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DIVISION S-8—NUTRIENT MANAGEMENT & SOIL & PLANT ANALYSIS

Ammonia Volatilization from Urea Amended with Lignosulfonate and Phosphoroamide

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ABSTRACT

Concern about losses of fertilizer N and pollution of the environment has stimulated research to find compounds that will reduce these problems by effectively inhibiting urease activity and nitrification. Laboratory studies were carried out to evaluate the effects of ammonium lignosulfonate (LS), thiophosphoric triamide (NBPT), and phenylphosphorodiamide (PPD) on NH_3 loss from urea fertilizer. We examined NH_3 losses from surface-applied urea and from banded urea (2 cm below the soil surface) in samples of Ste. Rosalie clay soil (fine, mixed, nonacid, mesic Typic Humaquept) exposed to an initial water pressure of −0.01 MPa. Formulations of solution, physical dry blend, and tablets were used. Measurements of NH_3 volatilization were carried out using an air train system. Ammonia loss from surface-applied unamended urea ranged from 16 to 21% of urea-N applied. Amending urea with LS induced between 46 and 85% reduction in NH_3 losses compared with the unamended urea. For tablet and physical dry blend formulations, banding reduced NH_3 loss compared with the surface application. There were no significant differences in NH_3 losses between surface-applied and banded treatments of solid fertilizer. Comparison of the effects of LS, NBPT, and PPD on NH_3 volatilization showed that both NBPT and PPD were more effective in reducing NH_3 loss than LS, but no significant differences between NBPT and PPD were found.

INCREASED APPLICATION of urea in agricultural practices has stimulated research to evaluate various materials that can be used as additives to inhibit the hydrolysis of urea fertilizer and thereby reduce NH_3 volatilization and nitrification (Fenn and Hossner, 1985; McCarty and Bremner, 1989; Al-Kanani et al., 1990a). Volatile NH_3 losses due to urea hydrolysis in the field may reach as much as 80% of the total N applied (Gould et al., 1986). In laboratory studies, Al-Kanani et al. (1990a) reported that NH_3–N volatilization from urea surface applied to sandy soil from eastern Canada can exceed 50% of the applied urea-N. Thus, methods to reduce NH_3 loss from urea fertilizer are needed in order to lessen adverse environmental impacts associated with the volatilization of NH_3, and to increase efficiency of the fertilizer.

Three major methods to reduce NH_3 loss from urea have been evaluated. They include (i) the use of compounds that retard hydrolysis of urea by reducing the activities of urease (Broadbent at al., 1985; Goos, 1985; McCarty et al., 1989), (ii) banding or incorporation of urea into the soil (Mengel et al., 1982; Fowler and Brydon, 1989; Campbell et al., 1990), and (iii) mixing soluble salts of K (KCl) or Ca ($CaCl_2$) with urea fertilizer (Fenn et al., 1982; Rappaport and Axley, 1984). These methods have been successful, to varying degrees, in reducing NH_3 volatilization from urea. The use of compounds that retard hydrolysis of urea is widespread with surface application, where conditions for high urea hydrolysis are favorable. Little attention, however, has been paid to the effect of chemical amendments on NH_3 loss from banded urea.

Of the many amendments reported in the literature as potential urease inhibitors, only a few compounds (i.e., NBPT and PPD) were found to be very effective in retarding urease activity in soil (McCarty and Bremner, 1989; McCarty et al., 1989). Low concentrations of these urease inhibitors are sufficiently active to retard hydrolysis of urea, and are thus agronomically feasible for field application. Phosphoroamides are relatively expensive, however, and require special handling techniques due to their unidentified toxic effects. Therefore, it seems logical to look for alternative compounds that are economically feasible, environmentally safe, and effective in improving the efficiency of urea fertilizer.

Ammonium LS may be helpful in making urea fertilizer applications more efficient by slowing or preventing NH_3 loss from urea. Because structures of some successful urease inhibitors contain a core of phenyl, quinone, or sulfide (Sahrawat, 1980; Medina and Radel, 1988; Bremner and Krogmeier, 1988) it was envisaged that LS, a product high in phenyl groups, would have potential as a urease inhibitor (Sarkanen and Ludwig, 1971). Furthermore, it is preferable that the fertilizer amendment should also provide additional plant nutrients to urea fertilizer (Al-Kanani et al., 1990b), which is not a characteristic of many effective urease inhibitors. Ammonium LS contains, on average, 4.7 g N kg^{-1}, 55 g S kg^{-1}, and 20 g kg^{-1} Fe. Ammonium-N and (NO_3 + NO_2)–N comprised, respectively, 2.2 and 0.2% of the total N content of LS. Lignosulfonate, a byproduct from the pulp and paper industry, is produced during the sulfite pulping process. Application of LS in agriculture was one of the approaches used to minimize the adverse environmental impacts caused by the dumping of LS in the rivers and waterways (Buylov et al., 1979; Ammar et

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Abbreviations: LS, lignosulfonate; NBPT, thiophosphoric triamide; PPD, phenylphosphorodiamide; U, urea; ANOVA, analysis of variance; LSD, least significant difference.

al., 1986; Singh et al., 1986). These studies indicated that LS has potential as a micronutrient carrier, but no report has been made on the effect of LS on reducing NH_3 loss from urea fertilizer.

Because N fertilizer solutions are common in many areas of the world, it is important to quantify the effect of chemical amendments on NH_3 loss from both urea solution and the dry urea. The comparison between dry and solution urea, with or without urease inhibitor, may provide more information on conditions for maximum N retention.

The objectives of this investigation were to (i) evaluate the effect of LS on NH_3 loss from urea by comparing it with known urease inhibitors like NBPT and PPD, and (ii) assess the effect of placement of urea fertilizer, amended with inhibitor or unamended, on NH_3 volatilization from urea.

MATERIALS AND METHODS

Surface soil samples (0–0.2 m, Ap horizon) were obtained from a cultivated Ste. Rosalie soil, which was chosen to represent the major corn-growing area in eastern Quebec, Canada. The soil was air dried and crushed to pass through a 2-mm screen. The soil has a clay content of 500 g kg^{-1}, 32 g organic matter kg^{-1}, 3.0 g total N kg^{-1}, 12.3 mg extractable P kg^{-1}, and pH of 5.2 (1:5, soil/water).

The NBPT and PPD were provided by the International Fertilizer Development Center (Muscle Shoals, AL). Ammonium LS (Tembind A002, fine powder <50 μm) was obtained from Temfiber Inc. (Temiscaming, Quebec). Fertilizer treatments utilized in this study were: urea prill, urea + LS, urea + NBPT, urea + PPD, urea + LS + NBPT, and urea + LS + PPD. In these treatments, the ratio of urea/LS/NBPT or PPD was 1:2.7:0.005 (dry-weight basis). Three formulations of these fertilizer treatments, including dry blend (a mixture of urea and the amendment), solution of urea (20% urea-N w/w), and tablets were used. Tablets were made by compressing the dry blend three to four times, using a hand compressor.

Jars (0.067-m i.d. by 0.124 m long) were filled with 150 g of soil. Prior to the application of the treatments, soil in the jar was moistened to a soil water pressure of −0.01 MPa, and incubated for 4 d at 23 °C. A constant flow of NH_3-free, water-saturated air was continuously passed through the jars (Al-Kanani et al., 1991). No water loss was detected during incubation.

At the end of the preincubation, formulations were either surface applied or banded 0.02 m below the surface. Tablets of urea amended with LS were 0.01 m (diam.) by 0.01 m thickness, and 0.01 m by 0.002 m with no added LS. The dry blend was surface applied as a clump approximately in the center of the jar. For banding, a 0.01-m-diam. by 0.02-m-deep hole was made at the center of the jar. The fertilizer treatment was placed in the hole followed by refilling the hole with the soil. The rate of urea-N was 135.5 mg jar^{-1}, which was equivalent to 387 kg N ha^{-1} (surface area basis). The rates of urease inhibitors were 810 mg LS and 1.54 mg NBPT or PPD jar^{-1}. A 0.678-μL fertilizer solution to give 135.5 mg urea-N jar^{-1} was surface applied. For solid fertilizer treatments, deionized distilled water was added prior to fertilizer application in order to obtain initial water pressure similar to the solution treatments (i.e., −0.01 MPa). Soil water was not replenished during the incubation period. Jars including control samples (no urea or LS added) were covered immediately following treatment applications and connected to an air train that provided a constant air flow of 0.1 L s^{-1} (Al-Kanani et al., 1991). This air flow was equivalent to 26 volume exchanges min^{-1}. Jars were incubated at an average temperature of 23.1 °C with maximum deviations of ±0.4 °C. Volatilized NH_3 was passed into 100 mL of 2% boric acid. Ammonia in the boric acid was determined by titrating with 0.05 *M* H_2SO_4 (Al-Kanani et al., 1991). Incubations were carried out in triplicate, and daily measurements of volatilized NH_3 were made for 15 d.

Following incubation, soil in the jars was shaken for 3 h with 300 mL of 2 *M* KCl solution containing phenylmercuric acetate. Treatments were measured for pH, and the filtrates were analyzed for urea-N, NH_4–N, and (NO_3 + NO_2)–N, using a Technicon Autoanalyzer (Bran & Luebbe, Milton, Canada).

Analysis of variance was done on the basis of a randomized complete block design with a factorial arrangement. Means were separated on the basis of least significant difference (SAS Institute, 1982).

Table 1. Analysis of variance values of cumulative NH_3 loss: degrees of freedom (df), sum of squares (ss), mean squares for NH_3 loss, *F* values, and variance percentage.

Source	df	SS	Mean square	*F* value	Variance† explained
					%
Corrected Total	57	918.5			100.0
Treatment (T)	5	190.5	38.1	91.0**	20.8
Form (F)‡	2	226.8	113.4	270.9**	24.7
Placement (P)§	1	74.8	74.8	178.7**	8.1
T × F	10	122.5	12.3	29.3**	13.3
T × P	5	14.8	3.0	7.1*	1.6
F × P	2	213.7	106.8	255.2**	23.3
T × F × P	9	65.8	7.3	17.5**	7.2
Error	23	9.6			

**, * Significant at the 0.01 and 0.05 probability levels, respectively.
† Calculated as (source SS/total SS) × 100.
‡ Refers to physical form (i.e., solution, dry blend, or tablet).
§ Refers to banded and surface-applied treatments.

RESULTS AND DISCUSSION

The ANOVA for NH_3 loss showed significant effects of amendment (T), the form (F) in which material was applied (i.e., solution, physical dry blend, or tablet), placement (P), T × F, F × P, and T × F × P interactions (Table 1). The effect of T × P interaction on NH_3 loss was negligible, as it explained <4% of the variance. In contrast, factors including T, F, and F × P explained between 21 and 25% of the variance for NH_3 loss. Because interactions between factors, particularly the three-factor interaction, were significant, and since

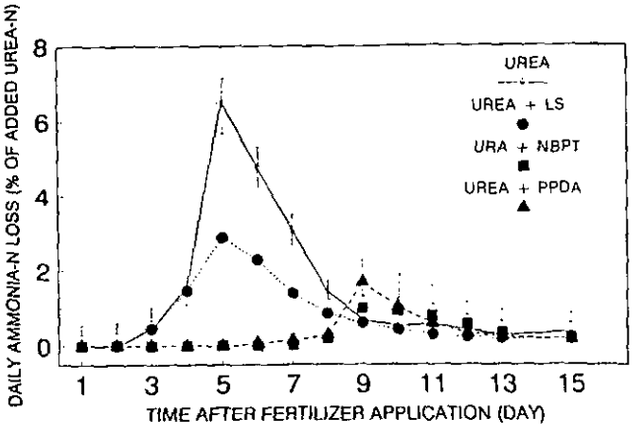

Fig. 1. Daily loss of NH_3 from dry fertilizer treatments surface applied. LS = lignosulfonate, NBPT = thiophosphoric triamide, and PPD = phenylphosphorodiamide.

Table 2. Effects of amendment and the physical form in which fertilizer was applied on N recovered from surface-applied urea.

Treatment†	N extracted from soil after 15 d‡			Cumulative NH_3 volatilized‡	Apparent recovery
	Urea-N	NH_4-N	NO_3-N + NO_2-N		
	% of added urea-N				
Dry blend					
Urea§	0.03	36.2	26.8	20.6	63.0
Urea + LS	0.47	53.8	24.0	11.2	78.3
Urea + NBPT	19.93	61.4	8.1	4.0	89.4
Urea + PPD	10.68	69.6	6.0	4.5	86.3
Urea + NBPT + LS	1.49	58.9	13.0	11.8	73.4
Urea + PPD + LS	0.51	56.0	16.1	12.4	72.6
LSD(0.05)	0.68	6.3	3.2	2.7	3.0
Tablet					
Urea¶	—	—	—	—	—
Urea + LS	1.30	59.6	20.0	2.4	80.9
Urea + NBPT	8.20	76.7	6.2	2.0	91.1
Urea + PPD	7.70	65.9	7.0	2.3	80.6
Urea + NBPT + LS	3.53	61.2	11.7	3.1	76.4
Urea + PPD + LS	2.45	59.5	12.0	3.5	74.0
LSD(0.05)	0.9	4.3	2.0	0.4	3.7
Solution					
Urea	1.10	38.9	29.9	15.9	69.9
Urea + LS	0.20	61.9	26.7	2.1	88.8
Urea + NBPT	11.90	78.5	3.6	0.4	94.0
Urea + PPD	10.63	75.5	4.8	0.5	90.9
Urea + NBPT + LS	1.47	69.4	14.1	2.7	85.0
Urea + PPD + LS	0.22	66.3	17.4	2.6	83.9
LSD(0.05)	0.81	3.2	1.5	0.7	2.6

† LS = lignosulfonate; NBPT = thiophosphoric triamide; PPD = phenylphosphorodiamidate.
‡ Corrections were made for NH_3–, NH_4–, and (NO_3 + NO_2)–N from a treatment containing only LS (no urea or inhibitor was added).
§ Urea prill.
¶ No tablets of unamended urea were made.

it is difficult to explain the biological and biochemical effects of such complex interactions, it seems logical to carry out the simple-effect examination. The simple-effect examination was carried out and mean comparisons were made using LSD (Tables 2 and 3).

Known Urease Inhibitors and Surface-Applied Lignosulfonate Compared

Losses of NH_3 from dry formulation of unamended U and U + LS treatments were detected at Day 3 after fertilizer application, and most of the NH_3 was lost within the first 7 d of incubation (Fig. 1). Losses of NH_3 from dry U + PPD and U + NBPT were detected at Day 6 and Day 7 after fertilizer application, respectively, indicating a delay in urea hydrolysis prior to those days. This suggests that the effects of PPD and NBPT were greatest in the early stages of urea hydrolysis.

Results indicate that up to 20.6% of surface-applied urea was volatilized as NH_3 (Table 2). The cumulative NH_3 volatilized during 15 d of measurements ranged from 4 to 20.6%, 2.0 to 3.5%, and 0.4 to 15.9% of urea-N surface applied as dry blend, tablet, and solution form, respectively (Table 2). It is worth mentioning here that pH values of the banded and surface-applied treatments at the end of incubation ranged between 5.1 and 5.4. This suggests that variations in NH_3 losses between treatments may not be explained on the basis of pH differences. Amounts of NH_3 volatilized from the U + LS were significantly less than from unamended U fertilizer, but greater reductions in NH_3 losses were observed with the U + NBPT or U + PPD than with the U + LS treatment, particularly when treatments were surface applied as a dry blend or as a solution. No significant differences, however, were observed among U + NBPT, U + PPD, and U + LS treatments when each of these treatments was surface applied in tablet form (Table 2). Ammonia losses from U + NBPT + LS and U + PPD + LS treatments were between the U + LS and U + NBPT or U + PPD treatments, although the differences were not always significant. These observations suggest that the addition of LS decreased the effectiveness of NBPT and PPD. Inhibition of urea hydrolysis with the NBPT was attributed to rapid decomposition of NBPT in soil with formation of *N*-(*n*-butyl) phosphoric triamide, which is a more effective urease inhibitor than NBPT (McCarty and Bremner, 1989). The exact mechanism(s) by which LS and PPD or NBPT interact has not been delineated, but one might hypothesize that it is a reaction between urease and the negatively charged LS, which makes urease less susceptible to the enzyme inhibitor.

In this study, similar amounts of NH_3–N were volatilized from U + NBPT and U + PPD treatments (Table 2). Ammonia losses from surface-applied treatments were greater from the dry blend than from the tablet or solution formulations (Table 2). In the surface-applied dry blend formulation, the effect of amendment on NH_3 volatilization from urea may have been limited due to the fact that the process of fertilizer application caused movement of some urea away from the bulk of the treatment immediately after treatment application (treatments were initially applied as a clump to the jar). For both the amended and unamended treatments, solution formulation lost less NH_3 per unit of added urea than either the dry blend or tablet formulation. This may be attributed to (i) greater homogeneity of the solution formulation in comparison with the dry blend and tablet formulations,

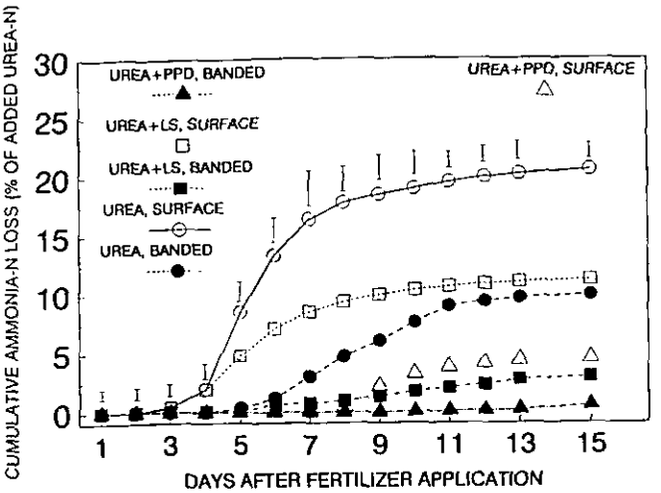

Fig. 2. Relationship between NH_3 loss from dry fertilizer treatments and time of incubation as influenced by fertilizer placement. LS = lignosulfonate, PPD = phenylphosphorodiamide.

and (ii) rapid diffusion of the solution treatment below the surface of soil in the jar.

Effect of Fertilizer Placement

When dry blend treatments were banded, NH_3 losses from unamended urea and from U + LS were detected at Day 5 and 6 after fertilizer application, respectively (Fig. 2). Ammonia loss from banded dry blend U + PPD was detected at Day 10; though loss was not apparent in Fig. 2 because data were reported as cumulative NH_3 loss. Ammonia losses from banded treatments of U + NBPT and U + (NBPT or PPD) + LS were detected at Days 5 and 6, respectively (data not shown). These observations suggest that banding could delay NH_3 losses from solid fertilizer by at least two more days in comparison with the fertilizer surface applied. Generally, banded application induced significant reduction in NH_3 losses compared with the surface application, which is in agreement with the findings of many investigators (Mengel et al., 1982; Fowler and Brydon, 1989; Campbell et al., 1990). Banded urea treatments may be in contact with less soil and exposed to fewer environmental factors (i.e., diurnal temperature and wind) than the surface-applied urea (Gould et al., 1986). Results, however, indicate that there was no difference between solution formulation and banded treatments on NH_3 volatilization.

Apparent Recovery of Inorganic Nitrogen from Soil

The apparent recovery values on Day 15 after fertilizer application ranged from 77.5 to 94.4% and 87.0 to 95.9% of added urea-N for the surface-applied and banded treatments, respectively (Tables 2 and 3). Urea-containing NBPT or PPD induced greater N recoveries than the U + LS and unamended-urea treatments, although differences were not always significant. This may, partly, be attributed to greater urea-N recovery with the U + NBPT and U + PPD treatments than with the U + LS, unamended urea, and U + (NBPT or PPD) + LS treatments (Tables 2 and 3). More urea-N was recovered from U + NBPT than the U + PPD treatment, which is in agreement with the findings of McCarty et al. (1989). Greater inhibition of urea hydrolysis with the NBPT than with the PPD was attributed to rapid decomposition of NBPT in soil with formation of *N*-(*n*-butyl) phosphoric triamide, which is a more effective urease inhibitor than NBPT (McCarty and Bremner, 1989).

The apparent recoveries of N from U + NBPT and U + PPD treatments were, in general, higher than the U + NBPT + LS and U + PPD + LS treatments (Tables 2 and 3). The reason(s) for lower recoveries with the U + NBPT + LS and U + PPD + LS is not clear, but it may be attributed to a mechanism by which less NH_4 fixation occurred with U + NBPT or U + PPD than with U + (NBPT or PPD) + LS. For dry fertilizer formulations, however, the apparent N recovery was greater in banded than in surface-applied treatments.

Table 3. Effect of amendment on N recovered from banded application of urea fertilizer.

Treatment†	N extracted from soil after 15 d‡			Cumulative NH_3 volatilized‡	Apparent recovery
	Urea-N	NH_4-N	$(NO_3 + NO_2)$-N		
	% of added urea-N				
			Dry blend		
Urea§	0.82	44.6	32.6	9.9	78.0
Urea + LS	0.34	63.9	25.6	3.0	89.8
Urea + NBPT	23.21	66.2	4.4	0.5	93.8
Urea + PPD	14.45	72.6	5.7	0.6	92.8
Urea + NBPT + LS	0.71	70.5	12.1	3.7	83.3
Urea + PPD + LS	0.45	70.3	14.3	3.6	85.1
LSD(0.05)	1.7	4.7	7.9	1.3	3.9
			Tablet		
Urea§	—	—	—	—	—
Urea + LS	1.80	60.3	24.5	1.4	86.6
Urea + NBPT	11.60	77.0	6.9	0.4	95.5
Urea + PPD	10.00	78.9	4.9	0.5	93.8
Urea + NBPT + LS	5.35	69.7	13.2	2.0	88.3
Urea + PPD + LS	4.20	76.6	10.0	2.2	90.8
LSD(0.05)	1.55	3.8	2.4	0.4	3.1

† LS = lignosulfonate; NBPT = thiophosphoric triamide; PPD, phenylphosphorodiamidate.
‡ Corrections were made for NH_3–, NH_4–, and $(NO_3 + NO_2)$–N from LS treatment to which no urea was added.
§ No tablets of unamended urea were made.

Despite the relatively high C/N ratio (10:1) of LS, the effect of LS on immobilization of N was probably very limited, as indicated by high N recovery with LS treatments (Tables 2 and 3). This suggests that the LS, which is water soluble, was either resistant to microbial degradation or behaved like a general metabolic inhibitor that requires a longer period to be degraded than the experimental conditions used in this study.

Less (NO_3 + NO_2)–N was recovered from U + NBPT or U + PPD treatment than from unamended urea, U + LS, and U + (NBPT or PPD) + LS treatments regardless of the form and placement in which treatments were applied. This indicates that NBPT and PPD may have an inhibitory effect on nitrification, although these two compounds were basically identified as urease inhibitors. Lignosulfonate also induced significant reduction in (NO_3 + NO_2)–N recovered, but only when applied in a solution form, suggesting a nitrification inhibition effect.

CONCLUSIONS

In this study, experiments were conducted under conditions that induce high NH_3 loss. Consequently, losses were close to the maximum level. There was a significant effect of LS- or urease-inhibitor-amended urea fertilizer in reducing NH_3 volatilization. The interaction of LS with urease inhibitors was complex, but it appears that the LS limited the effectiveness of the urease inhibitors. Consequently, LS or urease inhibitors should be used but not both together. Ammonia loss from the solution formulation compared favorably with losses from banded dry treatments, indicating that surface-applied solution was as effective as banding of dry fertilizer in reducing volatilization. More inorganic N was recovered from LS- or urease-inhibitor-amended urea than with the unamended urea and urea + (NBPT or PPD) + LS treatments, indicating a potential for improved N fertilizer efficiency.

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