

Liming and enhanced-efficiency nitrogen fertilizers: Ammonia volatilization losses from subtropical no-till

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ABSTRACT: In tropical and subtropical agricultural no-till (NT), lime and nitrogen (N) fertilizers are surface-applied. The pH increase from liming can enhance ammonia (NH₃) volatilization. This study aimed to quantify NH₃ losses from NT areas treated with different N fertilizers and lime rates and assess the effects on topsoil pH. Field experiments were conducted on clayey soil (*Latossolo Vermelho distroférrico típico*). The experimental design was a randomized complete block design with a 4 × 4 factorial split-plot arrangement and four replications. Main plots received four rates of dolomitic limestone (0, 2.6, 5.4, and 8.1 Mg ha⁻¹) and subplots received four N fertilizers. Nitrogen fertilizers were: conventional urea (46 % N), urea coated with copper (Cu) and boron (B) (Ur-Cu+B; 0.15 % Cu, 0.4 % B, and 44 % N), NBPT-treated urea (Ur-NBPT, 46 % N and 0.53 % NBPT), and ammonium sulfate (AMS, 21 % N and 22 % sulfur). Nitrogen fertilizers were applied during two second-crop corn growing seasons, at 24 and 36 months after liming. Soil pH in the 0.00-0.05 m layer was determined at the end of the experiment. The increase in soil pH due to liming enhanced NH₃-N volatilization losses, regardless of the N fertilizer used. Ammonia volatilization losses ranged from 16 to 32 % in urea plots, 12 to 31 % in Ur-Cu+B plots, 7 to 19 % in Ur-NBPT plots, and 0.2 to 5 % in AMS plots treated with lime rates of 0 to 8.1 Mg ha⁻¹, respectively. For every 1 Mg ha⁻¹ of applied lime, NH₃-N volatilization increased by 1.7 to 2.2 % in Ur plots, 1.6 to 5.1 % in Ur-Cu+B plots, 1.3 to 1.5 % in Ur-NBPT plots, and 0.7 to 0.9 % in AMS plots. These results highlight the need to adjust N management after liming, with ammonium-based fertilizers (e.g., AMS) or stabilized urea sources (e.g., Ur-NBPT) being more effective mitigation strategies than urea or Ur-Cu+B in reducing NH₃ volatilization under surface-limed no-till conditions.

Keywords: lime, nitrogen pollution, micronutrients, soil acidity, urea.



INTRODUCTION

No-till (NT) covers approximately 180 million hectares globally (Kassam et al., 2019), making it a key component of conservation agriculture. No-till management practices promotes agroecological sustainability, contributing to increased yields, profitability, and food security, while preserving environmental and social resources (Pittelkow et al., 2015). Most soils managed under NT in humid tropical and subtropical regions have problems with soil acidity, which restricts root growth and reduces crop yield.

Ammonia (NH_3) volatilization is one of the main routes of nitrogen (N) loss from agricultural soils (Chen et al., 2021). This process is a significant contributor to environmental damage, negatively affecting water and soil quality and promoting the formation of acid rain (Good and Beatty, 2011). Furthermore, recent studies demonstrated that NH_3 volatilization increases the concentration of fine particulate matter ($\text{PM}_{2.5}$) in the air, having a direct impact on human health (Hill et al., 2019; Wyer et al., 2022). The rate of NH_3 volatilization is influenced by numerous factors, including soil temperature and moisture content (Viero et al., 2014), method of application (Woodley et al., 2020), fertilizer rate (Oliveira et al., 2024), N source (Cassim et al., 2022), soil texture and pH (Sunderlage and Cook, 2018), and the presence of crop residues on the soil surface (Dick, 1984). In NT, greater urease and microbial activity stimulated by crop residues on the soil surface accelerate urea hydrolysis, increasing the risk of N losses.

In NT, lime is applied on the soil surface without incorporation (Caires et al., 2008). The benefits of this method of application are well documented and include a gradual pH increase along the soil profile (Minato et al., 2023a), the maintenance of soil physical quality (Brignoli et al., 2024), enhanced soil biological activity, increased carbon stocks, and improved crop yields (Inagaki et al., 2016). However, when lime and nitrogen (N) fertilizers are applied to the same plots, the increase in soil pH caused by lime dissolution, combined with urea hydrolysis, can promote NH_3 volatilization (Minato et al., 2023b; Cassim et al., 2024a). Consequently, losses due to NH_3 volatilization may decrease N fertilization efficiency and crop yields.

Urea is the most used N fertilizer worldwide. When applied to the surface, urea is hydrolyzed by urease, resulting in the formation of ammonium ions (NH_4^+) and carbon dioxide (CO_2). As urea hydrolysis consumes protons (H^+), the reaction increases the pH of the granulosphere, shifting the equilibrium toward the formation of NH_3 , which may be lost to the atmosphere (Rochette et al., 2009; Cantarella et al., 2018). Therefore, under conditions of increased soil pH, such as those found in topsoil following surface liming, N fertilizers, particularly urea-based fertilizers, may have reduced efficiency. Ultimately, this loss of efficiency translates into economic, environmental, and agronomic losses. Recently, Minato et al. (2023b) reported that N losses from urea applied to NT increased by 2.3 to 2.5 % for every 1 Mg ha^{-1} of lime applied.

Enhanced-efficiency fertilizers (EEFs) have emerged as a promising technology for reducing N volatilization losses. In a global meta-analysis, Pan et al. (2016) reported that EEFs reduced NH_3 volatilization by 54 %. This class of fertilizers includes slow-, controlled-, and stabilized-release formulations. Stabilized fertilizers can be further divided into those containing urease inhibitors and those containing nitrification inhibitors (Trenkel, 2010). Urea treated with *N*-(*n*-butyl)thiophosphoric triamide (NBPT) and urea coated with micronutrients such as boron (B) and copper (Cu) are prominent examples of stabilized N fertilizers (Mariano et al., 2019). The mechanism of action of these fertilizers involves the temporary inhibition of urease activity in soil, thereby decreasing the rate of urea hydrolysis (Cassim et al., 2024b).

An in-depth understanding of the relationship between soil chemical properties and N fertilizer dynamics enables the development of effective strategies to mitigate NH_3 emissions. In view of the intensification of production systems and the widespread use

of surface-applied lime in tropical and subtropical regions, it is crucial to investigate NH_3 volatilization losses after liming to increase N use efficiency (NUE) in NT. The hypothesis is that stabilized fertilizers are more effective than urea in reducing NH_3 volatilization loss from limed soils, particularly at higher lime application rates. This study aimed to quantify NH_3 volatilization losses from soils treated with different N fertilizers under increasing lime rates and to assess the associated changes in topsoil pH in a NT.

MATERIALS AND METHODS

Experimental design and environmental conditions

Field experiments were conducted in 2016 and 2017 at the Technology Diffusion Unit of the Cocamar Cooperativa Agroindustrial, Floresta, Paraná, Brazil (23°35' S and 52°04' W, 392 m a.s.l.). The climate of the area is of the Cfa type (humid subtropical) (Köppen, 2011), with average temperatures of 17 and 28 °C in the coldest and warmest quarters, respectively. Climate data for experimental periods are summarized in figure 1. The soil of the experimental field was classified as a *Latossolo Vermelho Distroférrico típico* with clay texture, according to the Brazilian Soil Classification System (SiBCS) (Santos et al., 2018), corresponding to a Rhodic Eutruxox (Soil Survey Staff, 2014). The field has been managed under NT for over 20 years. Analysis of soil properties in the 0.00–0.20 m layer prior to lime application in 2014 (baseline characterization of the experimental area), showed the following chemical (Pavan et al., 1992) and texture (Bouyoucos, 1962) properties: pH(CaCl_2) 4.9, 17.7 g dm^{-3} carbon, and 770 g kg^{-1} clay.

The experimental design was a randomized complete block design with a 4 × 4 factorial split-plot arrangement and four replications. Main plots were assigned to four rates of dolomitic limestone (0, 2.6, 5.4, and 8.1 Mg ha^{-1}) applied to the soil surface in 2014. The limestone levels evaluated represent different recommendation scenarios based on the base saturation method (BS%), following the approach proposed by Minato et al. (2023a). The rate of 2.6 Mg ha^{-1} corresponds to the amount required to increase BS% to 70, while 5.4 Mg ha^{-1} corresponds to raising BS% to 90. Finally, 8.1 Mg ha^{-1} refers to the lime requirement for a (theoretical) BS% of 110. The dolomitic limestone used contained 207 g kg^{-1} calcium, 114 g kg^{-1} magnesium, and 90 % effective calcium

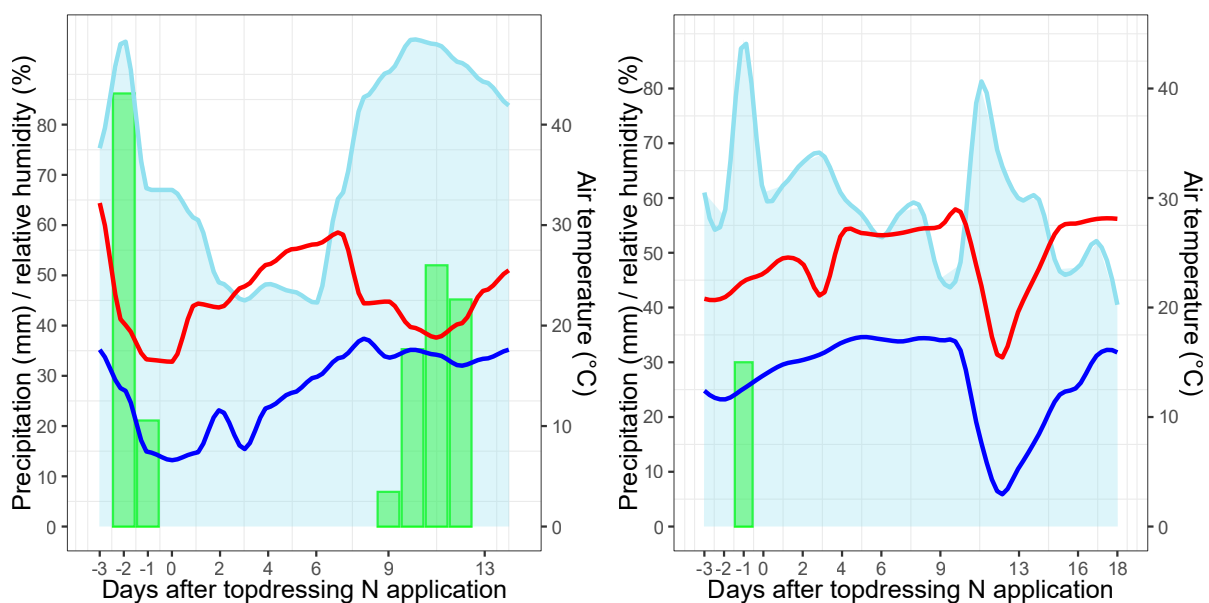


Figure 1. Rainfall, relative humidity, and maximum and minimum temperatures throughout the evaluation periods starting at 24 (left panel) and 36 (right panel) months after liming. The red lines represent the maximum air temperatures, while the blue lines represent the minimum air temperatures.

carbonate equivalent. Lime rates were calculated on a dry basis. After surface liming, plots were cultivated in a soybean-second-crop corn rotation. Subplots were assigned to four N fertilizers, applied at a rate of 100 kg ha⁻¹, and evaluated exclusively within the NH₃ volatilization chambers during two second-crop corn growing seasons. The first application occurred 24 months after liming, and the second, 36 months after liming. In the remaining main plot area (outside the chambers), corn also received 100 kg ha⁻¹, but solely as urea stabilized with NBPT (46 % N), applied at the V4 phenological stage. This procedure was adopted because the experiment was part of a long-term liming trial, and the application of EEFs to the entire main plot could have affected corn yield and soil chemical properties. The long-term results of this trial have been published by Minato et al. (2023a). At the subplot level, the N fertilizers were: conventional urea (46 % N), urea coated with copper and boron (Ur-Cu+B; 0.15 % Cu, 0.4 % B, and 44 % N), NBPT-treated urea (Ur-NBPT, 46 % N and 0.053 % NBPT), and ammonium sulfate (AMS, 21 % N and 22 % S). Nitrogen fertilizers were applied after 20 mm of rainfall.

Capture and determination of volatilized NH₃-N

The capture of volatilized NH₃-N was initiated shortly after N fertilizers were applied to the soil. For this, a semi-static open chamber was constructed using polyethylene terephthalate bottles (Araújo et al., 2009; Figure 2). Each chamber had an area of 0.007854 m². Chambers were placed at the center of each experimental unit and were rotated over three points after each sampling to minimize interference from environmental factors. Chambers contained a strip of filter paper (2.5 cm wide and 25 cm long) that captured the volatilized NH₃-N. The base of the strip was immersed in 20 mL of a solution containing 0.05 mol L⁻¹ sulfuric acid (H₂SO₄) and 2 % (v/v) glycerin in a 50 cm³ flask.

Samplings were carried out on days 3, 5, 7, 9, and 13 after N fertilizer application for evaluations performed at 24 months after liming and on days 2, 4, 6, 9, 13, 16, and 18 after N fertilizer application for evaluations performed at 36 months after liming. After each collection, the filter paper strips and the H₂SO₄ + glycerin solution were replaced, and this procedure was repeated until NH₃-N losses stabilized. For the extraction, the filter paper, along with the remaining H₂SO₄ and glycerin solution, was immersed in a container with 25 mL of distilled water. Subsequently, the N derived from volatilized NH₃ was indirectly quantified as NH₄⁺ by UV/Vis spectrophotometry, according to the method described by Bower and Holm-Hansen (1980). This methodology is widely employed

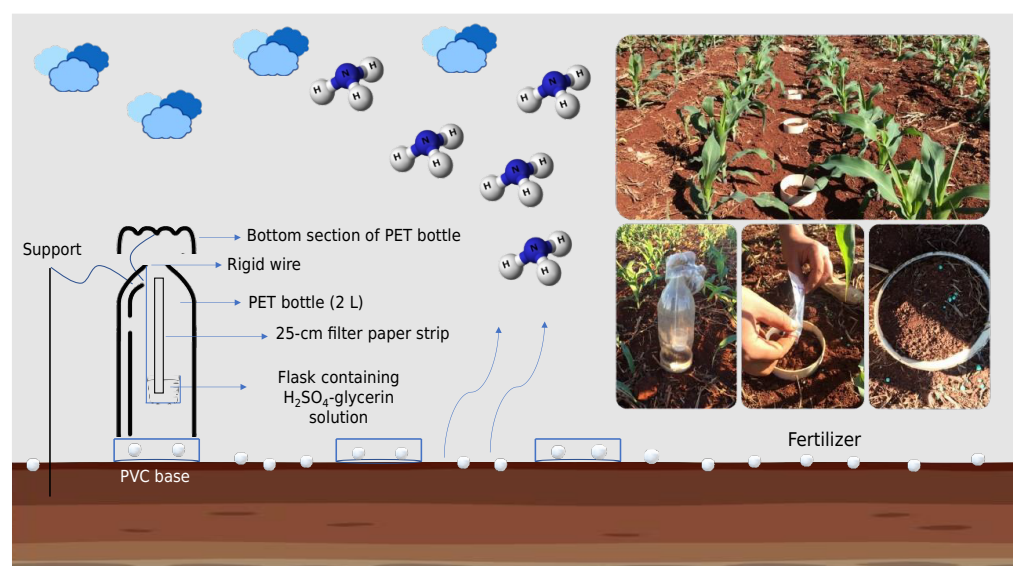


Figure 2. Schematic representation and representative images of the semi-static chamber used for capturing ammonia (NH₃) volatilized from nitrogen fertilizers.

in field experiments to estimate NH_3 volatilization, as reported by Minato et al. (2020, 2023b), Cassim et al. (2021, 2022), and Besen et al. (2022). Thirty-six months after liming, soil samples were collected from within each chamber at a depth of 0.00-0.05 m and subjected to pH analysis. Soil pH was determined in 0.01 mol L^{-1} CaCl_2 (soil/solution ratio of 1:2.5), as described by Pavan et al. (1992).

Calculation and statistical analysis

Data on NH_3 -N volatilization at 24 and 36 months after surface liming were subjected to nonlinear regression analysis (Silva et al., 2017; Minato et al., 2020; Souza et al., 2023; Soares and Cantarella, 2023; Lisboa et al., 2024). The model with the lowest Akaike information criterion (AIC) was selected (Akaike, 1974). The logistic model (Equation 1) includes three parameters (α , β , and γ), as described by Seber and Wild (2003).

$$\hat{Y} = \frac{\alpha}{1 + \exp[-(t-\beta)/\gamma]} \quad \text{Eq. 1}$$

in which: $\hat{Y}$ represents the quantity of N volatilized in the form of NH_3 -N (kg ha^{-1}) at time t (days); α represents the maximum cumulative volatilization; β represents the time at which the cumulative loss reaches 50 %, corresponding to the curve inflection point (day of the maximum daily loss of NH_3 -N); and γ is a parameter used to calculate the maximum daily loss (MDL) of NH_3 -N, as shown in equation 2.

$$\text{MDL} = \frac{\alpha}{4\gamma} \quad \text{Eq. 2}$$

Data regarding total NH_3 -N volatilization and soil pH were subjected to the Shapiro-Wilk normality test. The homogeneity of variances was assessed graphically. Given that the assumptions were met, the data were subjected to analysis of variance ($p < 0.05$). Regardless of overall significance, the interactions between lime rates and N fertilizer sources were further analyzed to identify more specific responses. When significant, quantitative factors were evaluated using polynomial regression, while qualitative factors were compared using Tukey's test ($p < 0.05$).

RESULTS

Effects of surface liming on NH_3 -N volatilization losses

Cumulative NH_3 -N volatilization followed a sigmoidal pattern. Losses increased gradually from the beginning of the experiment until the day the maximum daily loss was achieved, then stabilized (Figures 3 and 4). According to the adjusted logistic model, the maximum cumulative loss (α) varied according to N source, lime rate, and time after liming (Table 1). Regardless of the evaluation period and lime rate, treatments were found to promote the following descending order of cumulative NH_3 -N loss: urea > Ur-Cu+B > Ur-NBPT > AMS. An exception was observed 36 months after the application of 5.4 Mg ha^{-1} lime: the cumulative loss promoted by urea did not differ significantly from that induced by Ur-Cu+B (Figures 3 and 4, and Table 1).

Estimation of β revealed that Ur-NBPT was efficient in delaying the maximum NH_3 -N volatilization compared to urea. The peak was delayed by 1 and 4 days at 24 and 36 months after liming, respectively. The partitioning of the analysis of variance for total NH_3 -N volatilization revealed significant effects of N fertilizers within each lime rate, as well as significant effects of lime rates within each N source, at 24 and 36 months after liming (Table 2). The increase in volatilization was linear for all N fertilizers, except for Ur-Cu+B at 36 months after liming. For every 1 Mg ha^{-1} increase in applied lime, total NH_3 -N volatilization increased by 1.7 to 2.2 % in urea, 1.6 to 5.1 % in Ur-Cu+B, 1.5 to 1.3 % in Ur-NBPT, and 0.9 to 0.7 % in AMS at 24 and 36 months after liming, respectively (Figure 5).

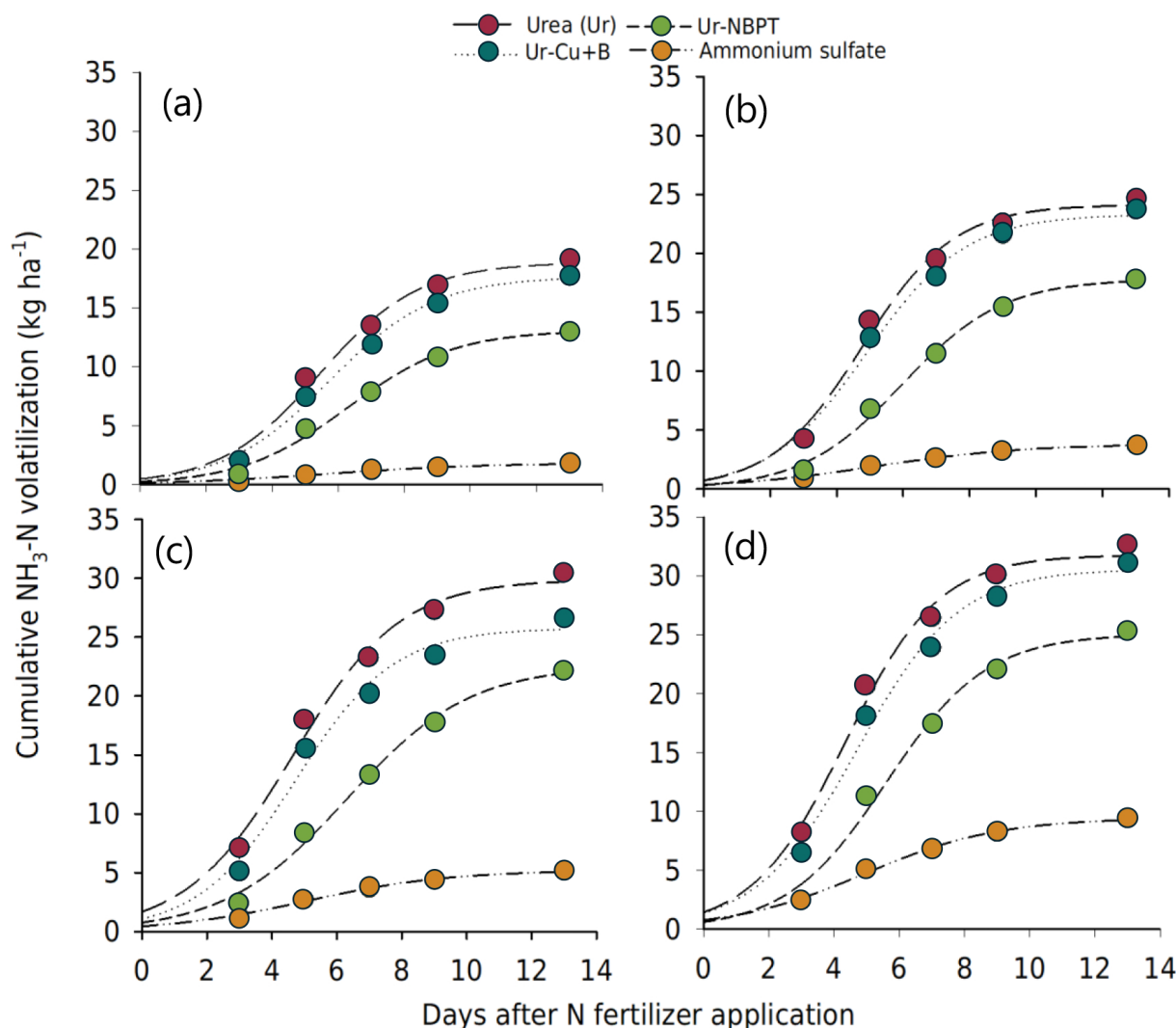


Figure 3. Cumulative ammonia (NH₃-N) volatilization loss from no-till under different nitrogen fertilization treatments ($n = 4$), as evaluated at 24 months after surface lime application at a rate of (a) 0.0, (b) 2.6, (c) 5.4, and (d) 8.1 Mg ha⁻¹. Fitted model parameters are shown in table 1.

At 24 months after application of the highest lime rate, the increase in NH₃-N loss compared to the unlimed control was 68, 72, 92, and 350 % in urea, Ur-Cu+B, Ur-NBPT, and AMS treatments, respectively (Table 1). Similarly, 36 months after application of the highest rate, the increase in NH₃-N loss was 100, 158, 171, and 2300 % in urea, Ur-Cu+B, Ur-NBPT, and AMS, respectively (Table 1).

Among the N fertilizers evaluated, AMS resulted in the lowest total NH₃-N volatilization means across all lime rates (Table 3). At 24 months after liming, Ur-NBPT significantly reduced volatilization compared to conventional urea only at the 5.4 and 8.1 Mg ha⁻¹ lime rates. However, at 36 months after liming, Ur-NBPT was effective in reducing volatilization losses at all lime rates. Overall, Ur-Cu+B did not differ significantly from conventional urea.

Effect of surface liming and N fertilization on topsoil pH

The increase in lime rate led to a rise in soil pH in the 0.00–0.05 m layer (Figure 6), which varied according to N source. For all lime rates, plots fertilized with AMS exhibited the greatest reductions in soil pH.

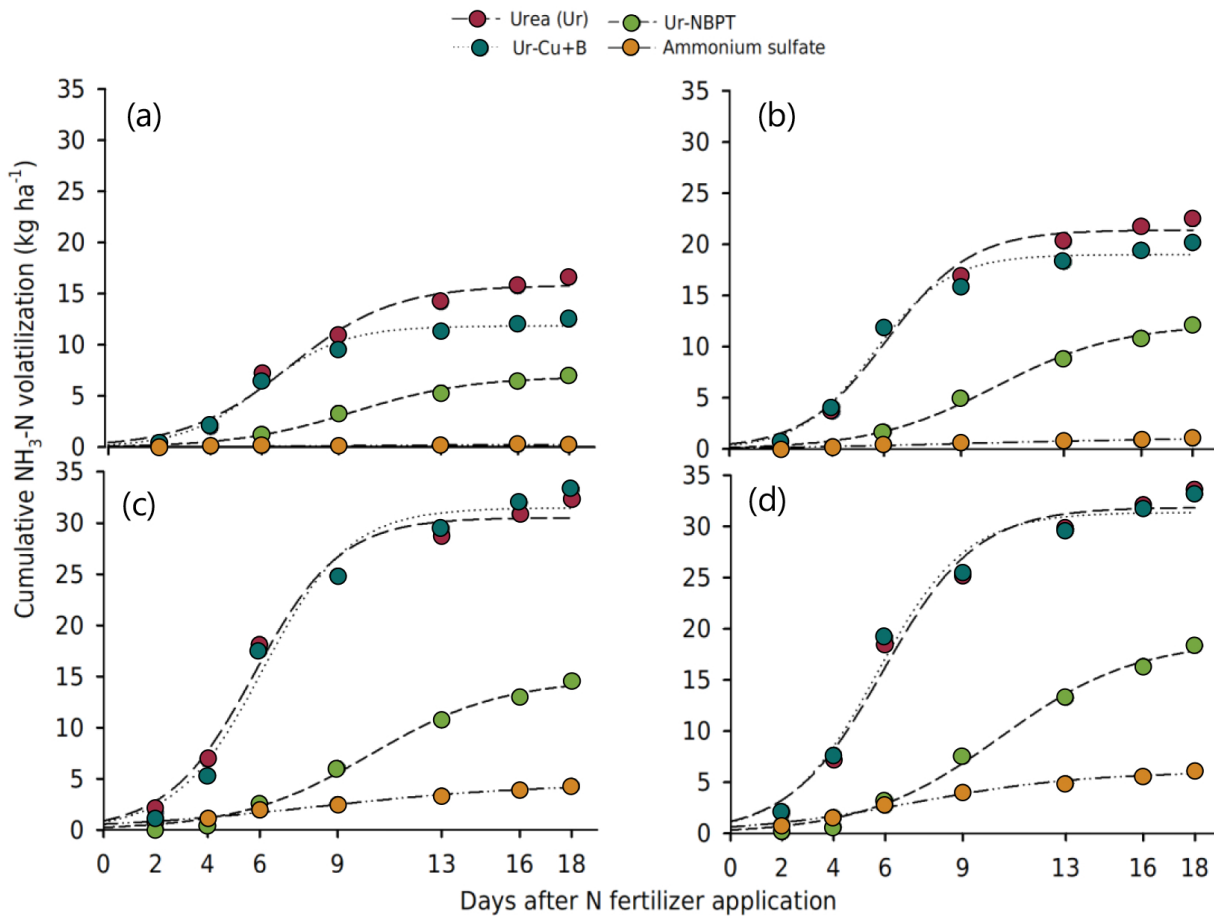


Figure 4. Cumulative ammonia ($\text{NH}_3\text{-N}$) volatilization loss from no-till under different nitrogen fertilization treatments ($n = 4$), as evaluated at 36 months after surface lime application at rates of (a) 0.0, (b) 2.6, (c) 5.4, and (d) 8.1 Mg ha^{-1} . Fitted model parameters are shown in table 1.

DISCUSSION

This study modeled the relationship of cumulative $\text{NH}_3\text{-N}$ loss as a function of lime rate and time at two evaluation periods (24 and 36 months after liming). The findings demonstrated that alternative N sources may contribute to enhancing N fertilizer utilization by crops grown on tropical soils under NT. Overall, the lower values of maximum cumulative volatilization (parameter α) achieved by other N sources underscore the advancement of urea-based EEFs. In general, EEFs act by reducing N losses in the soil and extending its availability to plants. This is achieved through mechanisms that slow down the transformation, solubilization, or release of the nutrient (Cassim et al., 2024b). In tropical and subtropical regions, which usually have more acidic soils, the magnitude of loss reduction depends on the applied rate and adopted technology (Otto et al., 2017; Cassim et al., 2021). Another important factor is rainfall after application, which facilitates the timely solubilization of N fertilizer (Viero et al., 2014).

Among urea-based fertilizers, Ur-NBPT proved to be the most efficient in mitigating NH_3 volatilization. After application to soil, NBPT is transformed into its oxygen analog NBPTO, which forms a tridentate bond with urease active sites, blocking the enzyme and thereby reducing urease activity (Manunza et al., 1999). On the other hand, Ur-Cu+B had little effect in mitigating NH_3 volatilization losses. Micronutrients such as Cu and B can temporarily inhibit urease. Copper promotes noncompetitive inhibition by binding to urease sulfhydryl groups (Shaw, 1954; Cantarella et al., 2018). In the case of B, the molecular conformation of boric acid (H_3BO_3) resembles that of urea, allowing it to bind

to urease at the same enzyme sites (Benini et al., 2004). The similar efficiency of Ur-Cu+B to urea is likely due to the low concentrations of B and Cu in the coating layer, which are not sufficient to mitigate NH_3 volatilization. For example, in a study conducted by Cassim et al. (2024c), urea coated with B in the form of 0.8 % H_3BO_3 reduced volatilization losses by 25 % in relation to urea.

Table 1. Parameters of the logistic model estimating the cumulative loss, maximum daily loss, and percentage increase in cumulative ammonia ($\text{NH}_3\text{-N}$) loss for each combination of nitrogen (N) fertilizer and lime rate

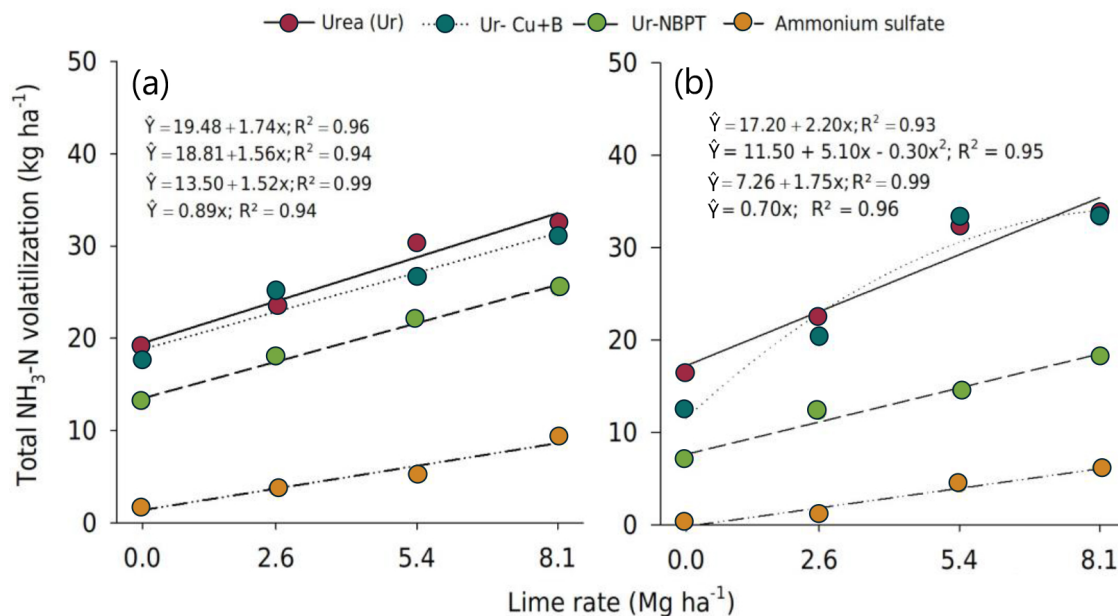
N fertilizer	Lime rate	Estimated parameters			MDL	Increase in cumulative $\text{NH}_3\text{-N}$ loss ⁽¹⁾
		α	γ	β		
	Mg ha ⁻¹	kg ha ⁻¹ $\text{NH}_3\text{-N}$	day	kg ha ⁻¹ day ⁻¹ $\text{NH}_3\text{-N}$		%
24 months after surface lime application						
Urea (Ur)	0	19	0.67	5	7	-
Ur-Cu+B	0	18	0.63	6	7	-
Ur-NBPT	0	13	0.62	6	5	-
AMS	0	2	0.42	6	1	-
Ur	2.6	23	0.68	5	9	21
Ur-Cu+B	2.6	24	0.73	5	8	33
Ur-NBPT	2.6	18	0.66	6	7	38
AMS	2.6	4	0.44	5	2	100
Ur	5.4	30	0.60	5	12	58
Ur-Cu+B	5.4	26	0.67	5	10	44
Ur-NBPT	5.4	23	0.53	6	11	77
AMS	5.4	5	0.46	5	3	150
Ur	8.1	32	0.70	4	11	68
Ur-Cu+B	8.1	31	0.65	5	12	72
Ur-NBPT	8.1	25	0.65	6	10	92
AMS	8.1	9	0.49	5	5	350
36 months after surface lime application						
Ur	0	16	0.50	7	8	-
Ur-Cu+B	0	12	0.64	6	5	-
Ur-NBPT	0	7	0.44	10	4	-
AMS	0	0.25	0.33	8	0.2	-
Ur	2.6	21	0.61	6	9	31
Ur-Cu+B	2.6	19	0.70	6	7	58
Ur-NBPT	2.6	12	0.42	10	7	71
AMS	2.6	1	0.23	9	1	300
Ur	5.4	31	0.60	6	13	94
Ur-Cu+B	5.4	31	0.60	6	13	158
Ur-NBPT	5.4	15	0.40	10	9	114
AMS	5.4	5	0.24	8	5	1900
Ur	8.1	32	0.55	6	14	100
Ur-Cu+B	8.1	31	0.59	6	13	158
Ur-NBPT	8.1	19	0.38	10	12	171
AMS	8.1	6	0.30	7	5	2300

⁽¹⁾ Compared to the same unlimed N fertilizer within the same year. MDL; maximum daily loss ($\text{MDL} = \frac{\alpha}{4\gamma}$); AMS: ammonium sulfate; α : maximum cumulative volatilization; β : time at which 50 % of the losses occur, corresponding to the curve inflection point; γ : parameter of the equation used to calculate the MDL.

Table 2. *P*-values for the effects of factors on total NH₃-N volatilization at 24 and 36 months after liming

Sources of variation	24 months after liming	36 months after liming
Block	-	-
Liming (L)	<0.001	<0.001
Error (L)	-	-
N fertilizer (N)	<0.001	<0.001
N × L	0.525	<0.001
N/L ₀	<0.001	<0.001
N/L ₁	0.046	<0.001
N/L ₂	<0.001	<0.001
N/L ₃	<0.001	<0.001
L/N ₁	<0.001	<0.001
L/N ₂	<0.001	0.045
L/N ₃	<0.001	<0.001
L/N ₄	<0.001	<0.001
Error (N)	-	-

L₀: 0 Mg ha⁻¹ of lime; L₁: 2.6 Mg ha⁻¹ of lime; L₂: 5.4 Mg ha⁻¹ of lime; L₃: 8.1 Mg ha⁻¹ of lime; N₁: Ur-Cu+B; N₂: AMS; N₃: Ur-NBPT; N₄: Ur.


Figure 5. Total ammonia (NH₃-N) volatilization loss as a function of lime rate under different nitrogen fertilization treatments (*n* = 4) at (a) 24 and (b) 36 months after liming.

In a recent study, Ur-Cu+B was effective in minimizing NH₃ volatilization in a tilled Oxisol, reducing losses by 23 %; however, no effect was observed on soil subjected to surface liming (Besen et al., 2022). These findings corroborate those of the current study and agree with the review by Cantarella et al. (2018), who reported that NH₃ volatilization mitigation with H₃BO₃ and Cu is inconsistent. Some studies indicated a reduction in NH₃ volatilization (Cancellier et al., 2016; Dominghetti et al., 2016), whereas others did not observe significant effects (Faria et al., 2014; Ribeiro et al., 2020).

The lower β values at 24 months (compared to 36 months on average) after surface liming suggest that a residual effect of the pH increase may have contributed to the acceleration of volatilization. Studying the effect of lime rates on N losses from urea through NH₃-N volatilization, Minato et al. (2023b) also observed an earlier peak in the evaluation year closest to surface liming.

Over the months, however, the increase in pH resulting from surface liming becomes less prominent, owing to the dissolution and degradation of limestone (Minato et al., 2023a). Thus, at 36 months after surface application, the limestone's residual effect was possibly less pronounced. Furthermore, the climatic conditions at 36 months may have delayed volatilization, helping to explain the differences between evaluation periods. Under dry soil conditions, urease hydrolysis rates are reduced, leading to lower volatilization losses (Das et al., 2024).

Table 3. Means of total $\text{NH}_3\text{-N}$ volatilization of N fertilizers within each liming rate at 24 and 36 months after liming

N fertilizer	Ammonia volatilization			
	0 Mg ha^{-1}	2.6 Mg ha^{-1}	5.4 Mg ha^{-1}	8.1 Mg ha^{-1}
kg ha^{-1}				
24 months after liming				
AMS	1.77 b	3.71 b	5.12 c	9.36 c
Ur-NBPT	13.06 a	17.84 a	22.15 b	25.36 b
Ur-Cu+B	17.68 a	23.67 a	26.66 ba	31.12 ba
Ur	19.13 a	24.74 a	30.45 a	32.57 a
36 months after liming				
AMS	0.24 c	1.01 c	4.27 c	6.10 c
Ur-NBPT	7.03 b	12.13 b	14.54 b	18.35 b
Ur-Cu+B	12.46 ba	20.13 a	32.30 a	33.16 a
Ur	16.58 a	22.46 a	33.26 a	33.61 a

Different lowercase letters in the same column indicate differences among N fertilizer means according to Tukey's test ($p < 0.05$).

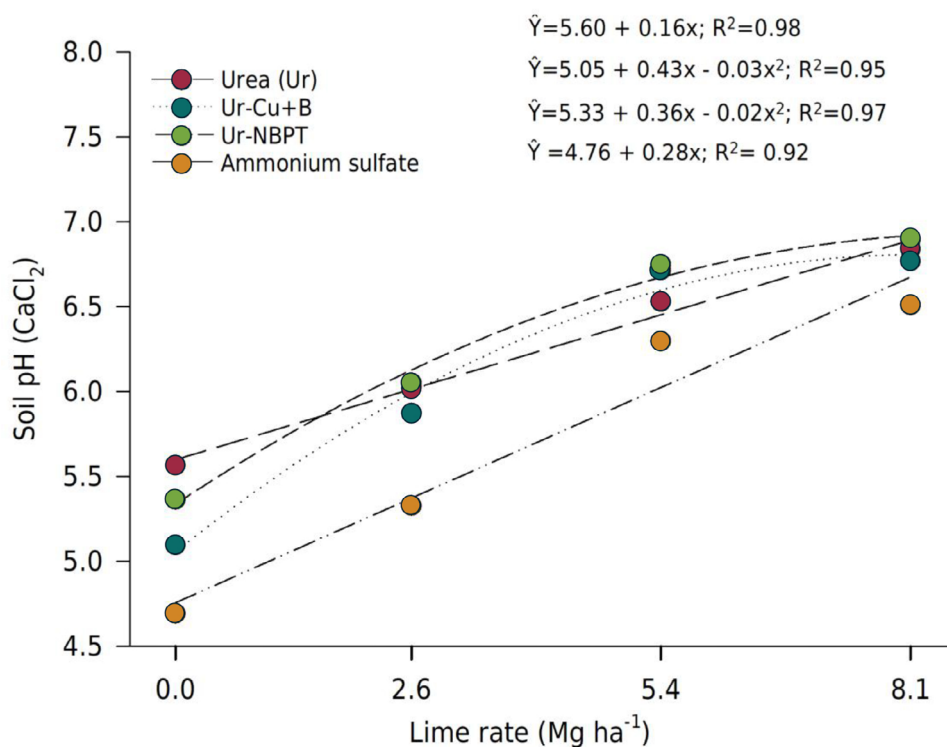


Figure 6. Soil pH in the 0.00-0.05 m layer as a function of lime rate under different nitrogen fertilization treatments ($n = 4$), 36 months after liming.

Liming enhances N losses via volatilization because it elevates the soil pH, as observed at 36 months after surface liming. Similarly, in an experiment conducted with soils from the state of Paraná, Cassim et al. (2024a) observed an increase in total NH_3 volatilization from EEFs when these sources were applied in combination with surface limestone and straw, simulating no-till conditions. Our finding is consistent with reports by Mandal et al. (2016) and Sha et al. (2019), who documented an increase in NH_3 volatilization losses with increasing soil pH. At higher soil pH, the rate of urea hydrolysis increases, leading to the formation of NH_3 and predisposing N to volatilization losses (Sengik et al., 2008). Furthermore, due to the increased pH, the equilibrium between NH_4^+ and NH_3 in the soil shifts toward NH_3 . This phenomenon is especially important in low-CEC soils, whose properties favor N losses via volatilization. For example, these soils have lower NH_4^+ adsorption power and lower capacity to resist the pH changes (buffering capacity) induced by liming and urea hydrolysis (Sunderlage and Cook, 2018).

In the case of the clayey *Latosso* (Rhodic Eutruxox) studied here, AMS had the lowest NH_3 losses after liming. In soils with pH below 7 and with AMS added, NH_4^+ remains in an ionic, stable form, whereas urea is enzymatically hydrolyzed to ammoniacal N, a process that increases pH in the granulosphere and, subsequently, leads to NH_3 volatilization (Cassim et al., 2024a). In both evaluation periods, total volatilization in AMS plots did not exceed 10 % of the applied N at any of the studied rates, in agreement with the findings of Powlson and Dawson (2022). The authors summarized data from 41 scientific publications and observed that, in soils with pH in water <7 , most N losses were <5 %. In soils with pH in water >7 , AMS application led to losses of 15–35 % of the applied N.

On the other hand, at the end of the experiment, it was observed that AMS resulted in the greatest reduction in topsoil pH at 36 months in plots without liming. This effect results from the nitrification process of ammoniacal N, in which, for each mole of NH_4^+ oxidized, two moles of H^+ are released (Cassim et al., 2024b). Furthermore, Fageria et al. (2010) suggest that during nitrate (NO_3^-) leaching, the removal of basic cations as accompanying ions may also contribute to acidification, since these cations are subsequently replaced by H^+ in the exchange complex. The less pronounced acidifying effect of urea-based sources is explained by their lower acidity index (Pauletti and Motta, 2019). Consistent with these results, Chien et al. (2008) reported that the liming requirements of soils fertilized with AMS may be 1.4 to 2.3 times those of soils receiving ammonium nitrate or urea to neutralize soil acidity.

The efficiency of N fertilizers in mitigating NH_3 volatilization losses may vary depending on the liming method used, particularly whether lime is surface-applied or incorporated into the soil (Besen et al., 2022). Given the well-known benefits of applying lime without mechanical incorporation in tropical and subtropical fields under NT, this liming method should be preferably maintained. Finally, even when applying lime at rates consistent with the regional recommendation for this study (2.6 Mg ha^{-1} to raise BS% to 70) (Pauletti and Motta, 2019), we still observed increases in cumulative NH_3 -N losses. Overall, considering the linear increase in total NH_3 -N volatilization with higher lime rates, our results suggest that surface applications above the recommended rate may enhance NH_3 -N losses from the N sources evaluated. This was evident with the 8.1 Mg ha^{-1} rate. It resulted in 32 and 50 % greater NH_3 losses from urea at 24 and 36 months after liming (Table 3), respectively, compared with the 2.6 Mg ha^{-1} rate.

Future studies should investigate the response of NUE to these amendment practices, given that, depending on the N source and lime rate, liming can significantly increase N losses, potentially reducing NUE. The use of specific tools, such as isotope labels (e.g., ^{15}N), can better qualify assessments of N fertilizer dynamics in soil-plant-atmosphere systems, contributing to the development of more sustainable N fertilization strategies.

CONCLUSION

Surface liming under NT increases topsoil pH, thereby enhancing $\text{NH}_3\text{-N}$ volatilization. For urea, losses reached up to 32 % of the applied N and increased with higher lime rates. Under the conditions of this study, the results indicate that surface applications of lime at rates exceeding those recommended by the BS% method can intensify $\text{NH}_3\text{-N}$ volatilization losses from N fertilizers.

Our findings also indicate that ammonium-based N sources, such as AMS, and EEFs, such as urea treated with NBPT (Ur-NBPT), should be preferred over conventional urea due to their greater effectiveness in reducing total volatilization and delaying the volatilization peak following liming under NT. However, when using AMS, the greater potential for long-term soil acidification must be considered.

DATA AVAILABILITY

All data were generated or analyzed in this study.

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