

## Glauconitic Siltstone: additive for ammonia retention and reduction of nitrogen volatilization loss

### *Siltito Glauconítico: aditivo para a retenção de amônia e redução de perdas de nitrogênio por volatilização*

Carlos Henrique Eiterer de Souza<sup>1</sup> , Miguel Martins Neto<sup>1</sup> , Fábio Aurélio Dias Martins<sup>2</sup> ,  
Débora Silvano Moreira<sup>3</sup> , Victor Gustavo Soares Ribeiro<sup>4</sup> 

<sup>1</sup>Centro Universitário Patos de Minas, Rua Major Gote, 808, Bloco H, sala 104, Caiçaras, CEP: 38702-054, Patos de Minas, MG, BR (carloshenrique@unipam.edu.br; miguelmartins189@gmail.com)

<sup>2</sup>Empresa de Pesquisa Agropecuária de Minas Gerais, Belo Horizonte, MG, BR (fabio.aurelio@epamig.br)

<sup>3</sup>Universidade Federal de Minas Gerais, Institute of Geosciences, Graduate Program in Geology, Belo Horizonte, MG, BR (moreiradebora@yahoo.com)

<sup>4</sup>Universidade Federal de Viçosa, Graduate Program in Soils and Plant Nutrition, Viçosa, MG, BR (victorgustavo.sr@gmail.com)

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#### Abstract

Glauconitic siltstone is sedimentary rock used as raw material to produce a multnutrient fertilizer that can combine with urea to increase their agronomic efficiency. The purpose of this study was to evaluate glauconitic siltstone as additive to reduce nitrogen losses by ammonia volatilization in nitrogen fertilizers. The experiment used randomized block, in a portion subdivided over time, following a 6 x 8 factorial with four replications and treatments comprising glauconitic siltstone mixture at a dose of 100 kg N ha<sup>-1</sup> (control, without the application of nitrogen; conventional urea – 45% N; commercial fertilizer registered with the trade name of Super N<sup>®</sup>; urea with addition of glauconitic siltstone: 9% N; 20 and 31% N) and the remaining portion divided in the collection time at 3, 6, 9, 12, 15, 18, 24 and 30 days after fertilizer application. The collection of the volatilized ammonia was made in a semi-opened free static chamber. The use of glauconitic siltstone associated with urea provided a reduction in loss by volatilization from 10 to 27% in relation to conventional urea. The treatments with application of urea with the urease inhibitor showed the lowest volatilization rate and delay in the volatilization peak, which occurred 17 days after fertilizer application. In addition to providing nutrients to the plants, glauconitic siltstone reduces ammonia losses.

**Keywords:** Glauconite; Urea-based fertilizers; N retention.

#### Resumo

O siltito glauconítico é uma rocha sedimentar usada como matéria-prima para a produção de um fertilizante multinutriente que pode ser combinado com ureia para aumentar sua eficiência agrônômica. O objetivo deste estudo foi avaliar o siltito glauconítico como aditivo para reduzir perdas de nitrogênio decorrentes da volatilização de amônia de fertilizantes nitrogenados. O experimento utilizou um delineamento de blocos casualizados, em parcela subdividida no tempo, seguindo fatorial 6 x 8 com quatro repetições e tratamentos com uma mistura de siltito glauconítico a uma dose de 100 kg N ha<sup>-1</sup> (controle, sem aplicação de nitrogênio; ureia convencional — 45% N; fertilizante comercial registrado com a marca Super N<sup>®</sup>; ureia com adição de siltito glauconítico: 9% N; 20% e 31% N); e a subparcela dividida no tempo de coleta aos 3, 6, 9, 12, 15, 18, 24 e 30 dias após a aplicação do fertilizante. Para a coleta da amônia volatilizada foi utilizada uma câmara semiaberta livre estática. A utilização do siltito glauconítico associado à ureia proporcionou a redução da perda por volatilização de 10 a 27% em relação à ureia convencional. Os tratamentos com aplicação de ureia com inibidor de urease apresentaram a menor taxa de volatilização e retardo no pico de volatilização, que ocorreu aos 17 dias após a aplicação dos fertilizantes. Além de fornecer nutrientes para as plantas, o siltito glauconítico reduz as perdas de amônia.

**Palavras-chave:** Glauconita; Fertilizantes de ureia; Retenção de nitrogênio.

## INTRODUCTION

The glauconitic siltstone is the main lithofacies in the Serra da Saudade Formation, Bambuí Group, cropping out along the ridge of the same name in Central-Western Minas Gerais, Brazil (Moreira et al., 2016). Glauconitic siltstone is composed of 40 to 80% of dark green micaceous glauconite, which is responsible for a whole-rock  $K_2O$  content of approximately 10 wt% (Moreira et al., 2016). The glauconite belongs to a mineral series ranging from smectite to glauconite, whereas dark green grains have  $K_2O$  content up to 8 wt%. It is typically formed during times of intense flooding in a transitional environment, between talud and platform at 50 to 500 m deep, normal salinity and basic pH, favored by reducing environments with the presence of organic matter (Amorosi, 1995).

The glauconitic siltstone is used as fertilizer, source of potassium, silicon, magnesium, manganese, and other micro-nutrients, which may increase soil fertility (Violatti et al., 2019; Brasil et al., 2020). The nutrient release is gradual, reducing losses by leaching, and offers the best benefit-cost ratio among the commonly used potash fertilizers, such as potassium chloride, potassium nitrate and potassium sulfate (Duarte et al., 2020). The production of fertilizer from the glauconitic siltstone relies on mechanical activation technology, which provides a physical improvement of the glauconite, performed by mineral processing operations such as size reduction, screening, and separation (Singla et al., 2019; Nader and Ackroyd, 2022). A combined measured and indicated mineral resource of 1,472 Mt at 9.28%  $K_2O$  and an inferred mineral resource of 1.850 Mt at 8.60%  $K_2O$  are estimated for the glauconitic siltstone. This amounts to 295.70 million tonnes of potash in  $K_2O$ . For context, in 2021, Brazil's total consumption of potash in  $K_2O$  was 7.92 million (Nader and Ackroyd, 2022).

Urea ( $CH_4N_2O$ ) is the major nitrogen-based fertilizer used in Brazilian agriculture, mainly due to the low cost of the nutrient and its high solubility in water when compared to other sources. However, under field conditions, urea show low use efficiency because of the high susceptibility to losses, due to the ammonia ( $NH_3$ ) volatilization (Pereira et al., 2009; Martins et al., 2014). The loss of nitrogen (N) to the environment as a consequence of volatilization results include nutrients not incorporated into the plant and soil pollution. The reduction of nitrogen availability in the soil compromises the productivity and quality of the crop, and leads to the appearance of visual symptoms, such as yellowing of older leaves and reduced growth (Pereira et al., 2009; Abranches et al., 2016; Martins et al., 2014).

Alternatives and technologies have been sought to mitigate reactions or loss processes, thus increasing the agronomic efficiency in the use of urea in agricultural systems, such as the application of additive and polymers (Abranches

et al., 2016). Glauconite has been used as natural sorbent in combination with urea to increase their agronomic efficiency (Bernardi et al., 2010; Werneck et al., 2012), due to their high cation exchange capacity (Malyovanny et al., 2013; Franus and Bandura, 2014; Stomaite and Zagorskis, 2014; Rudmin et al., 2019), which may justify its potential for ammonium retention in the soil and avoid nitrogen losses by volatilization.

In this study, for the first time, we investigated the glauconitic siltstone combined with urea fertilizers to reduce nitrogen losses by  $N-NH_3$  volatilization, in a greenhouse area.

## MATERIAL AND METHODS

The samples of glauconitic siltstone used for the test were obtained from the region of São Gotardo, in the State of Minas Gerais ( $19^{\circ}12'48''$  S/ $45^{\circ}52'43''$  W).

Samples were powdered with 95% particles smaller than 2 mm. Cation-exchange capacity (CEC) and the water-holding capacity (WHC) were analyzed in Labfert Análises Ltda according to the conventional methods for fertilizers (MAPA, 2017). The pH was determined in water; total organic matter (O.M.) by titration; K and P assimilable by Mehlich-1; exchangeable Ca, Mg and Al contents, extracted by KCl; (H + Al) potential acidity SMP solution; remaining phosphorus. Cation exchange capacity at pH 7.0, given by the expression  $T = [Ca + Mg + K + (H + Al)]$  (EMBRAPA, 2009; MAPA, 2017).

The main oxides ( $SiO_2$ ,  $Al_2O_3$ ,  $Fe_2O_3$ , CaO, MgO,  $TiO_2$ ,  $P_2O_5$ ,  $Na_2O$ ,  $K_2O$ , MnO and  $Cr_2O_3$ ) were determined by melting the samples with lithium tetraborate and subsequent X-ray fluorescence analysis (XRF) using an AxiosmAX-Minerals<sup>®</sup> spectrometer from PANalytical, at SGS Geosol Laboratórios Ltda.

For the mineralogical characterization, an X-ray diffraction (XRD) analysis was conducted in Professor Manoel Teixeira da Costa Research Center (CPMTC) of the Universidade Federal de Minas Gerais (UFMG). The whole rock sample was mounted as powder and randomly analyzed. The equipment used was a Panalytical XPERT PRO diffractometer (PW3050/60), with a  $CuK\alpha$  source, through the following parameters: scanning ranges from  $3.01$  to  $71.27^{\circ} 2\theta$ ; 40 kV and 45 mA;  $0.02^{\circ}$  step size and 0.5 s scan step time; continuous scan type; divergent slit size of  $0.9570^{\circ}$ ; 0.38 mm receiving slit size. The mineral phases were quantified using Rietveld method.

The experiment in a greenhouse area covered by a plastic tarp was conducted in the Campus II Experimental Farm of Centro Universitário Patos de Minas (UNIPAM —  $18^{\circ}34'31''$  S/ $46^{\circ}30'48''$  W). The climate in the region is classified by Köppen as Aw (a tropical climate with a dry season). Before installing the experiment, soil samples were collected at a depth of 0 to 20 cm for subsequent chemical

characterization. The samples were stored in a greenhouse covered with a plastic tarp to avoid the influence of rain. The area was subjected to the application of a 5 mm daily irrigation for five days, to promote the release of NH<sub>3</sub> that might be present. After five days, the surface was cleaned to remove organic residues, soil samples were collected for chemical characterization of the greenhouse area at a depth of 0 to 0.1 m (Table 1).

The experiment was designed in a randomized block, subdivided over time, following a 6 x 8 factorial with four repetitions. In the main portion, five types of coating applied to urea, that is, conventional urea, urea coated with N-(n-butyl) thiophosphoric triamide (NBPT), urea coated with glauconitic siltstone additive: UVA31 (31.0% additive and 31% N), UVA20 (54.5% additive and 20% N), and UVA9 (80.0% additive and 9.0% N) (Table 2), plus the control treatment without N application. The remaining portion was divided in the collection time at 3, 6, 9, 12, 15, 18, 24 and 30 days after fertilizer application. The fertilizers were applied in all treatments with a dose of 100 kg ha<sup>-1</sup> of N. Before application, the fertilizers were sieved through a 2 mm sieve to homogenize the granules and separate the sample to quantify the nitrogen dose.

A semi-open free static chamber was used, made with transparent polyethylene terephthalate (PET) flasks, with a volume of 2 L and an area of 0.008 m<sup>2</sup> (Araújo et al., 2009). To make the chamber, the base of the bottle was removed and placed on top with the aid of a rigid rope, to serve as protection. The opening at the top is used for air circulation inside the chamber (Figure 1).

The ammonia absorption system consists of a 2.5 cm wide, 25 cm long and 3 mm thick polyurethane foam sheet, which was placed vertically inside the chamber with the aid of the rigid rope. At the bottom of the rigid rope, a 50 mL plastic pot with a 1 mol L<sup>-1</sup> solution of H<sub>2</sub>SO<sub>4</sub> was attached using a rubber gum. The foam sheet placed in the plastic pot was compressed to absorb the main part of the solution, where it will remain in the pot until it is placed inside the chamber (Figure 1).

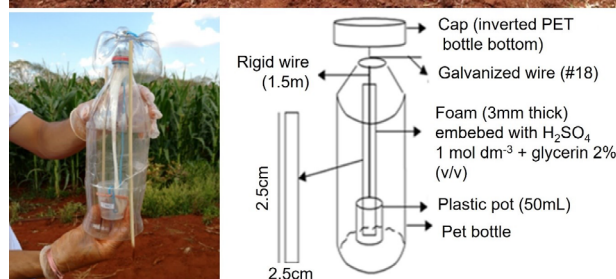
At the time of installation, the leaf was kept with the bottom part inside the 50 mL pot to avoid splashing over the fertilizer, and the other part attached to the top, so that it remained in an upright position. The rigid rope with the

50 mL pot and the foam was introduced into the chamber by the base and hung on the top edge, where they were suspended approximately 1.5 cm above the ground.

**Table 2.** Description of fertilizers applied to a greenhouse area covered with plastic tarp for quantification of volatile N-NH<sub>3</sub>.

Treatment code	Treatment	% of N
F1	Control	0
F2	Conventional urea	45
F3	<sup>1</sup> /NBPT urea	44
F4	<sup>2</sup> /UVA31 test	31
F5	<sup>2</sup> /UVA20 test	20
F6	<sup>2</sup> /UVA9 test	9

<sup>1</sup>Commercial fertilizer registered with the trade name Super N® (Fertigran); <sup>2</sup>Urea with addition of glauconitic siltstone: UVA31 (31% additive and 69% urea), UVA20 (54.5% additive and 45.5 urea), and UVA9 (80% additive and 20% urea)



**Figure 1.** Chamber model used to set up the NH<sub>3</sub> volatilization experiment as a function of the application of nitrogen-based fertilizers in no-till farming.

**Table 1.** Chemical characterization of the soil in a greenhouse area covered by plastic tarp located at the Campus II Experimental Farm of UNIPAM.

pH	O.M.	P	K	rem. P	Ca	Mg	(H + Al)	Al	T
H <sub>2</sub> O	dag dm <sup>-3</sup>	mg dm <sup>-3</sup>		mg L <sup>-1</sup>			cmolc dm <sup>-3</sup>		
5.53	3.41	27.77	143.54	6.28	2.70	1.40	5.60	0.06	9.81

pH in water; total organic matter (O.M.) by titration, according to EMBRAPA (2009); K and P assimilable by Mehlich-1; exchangeable Ca, Mg and Al contents, extracted by KCl; (H + Al) potential acidity SMP solution; rem. P: Remaining phosphorus; T: Cation exchange capacity at pH 7.0, given by the expression T = [Ca + Mg + K + (H + Al)].

After the application of fertilizers and installation of the chambers, the determinations were made from collections 3, 6, 9, 12, 15, 18, 24 and 30 days after the application of the fertilizer. The material was sent to the Soil Fertility Center (*Centro de Fertilidade do Solo — CEFERT*), located at UNIPAM, for further quantification of the ammonia captured in the chamber.

The volatilized  $\text{NH}_3$  captured by the foam sheets was quantified according to the methodology proposed by Araújo et al. (2009). The samples were transferred to a 125 mL Erlenmeyer flask, where 40 mL of distilled water were added. The Erlenmeyer flask with the diluted solution was shaken at 250 RPM for 15 min on a horizontal shaker. After stirring, a 20 mL portion was transferred to a digestion tube, being distilled in a Semimicro Kjeldhal system and titrated with  $\text{H}_2\text{SO}_4$  with a concentration of  $0.015 \text{ mol L}^{-1}$ .

The Index of Relative Volatilization (IRV – Machado, 2015) was used to compare the agronomic efficiency of treatments with conventional urea (Equation 1):

$$\text{IRV (\%)} = \frac{\text{NH}_3 \text{ Source} - \text{Control Treatment}}{\text{NH}_3 \text{ Conventional Urea} - \text{Control Treatment}} \quad (1)$$

Where:

IRV (%): Index of Relative Volatilization to conventional urea;  
 $\text{NH}_3$  Source: Amount of total  $\text{N-NH}_3$  volatilized from the coated urea;

$\text{NH}_3$  Urea: Amount of  $\text{N-NH}_3$  volatilized from conventional urea;

Control Treatment: Treatment volatilization without N application.

The results obtained were subjected to residual normality test (Shapiro-Wilk) and to the variance analysis. The treatment averages were compared by a Tukey test at 0.05 significance, using SISVAR software (Ferreira, 2000).

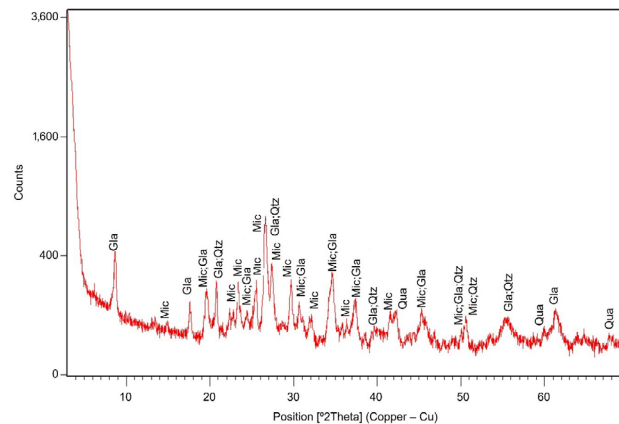
## RESULTS AND DISCUSSION

The XDR analysis indicated that the sample is a typical glauconitic siltstone, composed mainly by glauconite, microcline and quartz (Figure 2). The bulk chemical composition of sample is presented on Table 3. The sample has a  $\text{K}_2\text{O}$  content of 10.7%, plus other micro and micronutrients, such as Si, Mg and Mn.

The CEC of glauconitic siltstone, when grounded to a particle size of less than 2 mm, is  $20 \text{ mmolc kg}^{-1}$ , while the WHC is 34% m/m.

The highest volatilization values were observed in the treatments with the application of conventional urea, in the evaluations at 3, 6 and 10 days after the application of nitrogen, with the peak of volatilization occurring on the sixth day after the application (Table 4 and Figure 3).

The results corroborate those found by Duarte et al. (2007) and Cantarella et al. (2008). These authors explain



**Figure 2.** XRD pattern of glauconitic siltstone (Gla: glauconite  $[\text{K}(\text{Fe},\text{Al})_2(\text{Si},\text{Al})_4\text{O}_{10}(\text{OH})_2]$ , Mic: microcline  $[\text{KAlSi}_3\text{O}_8]$ ; Qua: quartz  $[\text{SiO}_2]$ ).

**Table 3.** X-ray fluorescence results.

Oxide	Result (w%)
$\text{SiO}_2$	58.2
$\text{Al}_2\text{O}_3$	15.1
$\text{Fe}_2\text{O}_3$	6.86
CaO	0.17
MgO	2.94
$\text{TiO}_2$	0.86
$\text{P}_2\text{O}_5$	0.12
$\text{Na}_2\text{O}$	0.12
$\text{K}_2\text{O}$	10.7
MnO	0.09
$\text{Cr}_2\text{O}_3$	0.02

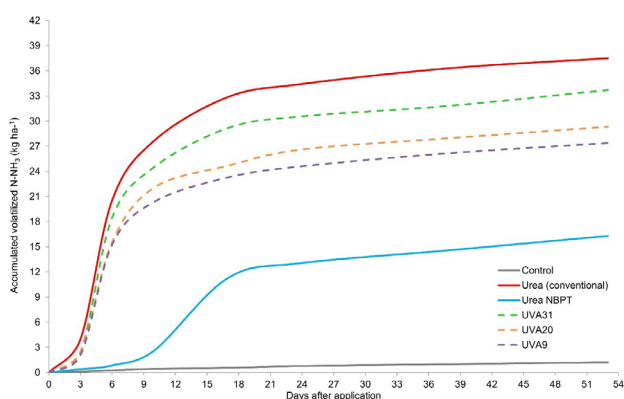
that, after the application of conventional urea on the soil surface,  $\text{NH}_3$  volatilization happens with greater intensity in the first week, and on the sixth day there is a peak of volatilization. The tested materials, except for UNBPT, also showed a volatilization peak on the sixth day after nitrogen application. However, the formulations UVA9 and UVA20, with lower proportions of urea in the mixture, showed lower values of  $\text{N-NH}_3$  captured in relation to UVA31, which contained nearly 70% of urea in the mixture. The applied nitrogen dose was  $100 \text{ kg ha}^{-1}$  of N, and the results indicated the effect of the additive, decreasing the volatilization potential of  $\text{N-NH}_3$  for urea (Table 4).

In the comparison between the conventional urea and UVA9 treatments, at the volatilization peak, on the sixth day after fertilizers were applied, the amount of  $\text{N-NH}_3$  captured in the UVA9 chambers was about 20% lower than that for conventional urea,  $13, 19 \text{ kg ha}^{-1}$  of  $\text{N-NH}_3$  (UVA9) and

**Table 4.** Average daily values of N-NH<sub>3</sub>, captured in semi-opened, free static volatilization chambers after the application of nitrogen-based treatments onto the soil surface inside a covered greenhouse\*.

Sources <sup>1</sup>	Days after application of nitrogen							Total	IRV2
	3	6	10	17	24	38	53		
	N-NH <sub>3</sub> (kg ha-1)								%
Control	0.11 Aa	0.14 Aa	0.19 Aa	0.12 Aa	0.22 Aa	0.22 Aa	0.22 Aa	1.22 a	
U <sub>CONVENTIONAL</sub>	3.96 Bc	16.57 Ec	7.14 De	5.21 Cc	1.55 Ab	1.88 Ab	1.19 Ab	37.50 e	100
U <sub>NBPT</sub>	0.39 Aa	0.44 Aa	1.79 Bb	8.66 Cd	1.78 Bb	1.53 Bb	1.71 Bb	16.30 b	43
UVA31	2.46 Bb	16.00 Ec	6.06 Dd	4.65 Cc	1.38 Ab	1.27 Ab	1.88 ABb	33.71 d	90
UVA20	2.49 Bb	13.06 Db	6.53 Cde	2.60 Bb	1.93 Abb	1.35 Ab	1.35 Ab	29.32 c	78
UVA9	2.07 ABb	13.19 Db	5.12 Cc	2.91 Bb	1.32 Ab	1.57 Ab	1.21 Ab	27.39 c	73
CV% Source = 15.89	DMSSource = 0.931				CV% total = 5.99				
CV% Time = 12.85	DMSTime = 0.899				DMS total = 3.338				

<sup>1</sup>Treatment: Control - Without the application of nitrogen; U<sub>CONVENTIONAL</sub>: Conventional urea (45% N); U<sub>NBPT</sub>: Commercial fertilizer registered with the trade name of Super N<sup>®</sup> (Fertigran); UVA9 (9% N) UVA20 (20% N), and UVA31 (31% N); Fertilizers being tested, urea with addition of glauconitic siltstone; <sup>2</sup>percentage of N-NH<sub>3</sub> captured to the treatment with the application of conventional urea; \*the means followed by the same letters in columns are not significantly different by Duncan's test (p ≤ 0.05).



**Figure 3.** Accumulated values of N-NH<sub>3</sub>, captured in semi-opened, free static volatilization chambers after the application of nitrogen-based fertilizers onto the soil surface inside a covered greenhouse after 53 days from application.

16.57 kg ha<sup>-1</sup> of N-NH<sub>3</sub> (urea) (Table 4). For the values accumulated on the sixth day, the effect was more pronounced with a 25.70% reduction of N-NH<sub>3</sub> from UVA9 (15.26 kg ha<sup>-1</sup> of N-NH<sub>3</sub>) for urea (20.54 kg ha<sup>-1</sup> of N-NH<sub>3</sub>) (Figure 3). The effect of reducing the volatilized nitrogen from urea guarantees an increase in the efficiency when it comes to fertilization of crops, for example.

In relative terms, the use of glauconitic siltstone associated with urea provided a 27% (UVA9), 22% (UVA20) and 10% (UVA31) reduction by volatilization compared to conventional urea. The total values of N-NH<sub>3</sub> captured were 37.5 kg ha<sup>-1</sup> of N-NH<sub>3</sub> from conventional urea; 33.71, 29.32 and 27.39 kg ha<sup>-1</sup> of N-NH<sub>3</sub>, when UVA31, UVA20 and UVA9 were applied, respectively. This result

is probably related to the CEC of the glauconitic siltstone on NH<sub>4</sub><sup>+</sup> retention. The CEC is the main factor affecting the losses of NH<sub>3</sub> volatilization in soils (Gasser, 1964). A higher cation exchange capacity of the soil is related to a greater number of exchange sites to retain NH<sub>4</sub><sup>+</sup> and less NH<sub>3</sub> remains in the solution, reducing losses due to NH<sub>3</sub> volatilization (Nelson, 1982).

On account of the higher value of WHC, glauconitic siltstone acts on the control of moisture during urea treatments. The elevated CEC promotes the subsequent hydrolysis of urea, allowing cationic exchanges between the potassium present in interlayers with NH<sub>4</sub><sup>+</sup> ions present in the soil. The exchangeable sites in the glauconite structure are outside the Si-Al-Fe spaces and the exchange reaction generally does not affect its structure. The CEC of glauconite could be improved by the mechanical activation (Singla et al., 2019), as occurs during the milling of the glauconitic siltstone to produce fertilizer.

The treatments with application of urea with NBPT showed the lowest rate of volatilization and delay at the volatilization peak, which occurred 17 days after the application of fertilizers. This could be due to the use of NBPT aiming to delay the hydrolysis of urea, as an inhibitor of the urease enzyme activity, which is responsible for the hydrolysis of urea (Bremner and Chai, 1986). The urea hydrolysis delay is approximately 14 days (Contin, 2007), which is remarkably close to the period reported on this research.

## CONCLUSIONS

The association of glauconitic siltstone with urea did not delay the peak of volatilization, when compared to conventional



urea, which occurred on the sixth day after application, but with less intensity.

The reductions in  $\text{NH}_3$  volatilization losses between 10 and 27%, due to the association of glauconitic siltstone with urea, when compared to conventional urea, showed that this technology is an alternative to reduce this type of loss, increase the efficiency of nitrogen fertilization, and reduce environmental impacts.

Among the sources studied, NBPT urea was the one that most delayed the peak of volatilization, on the 17<sup>th</sup> day after application, and had the lowest rates of volatilization of  $\text{NH}_3$  out of all times evaluated.

Glauconitic siltstone is a multinutrient fertilizer that provides nutrients to the plants over time. In addition, it promotes decreasing  $\text{NH}_3$  losses. Compared with conventional potash fertilizers and products containing the urease inhibitor, glauconitic siltstone production has two great advantages: the fertilizer has a high potassium grade (an average of 10%  $\text{K}_2\text{O}$ ), plus Si, Mg, Mn and other micronutrients, and the mine is close to the largest agricultural region in the country, the Brazilian savannah (Cerrado). The mineral resources are sufficient to guarantee the total Brazilian potash market demand projected for the following 60 years. Glauconitic siltstone can be used as a low-cost alternative to additive and polymers, and as a natural sorbent to increase the agronomic efficiency of urea-based fertilizers.

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