Colegio de
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Kinetics of ammonium volatilization in the form of ammonia in soils through a textural gradient

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ABSTRACT

Objective: To estimate the ammonia volatilization rate, using a texture gradient with increasing doses of ammonium in alkaline soils.

Design/Methodology/Approach: The experiment took place in the Análisis Químico de Suelos lab of the Soil Department of the Universidad Autónoma Chapingo. The incubations consisted of 25 g of soil from the former Texcoco lake, with 12.5% clay. The soil was air-dried and sieved with a no. 10 mesh. The soil was mixed with three different concentrations (17.5%, 22.5%, and 32.5%) of bentonite, in order to increase clay content. The mixtures were placed in plastic containers with airtight seals. Twelve-point five mL of an ammonium sulfate solution (with 150, 300, 450, 600, and 750 mg of nitrogen kg soil^{-1}) was added. The ammonia was recovered in a container with a boric acid solution. Volumetry was used to quantify ammonium. A completely randomized design with two factors (clay content and ammonium dose) was used. Data were analyzed with a regression analysis, analysis of variance, and Tukey Multiple Comparison Test, using the SAS OnDemand for Academics software.

Results: The ammonia volatilization has a linear trend, with the concentration of the applied nitrogen. The volatilization rate ranged from 0.02 to 0.03 mg of ammonium per milligram of the nitrogen applied per kg soil^{-1}. Significant statistical differences were recorded between the effect of the N dose and the clay content on the ammonia volatilization rate.

Study Limitations/Implications: Clay content in the soil and ammonia volatilization rate can be used as an indicator to estimate ammonia losses and the ammonia adsorption capacity of the soil.

Findings/Conclusions: Ammonia volatilization is independent from clay content and takes place immediately after its application. It has a linear trend regarding the ammonia dose applied. Ammonia volatilization rate decreases (asymptotic trend) as the clay content increases in the soil.

Keywords: clay, ammonium, alkaline, cationic exchange capacity.

INTRODUCTION

The nitrogen (N) available for plants in the soil is the highest limiting factor (Martínez-Dalmau *et al.*, 2021). Consequently, during the last decade, the annual use of nitrogen fertilizers has increased by ≈1.4% worldwide; however, their recovery efficiency is <50% (Panday *et al.*, 2020; Mahmud *et al.*, 2021). Approximately 80% of the N used in agriculture is lost as a result of leaching, runoff, and gaseous emissions. The predominant gases are nitrous oxide (N_2O-N) and ammonia (NH_3-N), with 4 Tg N year^{-1} and 12 Tg N year^{-1}, respectively (Krol *et al.*, 2020). The NH_3-N volatilization accounts for 10-60% of the total N applied as ammonium fertilizer. This process takes place a few days after the application (Chu *et al.*, 2020).

The NH_3-N volatilization is the result of the ammonium (NH_4-N) adsorbed by the colloids and its interaction with the physical and chemical properties of the soil (Wang *et al.*, 2023). Volatilization is highly sensitive to pH and NH_4-N concentration. The increases of pH promote the formation of NH_3-N (Shi *et al.*, 2024) —*i.e.*, a >7 pH increases the formation of NH_3-N, as a consequence of the high concentration of the hydroxyl radical (HR) that reacts with the NH_4-N (Skorupka and Nosalewicz, 2021). This dependency between pH and the NH_4-N/NH_3-N balance (Pelster *et al.*, 2019) increases exponentially the formation of NH_3-N (Ohnemus *et al.*, 2021). In calcareous soils, the losses account for 50% (Powlson and Dawson, 2021).

Clays, cation exchange capacity (CEC), texture, organic matter (Cassity-Duffey *et al.*, 2020), and damping capacity of soils determine the NH_3-N volatilization rate, because they correlate with higher NH_4-N adsorption rates. Clay soils have a lower volatilization than sandy soils (Pelster *et al.*, 2019; Mazloomi and Jalali, 2019): the amount of N lost in the volatilization is significantly influenced by soil texture and it is higher in coarse-textured soils than in fine-textured soils (Al-Badrani *et al.*, 2023). The NH_4-N adsorption and desorption in the soil is highly dependent of the type and quantity of clays; these minerals have chemical reactivity and are grouped according to their silicon tetrahedra (1:1) and aluminum octahedra (2:1). The 2:1 type clays have a high CEC and a higher specific surface than the 1:1 type clays, increasing their NH_4-N adsorption (350-3,800 kg ha^{-1} at ≤ 30 cm of depth); vermiculites, illites, and smectites predominate in these soils (Kome *et al.*, 2019). Consequently, the objective of this research was to estimate the ammonia volatilization rate, using a textural gradient and increasing doses of ammonium in alkaline soils.

MATERIALS AND METHODS

The experiment was carried out in the Análisis Químico lab of the Soil Department of the Universidad Autónoma Chapingo. To determine the volatilization rate, soils that had been previously air-dried and sieved with a no. 10 mesh were incubated.

Increasing concentrations of bentonite were added to the soil samples (texture gradient) to increase clay content. Bentonite is an expandible clay of the montmorillonite group (2:1) that has strong affinity and cation adsorption capacity in its internal and external layers (specific surface) (Alexander *et al.*, 2019). Bentonite has the following characteristics: $pH_{10:1} = 9.42$, $EC_{10:1} = 1.36$ dSm^{-1}, CEC$=63.23$ meq 100 g^{-1} S, carbonates$=1.5$ mmol$_c$ L^{-1}, bicarbonates$=82.49$ mmol$_c$ L^{-1}, and clay (particles>0.002 mm)$=95.84$%. Adding this mineral allowed the simulation of soils with higher contrasts regarding clay content (17.5%, 22.5%, and 32.5%). Soil without bentonite was used in the case of the 12.5% concentration. The soil samples collected from the former Texcoco lake had the following characteristics: 12.5% clay obtained with the pipette method (Van Reeuwijk, 2003); 9.6 pH, 389 μScm^{-1} EC, and 32.0 mg kg^{-1} phosphorous (SEMARNAT, 2002); 8.78 Cmol kg S^{-1} CEC (Pleyser *et al.*, 1986); and 1.60 g kg^{-1} soluble organic carbon (Harvey *et al.*, 2009). Once the sample and the bentonite were mixed, 25 g were weighted and placed in 15 cm $\times$ 20 cm transparent polypropylene bags with airtight seals. An ammonium sulfate solution (12.5 mL) was added to the bags. This solution was

determined based on the effective porosity saturation (Wallach, 2019); the percentage used in this experiment was 50% (W/V).

The N doses applied were based on the proposals of Liu *et al.*, 2007 and Fan *et al.*, 2011. Ammonium sulfate was used as source of N (150, 300, 450, 600, and 750 mg N kg soil^{-1}). A modified version of the quantification methodology proposed by Ahmed *et al.* (2008), Chen *et al.* (2014) and Palanivell *et al.* (2015) for the recovery of ammonium was used: a 100 mL container with a 50 mL boric acid solution (20 g L^{-1}) was mixed with the bromocresol green and methyl red indicators and placed in the airtight sealed bag with the soil sample (previously treated with ammonium sulfate). Finally, the bag was airtight sealed to prevent water loss and gas and it was incubated at room temperature for three days. Volumetry was used to determine the amount of ammonium recovered from the boric acid solution (Shen *et al.*, 2020); in addition, the sulfuric acid concentration was adjusted to 0.005 N. The experiment was carried out using a completely randomized design with two factors (NH_4-N concentration and clay content of the soil) and three replicates. The analysis of the volatilization rate was carried out with a simple linear regression analysis. The NH_4-N doses were analyzed per clay level. The linear regression analysis determined the NH_3-N volatilization rate. Subsequently, a graph of these volatilization was developed per clay level, in order to evaluate the influence of clay content on the NH_3-N volatilization. The recovered NH_3 (mg) was subjected to an analysis of variance and to a Tukey Multiple Comparison Test, with the SAS OnDemand for Academics software.

RESULTS AND DISCUSSION

Effect of the clay content on the soil and the NH_4-N dose on the NH_3-N volatilization
According to the analysis of variance and the regression analysis for the five NH_4-N levels in each clay layer, when $\alpha=0.01$ (Table 1), the behavior of the NH_3 volatilization has a proportional linear trend as the NH_4-N levels applied increase, given the positive correlation between NH_3-N emissions and NH_4-N concentration (Li *et al.*, 2020).

After the samples had been incubated for three days, a linear trend was identified between the concentration of NH_4-N applied and the amount of NH_3-N recovered, regardless of the clay content; this linear behavior is found in each clay layer evaluated, because high NH_4-N doses increase NH_3-N emissions (Wan *et al.*, 2021). The NH_3-N volatilization rate ranged from 0.02 to 0.03 mg per mg of N applied per kg of soil, with

Table 1. Analysis of variance of linear regression for four soil clay contents and five NH_4-N doses on NH_3-N volatilization.

| Source	DF	Sum of squares			
		12.5	**17.5**	**22.5**	**32.5**
		Clay (%)			
Reg	1	500.89**	430.56**	331.95**	282.87**
Error	13	0.48	0.57	0.61	0.69
Total	14				

DF: Degrees of Freedom; *$\alpha=0.05$; **$\alpha=0.01$; NS: Not Significant.

>97% correlation coefficients (Figure 1). Minato *et al.* (2020) mention that the accumulated NH_3-N volatilization has a sigmoid pattern with gradual increases. Its first stage had a high NH_3-N emission rate, reaching its maximum point 72 h after the application (0.4 kg ha^{-1} day^{-1} NH_3-N). It remained constant during the first 5 days.

The NH_3-N losses recorded during the incubation with the various N doses (150, 300, 450, 600, and 750 mg·kg $soil^{-1}$) suggest that, under experimental conditions, this situation takes place immediately after application and that, during the first stages, the soil lacks the capacity to absorb NH_4-N from the clay. Viero *et al.* (2014) report a similar trend: a maximum loss of 0.40 and 0.53 kg ha^{-1} day^{-1} was registered 5-10 days after the application. Dari and Rogers (2022) determined that maximum volatilization takes place from 4 to 8 days after the application (8 to 11% of the total N applied). These emissions are generated by the reactivity of NH_4-N to the OH^- content in the soil —*i.e.*, the post-application volatilization is mostly a chemical process, since the increase from 6 to 10 in pH increases the anionic association sites that react to NH_4-N, reducing the NH_4-N concentration and, in turn, the adsorption capacity of soil clays (Fan *et al.*, 2021).

The analysis of variance found significant differences on the effect on the NH_3-N volatilization of clay content, NH_4-N doses, and the interaction between clay content and NH_4-N doses (Table 2). Da Cruz Corrêa *et al.* (2021) established that an increase in the N doses results in a linear increase of volatilization, irrespective of the ammonium source. The NH_4-N adsorption increases as clay content in the soil increases, given the negative correlation between the NH_3-N volatilization, the clay content in the soil, and the CEC (Chuong *et al.*, 2020).

NH_3 volatilization is a chemical reaction determined by the pH of the soil; however, the clay content in the soil tends to diminish volatilization, since the ammonium applied in alkaline soils and soils with many clay layers or high CEC levels is absorbed by the sites where these clays are exchanged (Wester-Larsen *et al.*, 2022).
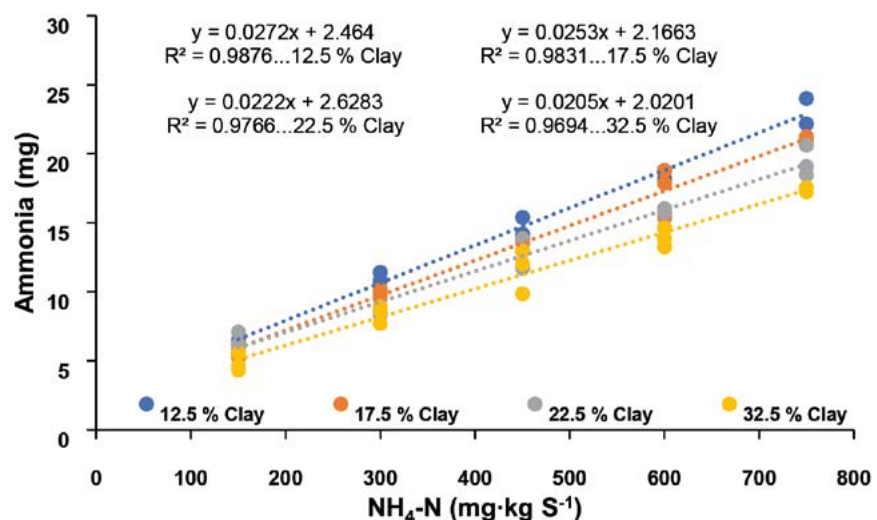
Figure 1. Linear regression model for the effect on ammonia volatilization of five levels of ammonium concentration and four levels of clay concentration found in the soil.

Table 2. Analysis of variance of the effect of four soil clay contents and five NH_4-N doses on NH_3-N volatilization.

Source	DF	SS (NH_3 mg)	F_c
Soil clay content (A)	3	98.9925	53.73**
Doses of ammonium sulphate (B)	4	1528.1210	622.03**
AB	12	23.0417	3.13**
Error	40	24.5667	
Total		1674.7218	

DF: Degrees of Freedom; SS: Sum of Squares; *α=0.05; **α=0.01; NS: Not Significant.

The multiple comparison of means determined that a greater clay content in the soil diminished NH_3-N volatilization. Likewise, statistical differences were found for each level evaluated (Table 3). An increase in the clay content of the soil increases cation exchange sites, enhancing the capacity of the soil to adsorb NH_4-N. Foereid *et al.* (2019) point out that the application of 10 t ha^{-1} zeolite increases NH_4 adsorption, without limiting the amount available for the crop.

Each level of the NH_4-N doses applied had statistical differences regarding NH_3-N volatilization. The 750 mg NH_4-N kg soil^{-1} dose generates the largest volatilized NH_3-N; likewise, there is a proportional relation between the NH_4-N dose applied and the NH_3-N generated (Table 4). These results match the findings of Wang *et al.* (2021) who mentioned that NH_3-N volatilization (4.3 mg, 5.27 mg, and 6.32 mg) gradually increases as the N dose applied increases (26.6 mg, 48.3 mg, and 69.0 mg).

The clay content of the soil and the application of ammonium sulfate showed statistical differences regarding NH_3-N volatilization: 750 and 600 mg kg soil^{-1} doses recovered

Table 3. Effect of soil clay content on NH_3-N volatilization.

Soil clay content (g kg S^{-1})	NH_3 (mg)
125.0	17.7333 a*
175.0	13.5467 b
225.0	12.6067 c
325.0	11.2267 d

* Means with the same letter are statistically equal (Tukey, α=0.05). MSD=0.767.

Table 4. Effect of ammonium sulfate application on ammonia volatilization.

Doses of ammonium sulphate (mg kg S^{-1})	NH_3 (mg)
750	20.1417 a*
600	16.4167 b
450	13.3083 c
300	9.5167 d
150	5.7583 e

*Means with the same letter are statistically equal (Tukey, α=0.05). MSD=0.9138.

more NH_3-N, an amount that gradually decreases as the N dose decreases (Table 5). Xie *et al.* (2019) reported a similar phenomenon: NH_3-N volatilization increases as a result of the fertilization dose applied.

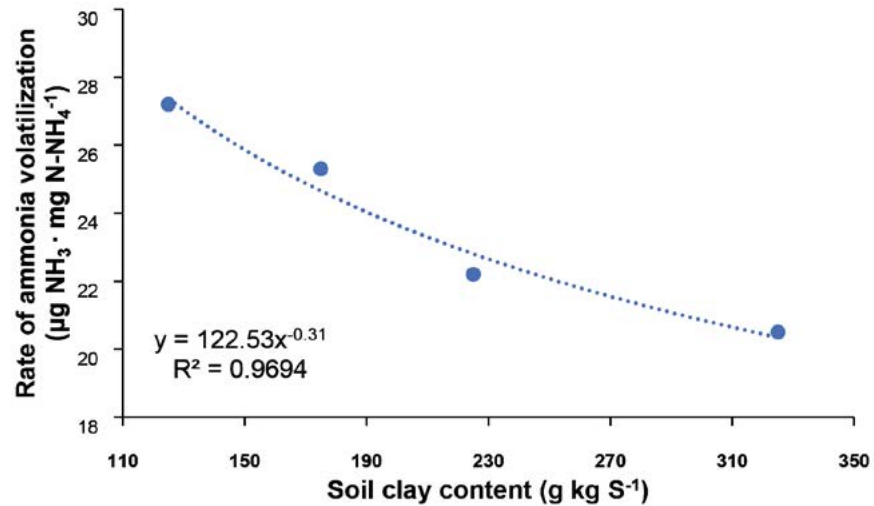**Effect of the clay content in the soil on the NH_3-N volatilization rate**

Figure 2 shows a slight decrease in volatilization speed as the clay content in the soil increases, based on the volatilization rates (27.2, 25.3, 22.2, and 20.5 μg NH_3 mg NH_4-N kg soil^{-1}) with the clay content (125, 175, 225, and 325 g kg soil^{-1}), which seemingly indicates an asymptotic trend for the study interval. This volatilization has a negative relation with the CEC and the clay content and its trend is adjusted to an exponential regression model (Hearn *et al.*, 2023), since bentonite is a 2:1 clay with a strong capacity to adsorb NH_4-N (19.01 mg NH_4-N g^{-1} of clay) and high CEC (40-130 Cmol kg^{-1}). NH_4-N adsorption in a soil to which bentonite has been added is a result of its electrostatic interaction with the negative charges of the surface, resulting from isomorphous replacement (Han *et al.*, 2020). Mazloomi and Jalali (2019) studied the relation between adsorbed NH_4-N and the concentration of the NH_4-N applied in soils treated with vermiculite and determined that the NH_4-N adsorption capacity of soils with 0% vermiculite is 285 mg kg^{-1}. It increases by 10.5%, 14%, and 24.6%, with

Table 5. Effect of soil clay content and ammonium sulfate application on ammonia volatilization.

Soil clay content (g kg S^{-1})	Doses of ammonium sulphate (mg kg S^{-1})	NH_3 (mg)
125	750	22.80 a
175	750	20.93 ab
225	750	19.40 bc
125	600	18.60 bc
325	750	17.43 cd
175	600	17.37 cde
225	600	15.80 def
125	450	15.00 efg
325	600	13.90 fgh
175	450	13.80 fgh
225	450	12.83 ghi
325	450	11.60 hij
125	300	11.20 ij
175	300	9.87 jk
225	300	8.60 kl
325	300	8.40 klm
225	150	6.40 lmn
125	150	6.07 mn
175	150	5.77 n
325	150	4.80 n

* Means with the same letter are statistically equal (Tukey, $\alpha=0.05$). MSD$=2.4241$.

Figure 2. Effect of the clay content on the ammonia volatilization rate.

the addition of 2%, 4%, and 8%, respectively. These results indicate that the adsorption capacity of the soil is directly dependent on clay, which can be interpreted through the CEC.

Significant differences were found as a result of the analysis of variance of the effect of clay content on NH_3-N volatilization. These results suggest that ammonia volatilization speed tends to decrease with a higher clay content; however, these losses continue (Table 6), since the adsorption capacity of bentonite behaves according to the Langmuir adsorption isotherm (Zaini *et al.*, 2021). Likewise, Linggi *et al.* (2020) mention that the capacity of various soils and clays (*e.g.*, bentonite) to adsorb ammonium can be explained with a non-linear model, using the Langmuir isotherm.

Significant differences were recorded in the effect of the clay content of the soil on the NH_3-N volatilization rate: the lowest clay content in the soil (125.0 g kg soil^{-1}) is more volatile (27.2409 μg NH_3 mg NH_4-N kg soil^{-1}) (Table 7). The decrease in the NH_3-N volatilization rate is the result of the NH_4-N adsorption in the exchange sites. This phenomenon prevents their reaction with the OH^- found in the soil solution, because bentonite has a wide specific surface, high CEC, and a high cation adsorption capacity (Barakan and Aghazadeh., 2020). In addition, it has negative charges generated through isomorphous replacement, which are not affected by pH (Jolin *et al.*, 2020).

Table 6. Analysis of variance of the effect of soil clay content on ammonium volatilization rates with three days of incubation with ammonium sulfate $\alpha=0.01$.

Source	DF	SS (μg NH$_3$ mg NH$_4$-N kg S^{-1})	F$_c$
Tratamientos	3	83.03	12.26**
Error	8	18.07	
Total	11	101.10	

DF: Degrees of Freedom; SS: Sum of Squares; *$\alpha=0.05$; **$\alpha=0.01$; NS: Not Significant.

Table 7. Effect of soil clay content and on the NH$_3$-N volatilization rate.

Soil clay content (g kg S^{-1})	Rate of ammonia volatilization (μg NH$_3$ mg NH$_4$-N kg S^{-1})
125.0	27.2409 a*
175.0	25.2560 ab
225.0	22.1760 bc
325.0	20.4711 c

* Means with the same letter are statistically equal (Tukey, α=0.05). MSD=3.9292.

CONCLUSIONS

Ammonium losses are recorded immediately after the application of ammonium sulfate, regardless of the clay content in the soil and they are proportional to the amount of ammonium that was applied. Significant differences have been recorded regarding the amount of ammonia produced as a result of the increase in the clay content in the soil and the ammonium doses. Clay content in the soil reduces the speed of ammonia volatilization —i.e., a greater clay content in the soil reduces the ammonia volatilization rate, as a consequence of the increase in the ion exchange sites in which the ammonium found in the solution is adsorbed. Ammonia volatilization takes place in alkaline soils and is more accentuated in sandy soils. Therefore, under these conditions, the use of ammonium sources must be limited and complemented with the application of soil improvers (organic carbon, fragments of recalcitrant carbon, and soil acidifiers) that increase exchange sites, in order to increase ammonium adsorption in the soil.

The ammonia volatilization rate helps to evaluate ammoniacal nitrogen sources, doses, and corrections, in order to increase the time that ammonium remains in the soil, as well as the efficiency of its recovery.

Clay content in the soil (measured as CEC) is an indicator of the ammonium adsorption capacity of alkaline soils. Although the NH$_3$-N volatilization is ruled by the pH, it is minimized by the type and amount of clay.

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