¹) | Clay (g kg⁻¹) |
| 0–20 cm     | 5.9     | 16.3          | 6.2          | 35.9        | 24.2         | 18.8          | 11.9           | 0.3          | 1.3          | 79.2      | 552          | 183          | 265          |
| 20–40 cm    | 5.8     | 12.9          | 2.4          | 16.8        | 21.9         | 16.6          | 12.0           | 0.2          | 0.9          | 76.8      | 489          | 194          | 317          |

(1)OM, organic matter. (2)V, base saturation.
capacity and 100-mm diameter (78.5 cm²), whose bottom was removed, leaving it with a final length of 260 mm. Inside each chamber, a 25-cm long wire was used to support a 2.5x25-cm foam strip with 3.0 mm of thickness and also a 50-mL plastic pot, placed at the bottom end. Before chamber deployment, the foam strip was pre-soaked in a 10-mL acid solution of 1.0 mol L⁻¹ sulfuric acid + 2.0% glycerol. After that, the upper side of the foam was hung on top of the wire support and the other extremity was put inside the plastic pot held by the wire; the solution that was not absorbed by the foam remained inside the pot. The foam was replaced daily every five days depending on the sampling scheme defined for each year of the study. According to Araújo et al. (2009), foam is efficient for trapping volatilized NH₃ for periods of up to six days.

To ensure the homogeneity of fertilizer application, a 0.9-m space in the plant rows was reserved to manually receive a corresponding amount of fertilizer, whose granules were carefully spread out. This area was used to monitor NH₃ volatilization time. When the foam was replaced, the chamber was reallocated to the next position in the reserved area based on the procedure described by Jantalia et al. (2012). This reallocation aimed to expose the fertilized area to rainfall, dewing, and evaporation (Cantarella et al., 2008). Foams and their respective plastic pots were replaced on a daily basis during the first 7 days after N fertilization (DAF) in both study years and on the ninth, thirteenth, and eighteenth DAF in 2015, and on the eighth, tenth, and thirteenth DAF in 2016. Still in the field, the replaced foam strips were immediately put in their corresponding plastic pot and sent to a laboratory for the NH₃-N analysis. There, ammonia-N was determined after a careful transfer of the foam to an Erlenmeyer flask, followed by rinsing the plastic pots with 50 mL deionized water. The procedure of NH₄⁺ quantification in the solution was detailed in Araújo et al. (2009). A correction factor was developed by Araújo et al. (2009) and Jantalia et al. (2012) using the ¹⁵N balance as a measure of the real NH₃ volatilization. The cumulative NH₃-N volatilization for the whole deployment period was calculated by summing up the results obtained for each intermediary sample. The fraction of the N fertilizer lost as NH₃ (Frac_GASF) was calculated as the ratio of the net NH₃-N volatilized and the N fertilizer rate, all in a same area basis (Eggleston et al., 2006). The net NH₃-N volatilized was calculated as the difference between the cumulative NH₃-N obtained for the N source and the control.

In the experiment carried out in 2015, five plants at the R1 growth stage were randomly chosen from each plot. The opposite leaf immediately below the first ear – index leaf for N fertility – was collected and processed for further analysis of N content (%) using Kjeldahl digestion (Silva, 1999). To determine grain yield, whole corn plants from a 1.5-m² area of each plot were manually harvested in late October each year – on the 29th in 2015 and on the 17th in 2016 –, when the plants were at or near physiological maturity. For mass determination, the grains were separated from the ears with a mechanical sheller, and yield was expressed on a 130-g kg⁻¹ moisture content basis.

The data of cumulative NH₃ volatilization and grain yield were subjected to the analysis of variance (Anova), and means were compared using Fisher’s least significant difference test, at 5% probability. The Anova was carried out after checking for normality of errors by Shapiro-Wilk’s normality test and for homoscedasticity by Levene’s test. Data were log (x) transformed when the assumptions for the Anova were not met. Pearson’s correlation was performed between volatilized NH₃ and the N content of the index leaf. Statistical analyses were performed using the Sisvar, version 5.6 (Ferreira, 2011), and the XLSTAT, version 2020.1.1, software (Addinsoft, Paris, France).

Results and Discussion

For both study years, NH₃ volatilization due to the treatments remained for no longer than 10 DAF, indicating that the monitoring time of 13 and 18 days was enough to compute the whole N loss, regardless of fertilizer type (Figure 2). The urea-based fertilizers – regular urea, OMU, and SU – had NH₃ volatilization rates well above those of the control, while AS was virtually inert to this N-loss process. The process of NH₃ volatilization is primarily dependent on the dissociation of NH₄⁺ into NH₃ and H⁺, whose magnitude will vary with NH₄⁺ and H⁺ availabilities in the soil. For instance, the application of AS increases the concentration of NH₄⁺ but contributes to soil acidification (Zhao et al., 2007). Therefore, NH₃ volatilization from this source is frequently very low when on neutral to acid soils...
Figure 2. Daily flux of ammonia volatilization and precipitation up to the eighteenth day after the application of 150 kg ha\(^{-1}\) N in the 2015 (A) and 2016 (B) corn (Zea mays) crop seasons. Bars indicate the default error. Control, without N fertilization; SU, sulfur-coated urea; OMU, organo-mineral-coated urea; Urea, granular urea; and AS, ammonium sulfate.
This justifies the similar N losses in the AS and control treatments for both experimental years; AS only caused losses 0.9 to 3.1 kg ha$^{-1}$ N above those of the control, representing less than 2.5% of the N applied (Table 2). In contrast, the alkaline reaction of urea removes H$^+$ from the soil solution and enhances the dissociation of NH$_4^+$ that may lead to significant N losses through NH$_3$ volatilization (Sommer et al., 2004).

In 2015, all the urea-based treatments presented NH$_3$ losses higher than those of the control until 7 DAF, which corresponded to 95.4% of the total NH$_3$-N loss. Lower volatilization rates were registered in 2016, with maximum volatilizations rates of 14 and 10 kg ha$^{-1}$, respectively, at 2 DAF in areas treated with SU and at 3 DAF with urea. After 6 DAF, NH$_3$ volatilization decreased to the basal levels measured in the non-fertilized control (Figure 2 B). Very often, NH$_3$ losses from urea are very intense within the first three days after the process starts, and most of the N loss is computed in no more than a week (Sommer et al., 2004; Cantarella et al., 2018). However, in the present study, fertilizers showed different patterns and magnitudes of NH$_3$ volatilization. In 2015, NH$_3$ volatilization increased very fast for the urea-based fertilizers, but following slightly different patterns (Figure 2 A). For the SU and OMU treatments, NH$_3$ volatilization peaked at 1 DAF, with a N loss of 9 and 4 kg ha$^{-1}$, respectively, while the regular urea treatment had a volatilization peak at 2 DAF, with a 27-kg ha$^{-1}$ NH$_3$-N loss. The volatilization rates tended to reduce, but in an oscillating manner, which was relatively more relevant for SU and OMU. This pattern was considered as a result of the gradual liberation of urea across the coating layer (Mariano et al., 2019). In 2016, this behavior was not observed, but there was a 1-day delay for the highest losses, together with an inversion in the

| Treatment | NH$_3$-N losses (kg ha$^{-1}$) | Total N applied (%) | Fraction of applied N lost as NH$_3$-N | Reduction compared with urea (%) |
|-----------|-------------------------------|---------------------|----------------------------------------|---------------------------------|
|           | Cumulative NH$_3$-N | NH$_3$-N fertilizer |                                        |                                 |
| OMU       | 17.2b                        | 15.6bc              | 10.4                                   | 0.10                            | 74.1                            |
| SU        | 30.2ab                       | 28.3ab              | 18.9                                   | 0.19                            | 53.0                            |
| AS        | 5.0c                         | 3.1c                | 2.1                                    | 0.02                            | 94.9                            |
| Urea      | 62.1a                        | 60.2a               | 40.1                                   | 0.40                            | -                               |
| Control   | 1.9c                         | -                   | -                                      | -                               | -                               |
| Mean      | 23.3                         | 26.8                | 17.9                                   | 0.18                            | -                               |
| Coefficient of variation (%) | 22.95                        | 33.27              |                                        | -                               | -                               |

2016 experiment

| OMU       | 9.3bc                         | 6.4b                | 4.3                                    | 0.04                            | 67.2                            |
| SU        | 31.1a                        | 28.3a               | 18.9                                   | 0.19                            | -45.1                           |
| AS        | 3.8cd                        | 0.9c                | 0.6                                    | 0.01                            | 95.4                            |
| Urea      | 22.3ab                       | 19.5ab              | 13.0                                   | 0.13                            | -                               |
| Control   | 2.8d                         | -                   | -                                      | -                               | -                               |
| Mean      | 13.9                         | 13.8                | 9.2                                    | 0.9                             | -                               |
| Coefficient of variation (%) | 22.41                        | 37.05              |                                        | -                               | -                               |

(1)Means followed by equal letters do not differ statistically by Fisher’s least significant difference test, at 5% probability. (2)OMU, organo-mineral-coated urea; SU, sulfur-coated urea; AS, ammonium sulfate; Urea, granular urea; and Control, without N fertilization.
importance of urea with SU in terms of the magnitude of the losses.

According to Sommer et al. (2004), the triggering of NH₃ volatilization after urea placement on the soil will depend on urea dissolution and hydrolysis into NH₄⁺ and HCO₃⁻. The latter produces a H⁺ sink around the urea granule and enhances the dissociation of NH₃. Soil temperature and moisture are key factors in the dissolution and hydrolysis of urea (Da Ros et al., 2005) and are likely to control the extension of the lag phase between urea application to the soil and the start of NH₃ volatilization.

In 2015, soil moisture was probably high due to the 12-mm rainfall the day before fertilizer application, contrarily to the observed in 2016, when rainfall was very low both on this day and throughout the year (Figure 2). This difference is the probable explanation for the shorter lag phase between fertilization and NH₃ volatilization in 2015. However, rainfall during the first days after fertilization was almost twice as great in 2016 and increased the chances of moving the dissolved urea into the soil, causing a reduction in NH₃ volatilization. Similar effects were reported by Black et al. (1987) in a study under controlled conditions. In 2015 and 2016, the use of regular urea resulted in 62.1 and 22.3 kg ha⁻¹ N lost as NH₃ or in a net loss of 60.2 and 19.5 kg ha⁻¹ NH₃-N if the NH₃ volatilization computed for the control is deduced (Table 2).

Consistently, the use of OMU, a urea-based product, caused a lower NH₃ volatilization in both years (Figure 2), amounting net losses of 15.6 and 6.4 kg ha⁻¹ in 2015 and 2016, respectively, representing 10.4 and 4.3% of the added N (Table 2). Compared with regular urea, OMU mitigated N loss due to a NH₃ volatilization of 74.1 and 67.2% in the two study years. Since the industrial process of urea coating is not available for this product, perhaps a mixture of sulfur, wax, and a conditioner was used to give it a degree of hydrophobicity associated with coating resistance (Hignett, 1985). The presence of such a barrier for urea dissolution explains the relative delay in NH₃ volatilization in OMU and SU, when compared with regular urea in the present study. Sulfur may help to neutralize, to some extent, the alkalinization associated with the released urea because of its acidic reaction in the soil. However, despite the possibility of similar effects due to the presence of either a coating barrier or acidification by sulfur, the SU treatment was not as effective in controlling NH₃ volatilization. The use of SU practically resulted in a 19% loss of the N applied as NH₃, regardless of the year (Table 2), or in about twice the loss of N in the OMU treatment, although it had approximately twice the sulfur concentration. However, the use of SU mitigated N loss in 53%, compared with regular urea, in 2015. Contrarily, the efficiency of this product was not confirmed in 2016, when the volatilized NH₃-N was 45% greater than that of the regular urea treatment.

The coating process and even the way the many components are mixed may result in fertilizers of contrasting efficiency, as reported by Rech et al. (2017). The accumulated NH₃-N loss observed by these authors in the treatment with SU, also from the Nitro Gold commercial fertilizer, represented 23.2% of the 120 kg ha⁻¹ N fertilization rate used, while the N loss for urea was equivalent to 26.8%. In their study, the presence of boron in the urea-based fertilizers also resulted in significant reductions in NH₃ volatilization, since it deactivates the urease enzyme; although the efficiency of this process is not always high, it was better than that of a SU-type fertilizer (Mariano et al., 2019). However, Cancellier et al. (2016) observed that SU-type fertilizers resulted in lower NH₃ losses than urea-based fertilizers with boron addition, making it challenging to reach a consensus on the topic.

Regardless of year-to-year differences, AS had the lowest N losses as NH₃, with a Frac_GAS close to zero, while the urea-based fertilizers induced higher NH₃-N losses. Regular urea and SU presented a Frac_GAS in the range of 0.13 to 0.40 kg NH₃-N per kilogram of N applied, while OMU was the most efficient N source, with a Frac_GAS of 0.04 to 0.10 kg NH₃-N per kilogram of N applied (Table 2). As a global average of all N sources, the Intergovernmental Panel on Climate Change (IPCC) (Eggleston et al., 2006) adopted a Frac_GAS of 0.10 kg NH₃-N per kilogram of N applied for the estimates of volatilized N for greenhouse gas inventories, which is in a reasonable agreement with the averages of the N sources evaluated in the present study, especially in 2016, even though disaggregating urea from other fertilizers would improve the precision of estimates.

Corn yield presented a contrasting response to fertilizer type only in 2015 (Figure 3). The fertilization with AS resulted in a grain yield of 6.5 Mg ha⁻¹, while the non-fertilized control exhibited...
a yield of 2.2 Mg ha\textsuperscript{-1}. The use of urea-based fertilizers caused similar responses in yield, which was 5.1 Mg ha\textsuperscript{-1} on average. In September 2015, the low precipitation during ear filling (Figure 1) reduced significantly the yield potential of corn. The highest net yield (above that of the control) in AS is in agreement with the lowest N loss as volatilized NH\textsubscript{3}, and a lower net yield for regular urea with the highest NH\textsubscript{3}-N loss contributes for a relationship between both variables. Despite the great differences between the amount of N volatilized between urea, SU, and OMU, a corresponding variation was not observed in corn grain yield. Although the total N accumulated by the plant was not assessed, the index leaf was used to evaluate the nutritional status of the plants. For AS, SU, OMU, urea, and the control, the respective N contents (%) in the leaf index were 3.83 (±0.25), 3.51 (±0.16), 3.37 (±0.03), 3.36 (±0.11), and 2.16 (±0.08). Rambo et al. (2011) showed that the total N accumulated by the plant and index leaf at the earing stage was a very good predictor of corn yield, indicating their interrelation. However, the net N volatilization as NH\textsubscript{3} only presented a trend of indirect relationship with the variation in the percentual increment in the N of the index leaf, taking the control as a reference (Figure 4). In 2016, average corn yield for all treatments was 10.5 Mg ha\textsuperscript{-1}, as it did not respond to N fertilization. In this year, yields were about 100% higher than the highest yield of 2015, even though rainfall volume was smaller (Figure 1), but possibly more evenly distributed.

Cantarella et al. (2018) reviewed several studies and verified that NH\textsubscript{3} volatilization rates are rarely accompanied by crop yields. The best explanation seems to be the use of N rates above the crop needs, which are the result of high soil fertility, cropping system, or other limitations related to nutrition, pests, and climate. Therefore, the better performance of AS in 2015 could be related to the provided 165 kg ha\textsuperscript{-1} S; however, the SU fertilizer with 16% S, which added 65 kg ha\textsuperscript{-1} S, was ineffective in incrementing corn yield, when compared with regular urea or with OMU, which added 47 kg ha\textsuperscript{-1} S. In 2016, no effect of S fertilization was observed on yield performance. A review on crop yield response to S fertilizer in Brazil pointed out that no more than ~26 kg ha\textsuperscript{-1} S would be enough to meet plant demand and replenish soil reserves (Pias et al., 2019).
Conclusions

1. The organo-mineral-coated urea is the best urea-based fertilizer for reducing NH$_3$ volatilization losses, although compromising corn (Zea mays) yield, while ammonium sulfate is the alternative for increasing yield and eliminating NH$_3$ losses.

2. For the semi-arid region of the state of Sergipe, Brazil, the amount of nitrogen lost by NH$_3$ volatilization has no significant effect on corn yield and N nutrition.

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