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Influence of Nano-Zinc Oxide Coated Urea Fertilizer on Ammonia Volatilization Loss and Inorganic Nitrogen Content in Loamy Sand Soil

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ABSTRACT



A laboratory incubation experiment in a closed dynamic air flow system was conducted for 30 days to estimate the volatilization loss of NH₃-N from 0, 1, 2 and 3% Nano-ZnO coated urea treated soil. The volatilized NH₃-N from the soil chambers was collected and titrated every 48 hours. Another incubation experiment was carried out to study the effect of coating urea with Nano-ZnO on the inorganic nitrogen (NH₄-N and NO₃-N) content in the treated soils at 2, 4, 8, 14 and 21 days of incubation period. The two experiments were arranged in a completely randomized design with three replications. The results of the first experiment indicated that coating urea fertilizer with Nano-ZnO reduced the total loss of NH₃-N from 22.82 % of applied N in the soil treated with uncoated urea (0 % coating) to 13.03% for the highest coating level (3%) with a reduction in NH₃-N loss by 42.90%. The results of second incubation experiment showed that the average of NH₄-N content increased from 86.52 mg Kg⁻¹ for 0% Nano-Zn coated urea to 131.04 mg Kg⁻¹ in the soil treated with 3% Nano-Zn coated urea, and the ratio of extractable NO₃-N average to the extractable NH₄-N average was 1: 1.06, 1: 1.17, 1: 1.50 and 1: 1.75 in the soil treated with 0, 1, 2 and 3% Nano-Zn coated urea, respectively. Also, the average of total inorganic N content increased from 168.19 mg Kg⁻¹ in the soil treated with uncoated urea (0% coating) to 205.70 mg Kg⁻¹ in soil treated with highest coating level (3%). Therefore, using the Nano-Zn coated urea fertilizer improves nitrogen use efficiency and reduce ammonia loss from urea fertilizer applied to loamy sand soil.

Keywords: coated urea, nano-zinc oxide, nanoparticles, ammonia volatilization, nitrification

INTRODUCTION

Nitrogen (N) element is responsible for energy synthesis and transfer as well as amino acids, proteins, vitamins and chlorophyll molecules formation, improving dry matter and crop yield of plants (Uchida, 2000; Ekbic et al., 2010; Xia et al., 2011). Plants need nitrogen in larger amounts comparing with other nutients (Marschner, 2005). Urea fertilizer contains high nitrogen percentage (46 % N), it is a cheap and very common source of nitrogen used in the agriculture over the world (Saima et al., 2016). The surface application of urea fertilizer accelerates the rapid urea hydrolysis rate by urease enzyme in soils to ammonium carbonate (NH₄)₂CO₃ and later to NH₃, CO₂, and H₂O (Havlin et al., 1999). Ammonia (NH₃) loss from urea by volatilization may reaches 60% of the applied N varies with soil type and temperature (Sommer et al., 2004), and close to 80% losses of N applied in some soils (Alberto, 2014), which decrease the urea fertilizer use efficiency and nitrogen recovery in soil (Kiran et al., 2010). The ammonium formed through urea hydrolysis may be oxidized to nitrate through nitrification process by Nitrosomonas and Nitrobacter bacteria. Nitrates could be lost by leaching or reduced to nitrites and then to nitrous oxide gas (N₂O) by denitrifying bacteria, which are lost into atmospheric air (Mohanty et al., 2009).

Coating urea granules with some chemical, biological and nanomaterials to produce a controlled release fertilizer is considered one of promising techniques needed to alleviate negative environmental problems, low nitrogen fertilizer use efficiency and to save human health (Zhao *et al.*, 2013 & Dimkpa *et al.*, 2020). These coating materials can affect on the urea hydrolysis and nitrification processes rate. Heavy metals are among these coating materials.

Tabatabai (1977) demonstrated that trace elements such as copper (Cu), zinc (Zn), cadmium (Cd) and lead (Pb) caused an inhibition range of 51%-94%, 23%-31%, 49%-67%, 2%-55% in urease activity and therefore inhibit urea hydrolysis and loss process. Chaperon and Suave (2007) and Nascimento et al. (2013) found that Zinc and copper have negative effect on the urease and dehydrogenase enzymes of N cycling bacteria), with the potential to reduce NH3 volatilization from urea in coated fertilizer formulations. Also, Khariri et al. (2016) indicated that Cu-coated urea and Zn-coated urea reduced NH₃ volatilization by 28.00-19.44 %, and N₂O emission by 17.57, 21.62 % respectively compared to uncoated urea treatment, which provide a positive impact on the environment. Adoty et al. (2016) demonstrated that 2% zinc sulfate coated urea minimized the cumulative ammonia loss by (9.88 - 39.55 %) in different soils compared to conventional urea fertilizer. The inhibition of urease by heavy metal ions is due to their reaction with a sulfhydryl group in the active center of the enzyme by a reaction analogous to the formation of insoluble metal sulfides which were reported to be the strongest inhibitors. The heavy metal ions were found to inhibit urease in the following decreasing order: $Hg^{2\scriptscriptstyle +} > Cu^{2\scriptscriptstyle +} >$ $Zn^{2+} > Cd^{2+} > Ni^{2+} > Pb^{2+} > Co^{2+} > Fe^{3+} > As^{3+}$ (Zaborska *et al.*, 2004). Yang et al. (2006) showed that urease was much more sensitive to heavy metal than the other soil enzymes. Kundu et al. (2016) recorded a decrease in N₂O emission by 44.95% from 2% Nano-ZnO (<100 nm) coated urea after 33 days compared with uncoated urea although surface coating in general reduces the release of N from urea fertilizer, the effect could be stronger with nano-scale coatings. Also, Jadon et al. (2018) found a reduction in NO₃-N leaching by 35.1% due to application of 2% Nano-ZnO-coated urea relative to the control. Nano ZnO particles have antimicrobial properties may be due to its smaller size and high surface reactivity might provide higher surface interaction and penetration of ZnO nanoparticles into the bacterial cells. The Nano-ZnO particles might destruct the bacterial cell integrity by direct contact of ZnO nanoparticles with cell walls (Zhang *et al.*, 2007, Espitia *et al.*, 2012; Singh and Nanda, 2013) that decreases the rates of urea hydrolysis and nitrification processes. Dimkpa *et al.* (2020) indicated that coating urea with 1% Nano-scale ZnO (18 nm) powder resulted in 4% higher uptake of N by wheat plants, 19% more residual soil N, and 66% higher grain yield, compared to uncoated urea.

This study aimed to evaluate the influence of various nano zinc oxide coated urea levels on NH₃-N loss from urea-N fertilizer applied to loamy sand soil and the inorganic nitrogen (NH₄-N and NO₃-N) content in the studied soil.

MATERIALS AND METHODS

1. Soil:

The used soil was brought from the farm of Agriculture Faculty, Sohag University, Sohag. The soil was air-dried and sieved to pass through 2-mm sieve and some soil physical and chemical properties were measured (Table 1) as the following: Mechanical analysis of the soil samples was determined using the pipette method (Richards, 1954). Soil pH was measured in a soil : water (1: 2.5) suspension using a pH meter with a glass electrode (pH 211, Microprocessor pH meter, HANNA Instruments) (Jackson, 1973). The electrical conductivity of the soil was estimated in the soil saturated paste extract using an electrical conductivity meter (Orion model 150) according to Hesse (1998), the soluble cations and anions were determined in this extract according to Jackson (1973). The soil CEC was determined using 1 M ammonium acetate solution (pH= 7.0) (Chapman and Pratt, 1961). The total calcium carbonate (CaCO₃) in soil was estimated using a volumetric calcimeter (Jackson, 1973). Soil organic matter was determined using the Walkley-Black method (Jackson, 1973), total N content of the soil was determined using microkjeldahl method (Jackson, 1973).

 Table 1. Some physical and chemical properties of the soil used in the experiment.

property	Unit	Value
Sand	8	3.12
Silt	%	6.33
Clay		10.55
Texture grade	-	Loamy sand
pH (1:2.5)	-	7.51
ECe	dS m ⁻¹	2.14
OM		0.25
Total CaCO ₃	%	4.40
SP	%0	33.00
FC		17.60
CEC	Cmol ⁺ Kg ⁻¹	11.92
Total N	mg Kg ⁻¹	168
Solube cations:		
Ca ⁺²		5.10
Mg ⁺²		2.00
Na ⁺	mmol L ⁻¹	7.20
K^+		1.10
Soluble anions:		
HCO ⁻ 3		5.30
Cl		13.00
SO ⁻² 4		1.00
* Each value in this Table	is the mean of 3 replicates.	

* Each value in this Table is the mean of 3 replicates.

2. Preparation of coated urea:

Nano-ZnO powder (39 nm - Strem Chemicals, INC., USA) was used for coating urea fertilizer. The X-ray diffraction (XRD) analysis of Nano-ZnO powder was carried out to determine the particle size of nano-ZnO powder (Fig. 1) using a Bruker D8 Advance X-ray diffraction equipment, Department of Physics, Faculty of Science, Sohag University.

One hundred grams of urea (46% N) was moistened with spraying a very small amount (1ml) of arabic gum solution (10%). The granules were homogeneously covered with one gram of Nano-Zinc oxid (nZnO) powder to produce 1% Nano-ZnO coated urea (UCZn1), the same method used to produce 2% and 3% Nano-ZnO coated urea (UCZn2 ,UCZn3) using 2 and 3 grams of Nano-ZnO powder, respectively. All prepared Nano-ZnO coated urea allowed to dry for three hours. The dry, coated urea was stored in glass containers.

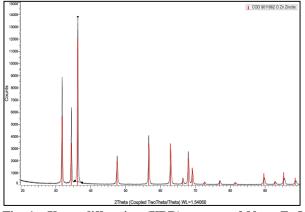


Fig. 1. X-ray diffraction (XRD) pattern of Nano-ZnO powder.

3. Ammonia volatilization loss experiment:

A laboratory incubation experiment was carried out to investigate the effect of coating urea fertilizer with Nano-ZnO on ammonia (NH₃) loss from urea amended loamy sand soil under room temperature using a close-dynamic air flow system described by Gameh (1987). This system consists of an exchange chambers with 400 cm³ in volume, 6.5 cm in diameter and 14 cm in height glass bottles containing 500 g soil sample and a trap chamber with a 250 ml Bochner flasks containing 25 ml of 2% boric acid mixed with bromo-cresol green and methyl red indicators, which were both stoppered and fit with an inlet/outlet. The inlet of the chamber was connected to an air compressor which pumped the air to pass through diluted H₂SO₄ (0.05 M) container to scrub any ammonia in the incoming air then to distilled water container to produce filtered and humidified air which was distributed through a manifold into soil chambers connected to the system. The outlet was connected by tubes to the trap containing boric acid solution to trap NH₃ gas. Boric acid flasks periodically replaced every 48 hours (2 days intervals) and titrated using 0.01 N H₂SO₄ for 30 days of incubation. Five hundred grams of air dried soil were placed in each glass bottle and then moistened to the field capacity level. Urea fertilizer (46 % N) was added to the soil surface at level of 230 mg N Kg⁻¹ through various treatments. Then, the bottles were immediately closed, and connected to the system. All treatments were arranged in a complete randomized design (CRD) with three replications and the treatments evaluated were as follows:

UCU: 0% Nano-ZnO coated urea (or uncoated urea) + soil

CUZn1:1% Nano-ZnO coated urea + soil

CUZn2: 2% Nano-ZnO coated urea + soil

CUZn3: 3% Nano-ZnO coated urea + soil

The collected NH₃-N was calculated daily and the cumulative loss was estimated as a percentage of the applied urea-N to soil.

4. Inorganic nitrogen changes experiment:

An incubation experiment was carried out to study the effect of Nano-ZnO coated Urea on the mineralization of applied urea-N over time. The treatments previously described were used. A 930 cm³ Plastic box filled with 500 grams of soil was used for each treatment. The treatments were replicated three times and nitrogen level of 230 mg Kg⁻¹ soil was used also. The soil moisture content was maintained at field capacity. The treatments were incubated for 21 days. Soil samples were taken from each box at 2, 4, 8, 14, 21 days after incubation to extract the inorganic nitrogen forms (NH₄⁺-N and NO₃⁻-N) with 2 M KCl solution. The NH₄⁺-N and NO₃⁻-N concentrations were determined in the supernatant using the modified micro Kjeldahl method (Jackson, 1973).

5. Statistical Analysis

The data statistically exposed to analysis of variance (ANOVA) using the GLM procedure in SAS software (SAS ver. 9.2, SAS Institute, 2008). The LSD at 5% significance level was calculated according to Peterson (1985) to compare the means of the studied treatments.

RESULTS AND DISCUSSION

1. Ammonia volatilization loss experiment:

The daily ammonia (NH₃-N) loss was calculated as a percentage of the applied urea-N to the soil. Clear differences were shown between the studied treatments. The highest daily NH₃-N loss (%) was shown between the 4th and the 6th day of incubation beginning and the loss was 2.53, 2.24 and 1.92% of the applied urea-N for CUZn1, CUZn2 and CUZn3, respectively compared to 3.18 % of the applied-N for uncoated (UCU) treatment (Fig. 2). Then, the daily NH₃ loss gradually decreased until the end of the incubation period for all treatments.

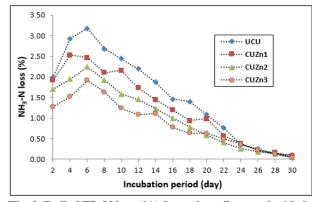


Fig. 2. Daily NH₃-N loss (%) from the soil treated with 0, 1, 2 and 3% Nano-ZnO coated urea (UCU, CUZn1, CUZn2 and CUZn3), respectively.

Fig. 3 shows the cumulative NH₃-N loss (%) recorded for the duration of the incubation. The total NH₃-N loss (%) significantly decreased from 22.82% for the uncoated urea (UCU) treatment to 18.86, 15.46 and 13.03% of the total applied urea-N for CUZn1, CUZn2 and CUZn3, respectively at the end of incubation time (30 days) (Table 2), with a reduction in loss by 17.35, 32.25, and 42.90% comparing with uncoated urea (UCU) (Table 2). It is obvious that coating urea with Nano-ZnO reduced the NH₃-N loss from this soil and this reduction increases as the coating level of Nano-ZnO increases. This reduction in the volatilization loss of NH₃ may be due to the inhibitory effect of Nano-ZnO for the soil microbes and urease which delay or slow down the urea hydrolysis and decrease the NH₃-N loss. These results coincide with the findings by Espitia *et al.* (2012) and Singh and Nanda (2013).

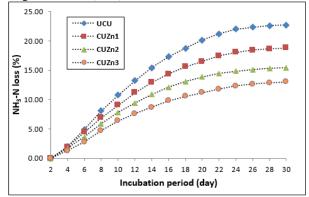


Fig. 3. Cumulative NH₃-N volatilization (%) from the soil treated with 0, 1, 2 and 3% Nano-ZnO coated urea (UCU, CUZn1, UCZn2 and CUZn3), respectively.

Table 2. The total and reduction of NH₃-N loss (%) from soil treated with Nano-ZnO coated urea after 30 days of incubation.

*Treatment	Total NH ₃ -N loss (%)	Reduction % in NH ₃ -N loss as compared with UCU
UCU	22.82A	0.00
CUZn1	18.86B	17.35
CUZn2	15.46C	32.25
CUZn3	13.03D	42.90
L.S.D 5%	1.80	-

Values with the same letter are not significantly different from each other at 5% significance level.

* UCU = soil treated with uncoated urea, where CUZn1, CUZn2 and CUZn3 treatments = soil treated with 1, 2 and 3% Nano-ZnO coated urea, respectively.

2.Inorganic soil nitrogen changes experiment:

a.Extractable Ammonium:

Results in Table 3 showed a gradual decrease in ammonium (NH₄-N) content of soil after the 4th day and till the end of incubation experiment time for all treatments; the overall average of NH₄-N concentration was 144.78, 188.53, 109.08, 65.68 and 31.50 mg Kg⁻¹ at day 2, 4, 8, 14 and 21, respectively. Results showed also an increase in the extractable NH₄-N concentration with application of Nano-ZnO coated urea to the soil. The average of extractable NH₄-N concentration increased from 86.52 mg Kg⁻¹ in the soil treated with uncoated urea (UCU) to 98.28, 115.83 and 131.04 mg Kg⁻¹ in the soil treated with 1, 2 and 3% Nano-ZnO coated urea, respectively (Table 3). The extractable NH₄-N from soil treated with urea alone was 126.00, 163.80, 75.60, 42.00 and 25.20 mg Kg⁻¹ at 2, 4, 8, 14 and 21 day, respectively, while the values were 163.33, 210.00, 149.33, 90.53 and 42.00 mg Kg⁻¹ in the same order when the soil treated with the 3% Nano-ZnO coated urea (CUZn3) (Table 3). It is noticeable that the extractable NH₄-N values were significantly higher for CUZn1, CUZn2 and CUZn3 treatments when compared to uncoated urea (UCU) at all of incubation days (Table 3).

Table 3. The effect of Nano-ZnO coated urea fertilized
application on the ammonium (NH4-N) conten
in the soil over time.

NH ₄ -N (mg kg ⁻¹)						
*Treatments	s Incubation time (days)					
	2	4	8	14	21	_
UCU	126.00A	163.80A	75.60A	42.00A	25.20A	86.52
CUZn1	138.60B	186.20B	91.00B	50.40AB	25.20A	98.28
CUZn2	151.20C	194.13C	120.40C	79.80C	33.60B	115.83
CUZn3	163.33D	210.00D	149.33D	90.53D	42.00C	131.04
Average	144.78	188.53	109.08	65.68	31.50	
L.S.D 5%	4.24	5.80	7.63	8.55	5.19	

Each value represents an average of three replications. Values with the same letter are not significantly different from each other at 5% significance level.

* UCU = soil treated with uncoated urea, where CUZn1, CUZn2 and CUZn3 treatments = soil treated with 1, 2 and 3% Nano-ZnO coated urea, respectively.

b. Extractable Nitrate:

The concentration of extractable nitrate (NO₃-N) from the soil treated with urea is presented in Table (4). Nitrate content increased gradually and slowly with time for all treatments. The overall average concentration of NO₃-N was 38.03, 54.95, 75.49, 103.25 and 125.53 mg Kg⁻¹ at days 2, 4, 8, 14 and 21, respectively (Table 4). The decrease in the NH₄-N concentration in the soil is paralleled to the increase in the NO₃-N concentration (Tables 3 and 4).The extractable NO₃-N values were higher in the uncoated urea treated soil (UCU) than all the nano-Zn coated urea treated soils till the 8th day of incubation period. Then the NO₃-N content increased till the end of the incubation time in the coated urea treated soils comparing with the uncoated urea treated soil (Table 4).

Table 4. The effect of Nano-ZnO coated urea fertilizer application on the nitrate (NO₃-N) content in the soil over time.

NO ₃ -N (mg kg ⁻¹)						
	Average					
2	21	_				
50.4A	63.00A	84.00A	95.20A	115.73A	81.70	
42.00B	67.20AB	80.27AB	109.20B	122.27AC	84.20	
34.53C	50.40B	74.67C	92.40AC	134.40B	77.28	
25.20D	39.20C	63.00C	116.20D	129.73C	74.70	
38.03	54.95	75.49	103.25	125.53		
5.90	5.82	4.50	7.23	8.08		
	2 50.4A 42.00B 34.53C 25.20D 38.03 5.90	Incul 2 4 50.4A 63.00A 42.00B 67.20AB 34.53C 50.40B 25.20D 39.20C 38.03 54.95 5.90 5.82	Incubation tim 2 4 8 50.4A 63.00A 84.00A 42.00B 67.20AB 80.27AB 34.53C 50.40B 74.67C 25.20D 39.20C 63.00C 38.03 54.95 75.49 5.90 5.82 4.50	Incubation time (days) 2 4 8 14 50.4A 63.00A 84.00A 95.20A 42.00B 67.20AB 80.27AB 109.20B 34.53C 50.40B 74.67C 92.40AC 25.20D 39.20C 63.00C 116.20D 38.03 54.95 75.49 103.25	Incubation time (days) 2 4 8 14 21 50.4A 63.00A 84.00A 95.20A 115.73A 42.00B 67.20AB 80.27AB 109.20B 122.27AC 34.53C 50.40B 74.67C 92.40AC 134.40B 25.20D 39.20C 63.00C 116.20D 129.73C 38.03 54.95 75.49 103.25 125.53 5.90 5.82 4.50 7.23 8.08	

Each value represents an average of three replications.

Values with the same letter are not significantly different from each other at 5% significance level.

^c UCU = soil treated with uncoated urea, where CUZn1, CUZn2 and CUZn3 treatments = soil treated with 1, 2 and 3% Nano-ZnO coated urea, respectively.

c.NO₃-N / NH₄-N Ratio:

The ratio of extractable NO₃-N average to the extractable NH₄-N average from the studied soil over the incubation period was 1: 1.06 in the uncoated urea treated soil (UCU), and 1: 1.17, 1: 1.50 and 1: 1.75 when 1, 2 and 3 % nano-ZnO coated urea were added to the soil. This observation may suggest a relative slower nitrification rate when urea fertilizer was coated with increasing nano zinc oxide material. These results may be due to the adverse effects of nano-ZnO material on the activity of nitrification bacteria (Singh and Nanda, 2013 and Khariri *et al.*, 2016). Similar results were also obtained by Kundu *et al.* (2016) and Jadon *et al.*(2018). This means that the released nitrate from nano-ZnO coated urea is not likely to be leached out easily as water passes through the soil amended with this Zn-coated urea, it

will move out slowly and be taken up by the plant, similar to the way as slow-release fertilizer works (Dimkpa *et al.*, 2020).

Data presented in Table (5) showed that the average of total inorganic nitrogen (NH₄-N + NO₃-N) content increased from 168.19 in UCU treatment to 182.47, 193.11 and 205.70 mg Kg⁻¹ in the soil treated with CUZn1, CUZn2 and CUZn3, respectively. These findings may be due to the effective role of coating urea with nano-Zinc oxide in reduction of ammonia loss and increasing the residual nitrogen in soil. These results matched with these reported by Dimkpa *et al.* (2020).

Table 5	. The	effect	of Na	no-Zn() coated	urea	fertilizer
	appli	cation	on the	e total in	organic	nitrog	gen (NH4-
	N + N	NO3-N) conte	nt in th	e soil ov	er tim	e

it i i i i i i i i i i i i i i i i i i						
NH4-N + NO3-N (mg kg ⁻¹)						
*Treatments	Incubation time (days)					Average
	2	4	7	14	21	
UCU	176.40	226.80	159.60	137.20	140.93	168.19
CUZn1	180.60	253.40	171.27	159.60	147.47	182.47
CUZn2	185.73	244.53	195.07	172.20	168.00	193.11
CUZn3	188.53	249.20	212.33	206.73	171.73	205.70
* LICIL - goil treated with unageted unage where CUZn1 CUZn2 and						

* UCU = soil treated with uncoated urea, where CUZn1, CUZn2 and CUZn3 treatments = soil treated with 1, 2 and 3% Nano-ZnO coated urea, respectively.

CONCLUSION

It was concluded from this study that application of Nano-ZnO coated urea to soil reduces the NH₃-N volatilization loss, slows down the nitrification process rate and increases the nitrogen recovery in soil.

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تأثير سماد اليوريا المغلف بمادة نانو أكسيد الزنك على فقد الأمونيا بالتطاير ومحتوى النيتروجين الغير عضوى فى التربة الرملية الطميية فاطمة نصر الدين ثابت*

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