

## Fly ash as zeolites for reducing nitrogen losses by volatilization

### Uso de cinzas como zeólitas na redução de perdas de nitrogênio por volatilização

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#### Abstract

The structural and chemical characteristics of fly ash from coal-fired mineral and fly ash zeolitized are similar to those of zeolites. Urea was added with these materials in the proportions of urea: fly ashes of 100:10, 100:20, 100:50, 100:100, with a control containing just urea. These treatments were applied in soil surface and the experimental design was a randomized block with clay and sandy soil. Nitrogen losses by ammonia volatilization and the chemical characteristics of soil fertility were evaluated. In sandy soil there was reduction of ammonia volatilization for the proportions of 100:10 and 100:20, while fly ash zeolitized and fly ash had no difference.

**Key words:** Alumino-silicate, ammonia, cation exchange capacity, residue, urea

#### Resumo

As cinzas provenientes da queima de carvão mineral, seja *in natura* ou zeolitizada, possuem estrutura e características químicas semelhantes às zeólitas. Verificou-se a adição destes materiais à ureia nas proporções ureia:cinzas 100:10, 100:20, 100:50, 100:100 e um controle (somente ureia), em delineamento com blocos casualizados em solo arenoso e argiloso sem incorporação, avaliando as perdas de nitrogênio por volatilização de amônia e os parâmetros químicos de fertilidade do solo. As proporções 100:10 e 100:20 promoveram redução significativa de perdas por volatilização da amônia, porém somente no solo de textura arenosa. Não há diferença entre a cinza zeolitizada e a cinza *in natura* visando à redução das perdas de N por volatilização de NH<sub>3</sub> da ureia.

**Palavras-chave:** Aluminossilicatados, amônia, capacidade de troca catiônica, resíduo, ureia

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Natural zeolites have been applied in agriculture and environmental protection, metal remediation in water and soils, animal nutrition and aquaculture (BERNARDI et al., 2008). Agriculture is identified as a promissory market for zeolite utilization due to competitive prices and the large quantities requested. Thermoelectric mills produce huge ash quantities by burning mineral-coal. Ashes may work as zeolite applied to urea due to their composition of silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ).

Studies have been developed to determine the use and value aggregation for residues generated in consequence of the intensive production of industrial residues and pressure of social and environmental institutions along with lack of raw materials. Agriculture is becoming the main destination for industrial residues in consequence to the possibilities of economic gains as alternative sources of nutrients with lower costs to farmers as well as to factories with valorizing sub-products.

Residues application in agriculture must promote benefits such as nutrient sources, increasing fertilizer efficiency in plants, stimulating soil microorganisms, etc.; however, they must not contain toxic elements.

Urea is the most frequently used nitrogen source in Brazil; however, its reaction in soil shows a large potential of loss by ammonia volatilization (CANTARELLA et al., 2003; VITTI et al., 2007). Materials to slow or control release may improve the efficient use of urea. Extended or slow initial availability of slow release fertilizers occurs via several mechanisms. These include the solubility of the material by semi-permeable coatings, occlusion, protein materials, or other chemical forms, by slow hydrolysis of water-soluble low molecular weight compounds or by other unknown means (TRENKEL, 2010).

Zeolite added to urea has been evaluated as a method to reduce ammonia volatilization through decrease of N content in soil solution by cationic exchange (BERNARDI et al., 2008).

Slow- and controlled-fertilizers have been developed through materials added to urea to reduce nitrogen losses by ammonia volatilization. These materials act by inhibiting the urease enzyme and the time of urea solubilization in soil (SOARES, 2008). Zeolite belongs to these materials that can be added to urea to reduce ammonia volatilization.

According to Soares (2008), zeolite may increase fertilizer efficiency through its high capacity of cation exchange, water retention and adsorption ability. The slow release of nitrogen is controlled in two ways: physical coat and exchange of ammonium inside of the zeolite (ELLIOT; ZHANG, 2005). Zeolite consists of tetrahedrons of  $[\text{SiO}_4]^{4-}$  and  $[\text{AlO}_4]^{4-}$  whose organization determines its classification. The structure of zeolite consists of channels and cavities that work as a sieve with selective cationic exchange properties (OSTE, LEXMOND, VAN RIEMSDIJK, 2002).

The mechanism of action of zeolite involves the decrease of N content in soil solution by cationic exchange, mainly in soils with lower exchange cationic capacity, and ammonium retention resulting in high rates of volatilization (OLIVEIRA, 2001). In this way it retains large quantities of ammonium and also, interferes in the nitrification process (BARTZ; JONES, 1983; FERGUSON; PEPPER, 1987).

This study was carried out to determine if urea coated with ashes *in natura* and zeolitized are capable of reducing nitrogen loss by volatilization compared to urea, as well as if they can modify chemical and fertility parameters of soil.

The study was carried out in a greenhouse under temperature- and humidity-controlled conditions at the Department of Soil Sciences of Escola Superior de Agricultura Luiz de Queiroz – University of São Paulo in Piracicaba – SP.

The ash (Table 1 and 2) was stirred along with urea until the granules showed total and homogeneous coverage of ash. The urea quantity applied in the soil surface was equivalent to  $100\text{kg ha}^{-1}$  of N as urea. Fertilizer was applied in the soil surface of

pots as well as in the inter-row management of crops N in each experimental unit (17.2cm diameter of (0.8m), i.e. 3.06g of urea corresponding to 1.38g of each pot).

**Table 1.** Characteristics of ashes.

Ashes analyzed as fertilizers											
Material	K <sub>2</sub> O	Ca	Mg	S	Fe	Mn	Zn	N	B	Cl	Cu
	%										mg kg <sup>-1</sup>
Ash <i>in natura</i>	0.05	2.53	0.24	1.17	3.56	0.05	0.08	0.01	0.08	0.06	15
Zeolitized ash	0.18	1.22	0.58	0.36	3.30	0.03	0.12	3.10	0.03	0.13	23
Ashes analysed as limestones											
Material	CaO	MgO	Silica and insolubles		NP	RPTN	CEC	W-HC			
	%										
Ash <i>in natura</i>	4.37	0.0	72.94		2.50	2.50	40	94			
Zeolitized ash	3.76	1.86	41.0		18.0	18.0	80	96			

Source: Elaboration of the authors.

**Table 2.** Analysis of physical and chemical contents by IAC.

Material	Unit <sup>(1)</sup>	Ash <i>in natura</i>	Zeolitized ash
Humidity 60-65°C	% (m/m)	5.8	6.7
Organic carbon	g kg <sup>-1</sup>	146	59.7
Nitrogen (Kjeldahl)	g kg <sup>-1</sup>	2.2	1.3
C/N relation		66.8	45.8
As		450	490
B		63.3	34.6
Cd		1.4	7.9
Ca		29.5	9.7
Pb		44.5	265
Cr		13.2	38.2
Cu		11.1	19.8
Fe	mg kg <sup>-1</sup>	9582	16847
Mn		558	233
Hg		<1.0 <sup>(2)</sup>	2.8
Ni		6.2	22.0
Se		6.3	11203
K		1386	2.1
Zn		207	1160
Soluble Si		1093	74.6
P		0.25	0.14
Mg	g kg <sup>-1</sup>	0.59	7.8
S		5.9	2.9

<sup>(1)</sup> Results expressed in dry samples.

Source: Elaboration of the authors.

Two soils were used, a clayey soil (Rhodic Kandiudalf) and a sandy soil (Arenic Hapludult) with clay contents of 577 and 150, sand 215 and 836 and, silt of 208 and 14g kg<sup>-1</sup> respectively. Soils were collected at depths between 0 and 20cm, sieved and distributed to pots with 4.5kg of dry soil.

Calcium carbonate was applied in both soils to increase the pH to approximately 6. Both soils were homogenates that were humidified (50% of water retention capacity) and incubated for 30 days. Soils were sampled and analyzed after incubation (Table 3).

**Table 3.** Chemical analysis of clayey and sandy soils after incubation.

Soils	pH (CaCl <sub>2</sub> )	O.M. g dm <sup>-3</sup>	P mg dm <sup>-3</sup>	K -----mmol <sub>c</sub> dm <sup>-3</sup> -----	Ca	Mg	H+Al	SB	CEC	V %
Sandy	6.2	11	3	1.0	30	2	15	33	48	69
Clayey	6.9	19	17	2.4	120	22	11	144	155	93

**Source:** Elaboration of the authors.

Treatments consisted of urea coated in ashes *in natura* and zeolitized ash in rates of urea:ash (mass/mass) with 100:10, 100:20, 100:50 and 100:100 along with a control of urea applied with no ash (100:0). The experimental design was randomized blocks with 4 replicates, including two of soils carried out separately.

Experimental units were arranged on the greenhouse floor to avoid interference on the capitation of volatilized ammonia by the air stream from exhausts. Experimental units were weighed weekly and irrigated to keep soil humidity to 50% of the capacity field.

Losses of ammonia were quantified through foam absorbers (18 x 18cm, density of 0.02g cm<sup>-3</sup>) treated with 90mL of phosphoric acid solution (0.25 M) containing 50mL L<sup>-1</sup> of glycerin. Treated absorbers were placed under PVC plates (20 x 20 x 0.2cm) and covered by a polytetrafluorethylene (PTFE) that is permeable to ammonia and impermeable to water (ALVES et al., 2007).

Each experimental unit received an absorber foam plus PVC plate in order to avoid ammonia capitation from atmosphere. Absorber foam was placed to 1cm of height supported by stems and it was replaced at 2, 4, 6, 9, 13 and 17 days after

fertilizer application. The collected foams were stored in a freezer (-4°C) until it was extracted and analyzed (ALVES et al., 2007).

Ammonia volatilized from soils was quantified by absolute control treatment with no N or ash and it carried out along with the experiment, under the same conditions, with four replicates for each soil. Data from absolute controls were used to quantify ammonia volatilized from the treatments.

The foam containing ammonia was individually washed with 300mL of deionized water in a Buchner-Kitassato vacuum funnel system. An aliquot of 50mL of each wash from this disc was distilled and quantified by the method of Flow injection analysis (FIA) according to Su et al. (1998) and Kamogawa and Teixeira (2009).

Data of total N volatilized (TNV) were obtained from N volatilized from soil and fertilizer. N volatilized from fertilizer (NVf) was determined using the equation of NVf = TNVt - NVc. The letters t and c indicate the fertilizer treatment and absolute control, respectively.

After volatilization evaluations, soils were sampled in each experimental unit between 0 and 10cm of depth using a probe to verify the chemical content of soils.

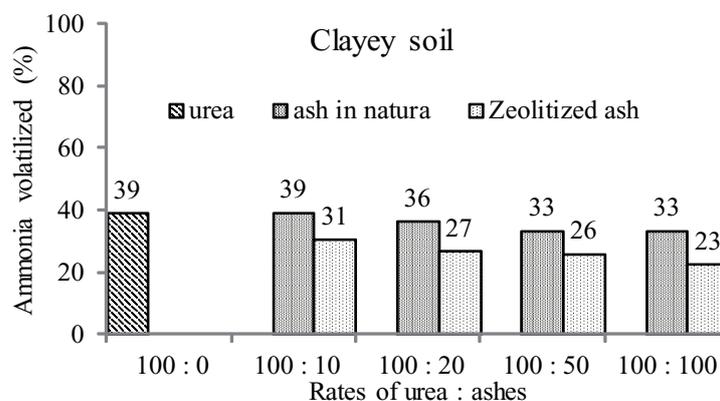
Data obtained were analyzed by PROC GLM/SAS (2004) through orthogonal contrasts comparing ashes with urea and ashes comparison among coverage rates.

Total ammonia volatilized showed no difference between the ashes used in urea coverage in both soils; however, urea coated with zeolitized ash had lower N loss when compared to control treatment in sandy soil ( $p=0.02$ ). Although zeolites contain distinct properties, they showed efficient results in the release of fertilizers, independently of the zeolites (ELLIOT; ZHANG, 2005).

Total ammonia volatilized, independent of the rates, for control treatment (urea), urea coated with ash in nature and urea coated with zeolitized ash were respectively 0.54, 0.49 and 0.37g in clayey soil and 0.85, 0.65 and 0.54g in sandy soil, representing respectively losses of 39, 36 and 27% and 62, 47 and 39% from the total N applied.

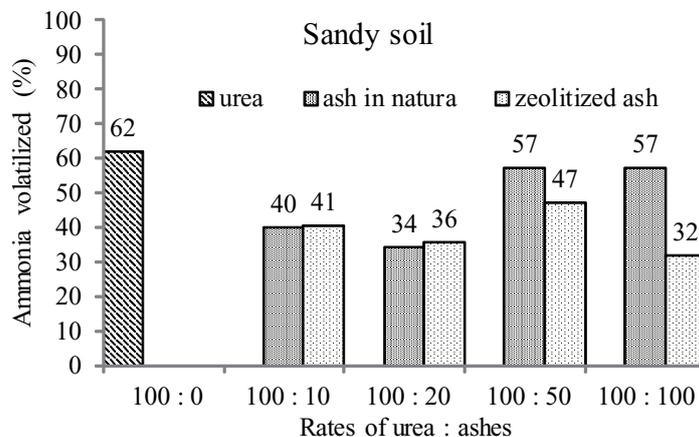
Ashes evaluated in urea coverage only had an effect over total ammonia volatilized in sandy soil, independently of coverage rates (Table 4). The coverage rates in urea had similar ammonia losses in both evaluated ashes, with no statistical difference (Figure 1 and 2) for both soils (Table 4).

**Figure 1.** Total ammonia-N volatilized (%) for rates of ashes added in urea in clayey soil. Averages followed by different letters are significant 5% by the Tukey test (CV 46%).



Source: Elaboration of the authors.

**Figure 2.** Total ammonia-N volatilized (%) for rates of ashes added in urea in sandy soil. Averages followed by different letters are significant 5% by the Tukey test (CV 21%).



Source: Elaboration of the authors.

The reduction of total ammonia volatilized showed a significant reduction as a consequence of the urea coverage rates applied in sandy soil; however, this effect was not observed in clayey soil (Table 4).

There is a different potential of volatilization for each soil. Clay and sand contents are the main components that affect ammonia volatilization. Clay may significantly reduce the ammonia volatilization

as a consequence of its capacity to fix ammonium (FRANCISCO et al., 2011). Rates of urea:ash of 100:10 and 100:20 significantly reduced the total quantity of ammonia volatilized, independently of the ash, but only in sandy soil. With the rate of urea:ash of 100:50 there was a high quantity of ammonia volatilized allowing no establishment of a regression curve, similar to that observed by Alves et al. (2007) with levels of zeolite added to urea.

**Table 4.** Ammonia-N accumulated losses for rates of urea:ashes.

Rates of urea: ashes <sup>1</sup>	Clayey soil	Sandy soil
	Ammonia-N volatilized (%)	
100:0	39 a	62 b
100:10	35 a	40 a
100:20	32 a	35 a
100:50	30 a	52 ab
100:100	28 a	45 ab

<sup>1</sup> Rates of urea: ashes (mass/mass); Averages followed by different letters are significant 5% by the Tukey test.

**Source:** Elaboration of the authors.

The high quantities of ammonia loss in this study were similar to ammonia losses from urea applied to the surface in field as verified by Lara Cabezas, Korndorfer and Motta (1997). The high ammonia losses were a consequence of high pH and the initial soil humidity.

Curves of ammonia losses showed the largest volatilization reduction through ashes use, and this effect was verified statistically in sandy soil (Table 5). The largest ammonia losses were concentrated in the fourth day after fertilizer application (Figure 3

and 4), which was also verified by Alves (2006) and Lara Cabezas and Trivelin (1990). The volatilization peak occurred between the second and fourth day after urea application, but nitrogen losses may occur rapidly when urea is applied in humid soils with appropriate temperatures (CANTARELLA, 2007).

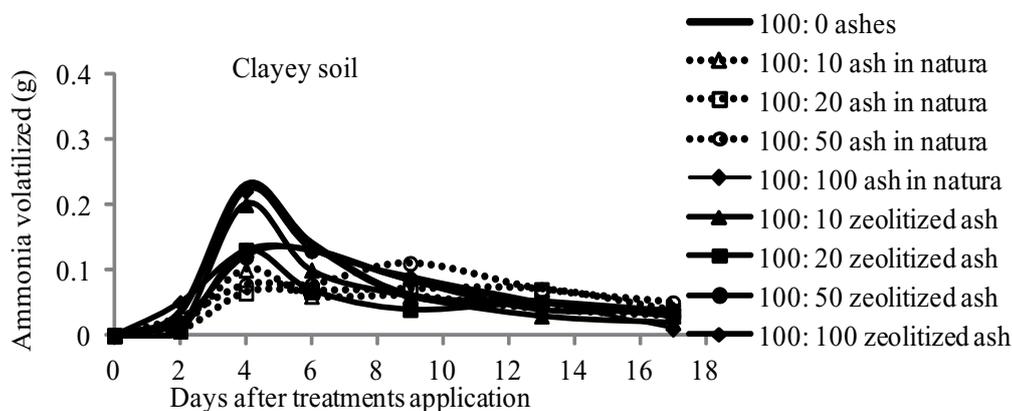
The effects of the ashes on the chemical characteristics of soil were identified by orthogonal contrast with a 5% level of significance (Table 6), possibly as consequence of the levels of soil acidity and different contents of clay and sand.

**Table 5.** Orthogonal contrasts and significance of total ammonia-N volatilized (%) in clayey and sandy soils.

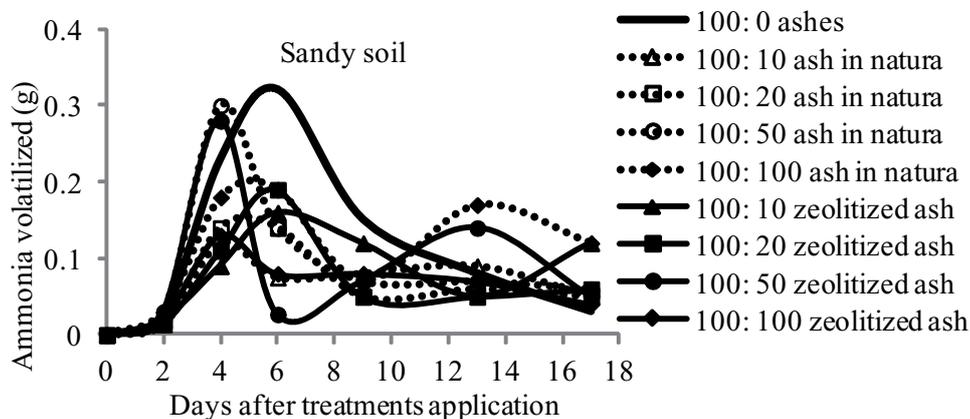
Orthogonal Contrasts				Clayey soil		Sandy soil	
				Ammonia-N volatilized (%)		Ammonia-N volatilized (%)	
				F test		F test	
Urea	vs	Ash <i>in natura</i> + Zeolitized ash		8	ns	20	*
Urea	vs	Ash <i>in natura</i>		9	ns	16	ns
Urea	vs	Zeolitized ash		6	ns	23	*
Ash <i>in natura</i>	vs	Zeolitized ash		-3	ns	6	ns
100:10	Ash <i>in natura</i>	vs	Zeolitized ash	-6	ns	-8	ns
100:20	Ash <i>in natura</i>	vs	Zeolitized ash	-4	ns	-1	ns
100:50	Ash <i>in natura</i>	vs	Zeolitized ash	-3	ns	10	ns
100:100	Ash <i>in natura</i>	vs	Zeolitized ash	3	ns	25	ns

\* = significant to 5% by F test.

Source: Elaboration of the authors.

**Figure 3.** Ammonia-N losses from urea in 17 days after fertilizers application in clayey soil.

Source: Elaboration of the authors.

**Figure 4.** Ammonia-N losses from urea in 17 days after fertilizers application in sandy soils.

Source: Elaboration of the authors.

**Table 6.** Chemical analysis of sandy soil for rates of urea:ashes 17 days after fertilizer application.

Sandy soil	pH	O.M.	P	K	Ca	Mg	H+Al	SB	CTC	V
	CaCl <sub>2</sub>	g dm <sup>-3</sup>	mg dm <sup>-3</sup>	-----mmolc dm <sup>-3</sup> -----						
Urea	7.0	11.3	4	0.6	36	1	11	37	48	77
100: 10 <sup>1</sup> Ash in natura	7.0	11.8	4	0.5	30	1	10.8	31	43	73
100: 20 Ash in natura	7.1	10.8	4	0.5	38	1	10.3	39	50	79
100: 50 Ash in natura	7.0	11.8	5	0.7	35	1	10.5	36	47	77
100: 100 Ash in natura	6.9	10.8	3	0.6	28	1	9.8	29	41	74
100: 10 Zeolitized ash	7.2	11.5	5	0.5	37	1	10	38	50	80
100: 20 Zeolitized ash	6.8	11.5	4	0.6	35	1	10.8	37	48	77
100: 50 Zeolitized ash	6.9	12.3	4	0.6	40	1	11.3	42	53	78
100: 100 Zeolitized ash	7.0	11.8	4	0.6	36	1	12	38	49	77
CV (%)	2	11	30	27	17	23	5	16	11	2
Clayey soil										
Urea	7.4	20.7	20	2.6	109	10	11	121	132	92
100: 10 Ash in natura	7.4	21.5	19	2.6	106	8	10.8	116	127	92
100: 20 Ash in natura	7.4	22.7	20	2.4	101	9	10.3	112	122	92
100: 50 Ash in natura	7.4	22.7	20	2.4	105	10	10.5	118	128	92
100: 100 Ash in natura	7.5	23.0	20	2.4	108	9	9.8	119	129	93
100: 10 Zeolitized ash	7.4	21.2	19	2.4	96	9	10	107	117	92
100: 20 Zeolitized ash	7.5	22.0	19	2.2	96	9	10.8	106	117	91
100: 50 Zeolitized ash	7.5	21.2	19	2.2	119	10	11.3	131	142	92
100: 100 Zeolitized ash	7.4	21.3	18	2.4	101	9	12	112	124	90
CV (%)	1	8	7	7	13	13	5	12	11	1

<sup>1</sup> Rates of urea: ashes (mass/mass)

**Source:** Elaboration of the authors.

In clay soil, urea coated with ash *in natura* had significantly reduced potential acidity (H+Al) when compared to the control treatments and urea coated with zeolitized ash. Potential acidity contents were 10, 11 and 11mmolc dm<sup>-3</sup>, respectively, for treatments with urea coated with ash *in natura*, control and urea coated with zeolitized ash.

Potassium content in clayey soil, analysed through orthogonal contrasts, were 0.2mmolc dm<sup>-3</sup> lower for treatments with urea coated with ashes than control, while the use of zeolitized ash resulted in levels 0.16mmolc dm<sup>-3</sup> lower than for ash use *in natura*.

The result of low potassium in soil with application of treatment urea coated with zeolitized

ash is possibly related to cation exchange. Cation exchange is a reversible chemical reaction between a solution and cations from the solid phase (zeolite), thus its dynamic depends on zeolite selectivity (ELLIOT, ZHANG, 2005). The contents of calcium, CEC and Bases saturation (BS) in sandy soil were influenced by treatments through significant contrasts between the use of ashes *in natura* and zeolitized ashes for a rate of 100:100. Treatment of ash *in natura* showed contents of calcium, CEC and BS lower (4, 5 and 9 mmolc dm<sup>-3</sup>, respectively) than zeolitized ash. In the rate 100:100, the same contrast showed that ash *in natura* was lower (9, 8 and 9 mmolc dm<sup>-3</sup>, respectively) for contents of calcium, CEC and BS.

The use of urea coated with ashes in rates of (urea:ash) 100:10 and 100:20 contributed to a significant reduction of ammonia volatilization in sandy soil.

There was no difference between ashes *in natura* or zeolitized ashes for reduction of nitrogen losses by ammonia volatilization, i.e., there is no need to process ash to this goal if it is used in adjusted rates.

Fertility parameters have a low influence from ashes added to urea, thereby the effects depend on soils and ash treatment.

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