

Volatilization losses from surface-applied urea during cold weather months

Rick Engel

Department of Land Resources and Environmental Sciences

Montana State University

Bozeman, MT 59717-3120

rengel@montana.edu (406)-994-5295

ABSTRACT

Surface application of urea is common management practice on winter wheat farms in the semiarid climate of Montana. Often this occurs during the late fall, or winter if the snowpack is shallow or early spring when soil temperatures are cold (e.g. $< 5^{\circ}\text{C}$). The objectives of this study were to quantify NH_3 losses from urea applied during this period; and to evaluate the use of the urease inhibitor, NBPT (N-(nbutyl) thiophosphoric triamide), to mitigate losses. Ammonia losses were quantified on private farms using a micrometeorological mass-balance approach. Leuning samplers were placed on a mast in the center of circular plots (40 m dia.) receiving urea (100 kg N ha^{-1}) and NBPT-coated (1.0 g kg^{-1}) urea. Ammonia losses were variable but averaged 20.5% of the application rate over the 13 trials conducted. The largest cumulative losses (30-44%) occurred after urea was applied to high water content soil surfaces, followed by a period of slow drying with little or no precipitation. Losses were moderated by applying urea to dry soil surfaces. Emissions were found to occur over a prolonged time period often lasting 6 wks. Applying urea to cold soils, or onto snow packs of modest depth ($< 15 \text{ cm}$), did not provide protection against volatilization losses. Surprisingly, some of the greatest NH_3 losses occurred periods when mean daily soil temperatures were -2 to 5°C . Coating urea with NBPT reduced cumulative NH_3 losses by approximately 2/3, though the efficacy of this compound was tied to the size and distribution of precipitation events following fertilization. Protection from NBPT lasted 2-3 wk on acidic soils, but > 7 wk at one field site with a calcareous soil (pH 8.4) suggesting NBPT breakdown occurs more slowly at high pH, and which was validated in a lab incubation study. This study demonstrated that significant NH_3 losses from surface-applied urea could occur during cold weather months in a semiarid climate.

INTRODUCTION

Surface application of N fertilizer is a common practice for winter wheat producers in the semiarid climate of Montana. The popularity of this practice can be traced to the desire to streamline operations at seeding. Farms in Montana are characterized by large acreage, no-till or minimal disturbance management, and wheat or related grains as the primary incoming producing crop. Optimum winter wheat seeding occurs over a short time window ($< 2 \text{ wk}$) in September resulting in considerable time constraints on individual farmers who often seed 1000+ hectares. Although most modern air-drills are capable of injecting N fertilizer below the soil surface, where it is less susceptible to volatilization, this practice is often not performed as it slows seeding operations down. Therefore, most growers defer N fertilization until after crop emergence. Typically, this will occur during cold weather months (October to April) when N is broadcast applied as urea (46-0-0) to the soil surface.

Ammonia (NH₃) volatilization from surface-applied urea has long been recognized as an important mechanism of N loss. After urea fertilizer is added to the soil it undergoes hydrolysis to form ammonium bicarbonate. The reaction is catalyzed by the enzyme urease, and the leads to alkaline microsites with a high concentration of ammonium that drives the production of NH_{3(g)} (Fenn and Hossner, 1985). The use of urease inhibitors, such as N-(n-butyl) thiophosphoric triamide (NBPT), is an acknowledged way to reduce NH₃ emissions to the atmosphere from urea (Grant et al., 1996). Inhibiting urea hydrolysis suppresses the rise in pH (Mikkelsen, 2009) and reduces the concentration of ammonium in the microsites around the fertilizer prill. In addition, urea is water soluble and mobile in the soil. Delays in urea hydrolysis allow more time for precipitation events to carry the urea below the soils surface where it is protected against release into the atmosphere.

Although, environmental factors controlling NH₃ volatilization from urea have been reported on extensively, very few studies have utilized micrometeorological approaches to quantify losses in the field (McInnes et al, 1986, Fox et al., 1996). In particular, we know of no study that has specifically targeted the measurement of NH₃ losses from cold soils (< 5 °C) when surface-applied urea is most frequently applied to wheat in Montana. Micrometeorological techniques with their nominal influence on the field environment are generally recognized as the preferred technique for quantifying NH₃ losses (Denmead, 1983). Approaches, such as the integrated horizontal flux method coupled with passive NH₃ samplers or shuttles described by Leuning et al. (1985) provide a comparatively easy method for quantifying NH₃ losses from urea. In addition, they enable measurements to be made over moderate size plots (0.12-0.20 ha). This article reports on the results of a micrometeorological study to quantify ammonia losses from surface-applied urea made during the October to April period to no-till winter wheat in Montana. In addition, we report on the efficacy of the urease inhibitor, NBPT, in mitigating NH₃ volatilization, and results from a laboratory on NBPT degradation as affected by soil pH.

MATERIALS AND METHODS

Field locations and climatic information

Field experiments were carried out at private dryland winter wheat farms in three Montana counties (Fig. 1). The farms in Hill and Fergus County were all managed under no till wheat-fallow rotations. Treatments (discussed below) were established in fields (>100 ha) that had been seeded to winter wheat in September. Field experiments conducted in Gallatin County (Campaigns 9 and 10) were managed under a tilled wheat-fallow system. At this farm, treatments were applied to a field with standing wheat stubble that had been harvested the previous August. Long-term annual precipitation at the Hill, Fergus, and Gallatin County sites is approximately 291, 384, and 326 mm, respectively. Location, soil series name and classification, and selected soil properties of the surface 10 cm are described in Table 1. Soil pH of the fields was acidic (pH 5.5 to 6.8) at the Hill and Fergus County sites. The Gallatin County field site was alkaline (pH 8.4) and calcareous (12.1 g CaCO₃ kg⁻¹ equivalent).



Fig 1. Location of field experiments

Table 1. Location and selected soil characteristics of field sites.

Campaign	County	Soil series	Sand	Clay	Silt	CEC	pH	Organic C
			----- % -----			cmol kg ⁻¹		g kg ⁻¹
1	Hill	Phillips-Elloam	44.7	26.8	28.5	10.4	6.1	9.2
2,5†	Hill	Phillips-Elloam	45.5	31.1	23.4	11.7	6.4	12.8
3,4	Hill	Telstad-Joplin	61.6	22.6	15.9	7.6	5.5	10.2
6	Hill	Phillips-Elloam	41.8	26.2	32.0	10.0	6.0	13.2
7,11†	Hill	Scobey-Kevin	38.3	31.7	30.0	10.9	6.5	19.0
8	Hill	Telstad-Joplin	58.4	24.1	17.5	9.0	5.5	14.3
9,10†	Gallatin	Brocko	33.8	28.6	37.5	10.6	8.4	10.1
12	Hill	Scobey-Kevin	39.6	27.7	32.7	10.1	6.0	14.7
16	Fergus	Danvers	39.3	25.5	35.2	-	6.8	25.5

† Campaigns conducted in the same field but with different fertilizer application dates.

Integrated Horizontal Flux (IHF) System

The IHF system utilized consists of a center mast with multiple shuttles (Leuning et al., 1985) arranged in a gradient spacing going up the mast. Masts are placed in the center of circular plots treated with the NH₃ emitting source material (e.g. urea or NBPT coated urea). Each shuttle consists of an entrance hole in the front cone section, a PVC pipe cylinder with mounting pivots, and a rear tail section with Venturi shield and exit hole. Stainless steel fins are mounted to the rear tail section to keep the shuttle aligned into the wind. The interior of the shuttle contained a stainless spiral that is coated with oxalic acid. During operation, convection forces move air containing NH₃ into the shuttle through the cone shape entrance hole. Once inside the shuttle the air is stripped of its NH₃ by sorption to the spiral treated with oxalic acid. Ammonia lost from the soil surface is calculated by a mass balance calculation that has been discussed in the literature (Denmead, 1983). Briefly, the mass of NH₃ collected by the shuttle (M) is divided by the product of the shuttle's effective cross-sectional sampling area (A) and time (T) to give the horizontal flux at each measurement height. The effective cross-sectional sampling area was defined by Leuning et al. (1985) in wind tunnel tests. During these tests, air flow through the shuttle was found to be linearly related to wind speed. The slope of this line, $2.42 \times 10^{-5} \text{ m}^2$, defined the effective sampling area. Net horizontal fluxes from the treated areas are calculated by subtracting background emissions at each respective height. Vertical flux (F_v) is then calculated by integrating the net horizontal fluxes over the height profile (0 to Z , or height intervals), and dividing by the fetch distance or plot radius (x) to give the mass of NH₃ lost per unit area-time (see equation below). Once F_v is calculated, it is multiplied by the sampling time interval to determine the N loss, or divided by the N rate to determine the fraction of urea-N lost as NH₃.

$$F_v = \frac{1}{x} \left[\int_0^Z \frac{M}{AT_{trt}} - \frac{M}{AT_{bkg}} \right]$$

Field design, gas sampling, shuttle preparation, and NH₃ analysis

Field experiments consisted of two N fertilizer treatments, urea and NPBT (1 g kg⁻¹) coated urea applied at a rate of 100 kg N ha⁻¹. The NPBT material was a liquid formulation sold under the trade name, Agrotain. Five Leuning shuttles were placed on the mast at heights 0.25, 0.50, 1.00, 1.50, and 2.75 m; (2.50 m used in Campaign 1) above the soil surface during Campaigns 1-12. Six shuttles were placed on the masts during Campaign 16 at heights of 0.25, 0.50, 1.00, 1.50, and 2.25, and 3.00 m above the ground. A third mast with Leuning samplers placed at identical heights was installed in an unfertilized area of the field to correct for background emissions. All masts were positioned a minimum of 200 m apart. Gas sampling campaigns were conducted over ~8 weeks following fertilization. Shuttles were charged with a solution of 30 g L⁻¹ oxalic acid in acetone. During the charging phase, shuttles were warmed in an oven (50 °C), removed and placed inside

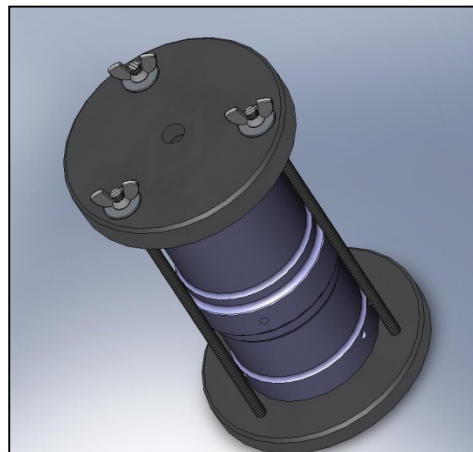


Fig 2. Shuttles with end-caps for NH₃ elution.

a ventilated hood, sealed at one end with a custom designed cap, and the oxalic acid-acetone solution was poured from a beaker into the shuttle in sufficient volume to completely immerse the stainless steel coil. The solution was left inside the shuttle for approximately 15 to 20 seconds and then drained back into the beaker, before placing back in an oven (50°C) to dry. In the field, shuttles were exchanged on a mostly weekly interval over the gas sampling campaigns. Immediately after removing from the mast, spent shuttles were sealed by inserting a rubber stopper into the front cone section, and covering the rear exit hole with tape. Spent shuttles were then placed inside large, 11 L Zip-lock plastic bags and transported to the lab for elution. A custom designed washer system was used during the elution phase (Fig. 2). The washer consisted of two PVC end-caps with O-rings that fit over each end of the shuttle. The end caps were held tight against the shuttle with four long threaded rods that formed a water-tight seal. During elution phase, the front cone and rear tail sections were removed and the shuttle was placed inside custom designed washer. Deionized water was added through an opening in one of the end caps and shuttles were shaken vigorously. The contents were then poured into a polypropylene bottle, and an aliquot was removed for analyses on a Timberline TL-2800 NH₃ analyzer (Timberline Instruments, Boulder, CO 80301).

Portable weather stations were installed at farm-sites to record air-temperature, soil temperature (1 cm depth), wind speed, and relative humidity.

Lab incubation study

Soil samples from surface horizon of a Telstad-Joplin loam and Brocko silt loam were collected from the field sites where Campaigns 3 and 9 were conducted. The Telstad-Joplin and Brocko soils were dried, sieved (<2 mm), and a 10 g subsample was placed in a 60 mL high-density polyethylene bottle. A third soil was constructed by adding 0.3 g of CaCO₃ to a set of bottles with 10 g of Telstad-Joplin (hereafter referred to as Telstad-Joplin + CaCO₃). To each bottle 2.55 mL of water was added. The samples bottles, 160 for each soil, were covered with loosely fitted caps and placed in an incubator at 20 °C for 1 wk. At end of the 1 wk pre-incubation, the bottles were removed and two fertilizer treatments, 20 mg urea and 20 mg urea + 20 µg NBPT were imposed. Forty bottles of each soil x fertilizer treatment (urea and urea+NBPT) were capped loosely and placed in dark incubators maintained at 0.5 and 20 °C.

Four replicated bottles of each treatment were removed at 1, 2, 4, 6, 8, 10, 14, 18, and 28 d. The samples were extracted with 50 mL 1 N KCl by placing on a reciprocal shaker for 30 min. The extracts were allowed to settle for a few minutes and a 500 μ L aliquot was transferred to microcentrifuge tube with 50 μ L of dimethyl sulfoxide (DMSO) in preparation for NBPT analysis (described below). The remainder of the extract was then filtered and a second aliquot (10 mL) was transferred to a disposable tube for urea analysis. This aliquot was spiked with a few drops of a concentrated phenylmercuric acetate solution to give 5 mg L⁻¹ and to prevent further hydrolysis in the test tube before urea was quantified using an automated flow injection analyzer.

Quantitation of NBPT was performed using HPLC-mass spectrometry. Soil extracts were centrifuged for 5 minutes at 10000G to remove residual soil particulates. The supernatant was transferred to polypropylene auto-sampler vials and capped. Reverse-phased chromatography was used to separate the NBPT and similar compounds using an Agilent RRHD 100 x 2.1mm 1.8 μ m C18 column. The solvents consisted of H₂O with 0.1% formic acid in channel "A" and acetonitrile with 0.1% formic acid in channel "B". This chromatography method provided isocratic elution of the NBPT-related compounds at approximately 2-3 minutes. The mass spectrometer used was an Agilent 6538 using the DualSpray source and positive ionization mode, operating in the high dynamic range mode with the low mass range. The source used 55 psi for the nebulizer and 10 L min⁻¹ of drying gas. The fragmentor (capillary exit) voltage was 150V, and the capillary voltage was 3500V. Data quantitation was carried out using the Agilent MassHunter Quantitative Analysis software package. Extracted ion chromatograms were defined using the accurate-mass monoisotopic peaks for each appropriate compound: NBPT, deamidated NBPT, NBPT_o (oxygen analog).

RESULT AND DISCUSSION

NH₃ losses from surface-applied urea

Total cumulative NH₃ losses, expressed as a percentage of the applied N rate (100 kg N ha⁻¹), averaged 20.5% for the 13 field trials but were quite variable (Table 2) as a result of differences in environmental conditions at the time of fertilization and precipitation patterns during the weeks following fertilization. The largest cumulative NH₃ loss, 44.1% of applied N, occurred during Campaign 10 which was conducted on a Brocko (pH 8.4) soil. For this campaign, NH₃ flux peaked during the second sampling period (5-12 d) but never fell to background levels even after 61 d – or the end of the campaign (data not shown). Total cumulative NH₃ losses exceeded 30% of applied N in three trials conducted on acidic soils, including Telstad-Joplin (pH 5.5, Campaigns 3 and 4), and Phillips-Elloam (pH 6.4, Campaign 5). Ammonia flux peaked in the first (0-7 d) or second sampling period (7-14 d). A commonality of all large NH₃ loss campaigns (>30% of applied N) was that fertilizer applications were made to a visibly damp or high water content soil surface that resulted in dissolution of urea granules. Precipitation events that followed were light (<8 mm) and isolated at least through the first 30 d post-fertilization. Conditions were particularly wet at the beginning of Campaign 5. A trace amount of snow was present on the ground and the soil water content (0-8 cm) was near-saturated ($\theta_v = 50\%$). Over the first 7 d, no precipitation fell and the volumetric soil water content fell to 24.6%. The NH₃ loss over this sampling period was equivalent to 22.4% of the applied N rate (Fig. 3), the largest NH₃ emission period observed in this study.

Broadcast application of urea onto snow packs of modest depth (< 150 mm) is sometimes practiced in semiarid NGP, as growers believed that NH₃ losses are negated by this practice. We conducted two such applications during this study (Campaigns 9 and 16) at a field site with a calcareous Brocko silt loam soil and Danvers loam (pH 6.8). The resulting 24.3% and

Table 2. Percentage of applied N lost as ammonia-N following broadcast applications of urea and NBPT- coated urea.

Campaign	Fertilization date	Urea	NBPT
% applied N loss as NH ₃			
1	3 Apr. 2008	8.4	4.4
2	8 Oct. 2008	3.1	1.4
3	14 Nov. 2008	31.3	3.8
4	25 Mar. 2009	35.6	18.0
5	26 Mar. 2009	39.9	18.1
6	6 Oct. 2009	11.6	4.3
7	13 Oct. 2009	10.4	4.8
8	19 Oct. 2009	15.7	3.4
9	27 Jan. 2010	24.3	9.3
10	26 Feb. 2010	44.1	11.9
11	29 Mar. 2010	6.3	1.7
12	20 Apr. 2010	14.7	1.8
16	2 Mar. 2011	20.7	10.1

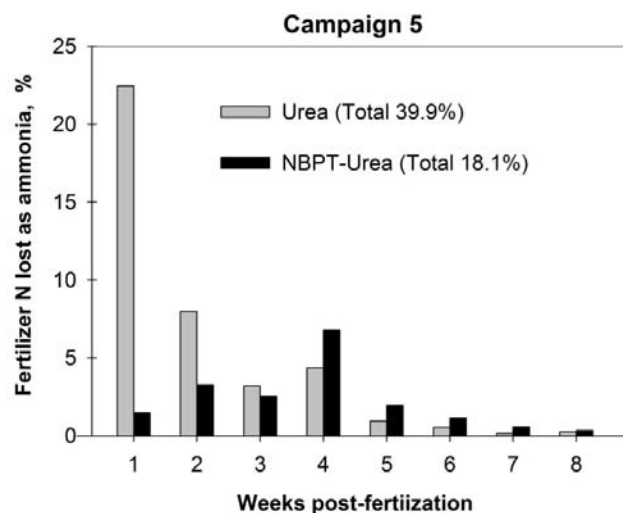


Fig. 3. Field site conditions at fertilization (26 March 2009) and cumulative NH₃ losses over each week during Campaigns 5 expressed as a percentage of the applied fertilizer N (100 kg N ha⁻¹).

20.7% applied N loss was surprising (Fig. 4), as well as our finding that a small quantity of NH₃ (i.e. 2-3 kg N ha⁻¹) was lost prior to the disappearance of the snow pack during Campaign 9. Emissions of greenhouse gases have been reported through snow-covered soils however, we are unaware of a previous report of NH₃ emissions through snow. It is unknown whether hydrolysis of urea at this field site was occurring inside the snowpack or on the soil surface. Urease is both an intracellular and extracellular enzyme and its presence in the environment is considered ubiquitous. Plant residues, including from wheat, are known to possess significant urease activity (Kissel et al., 2008). The wheat residue at this field site was comparatively

undecomposed as the soil had not been tilled following its harvest in August. Hence, it seems plausible that at least some urea hydrolysis may have occurred in the snowpack.

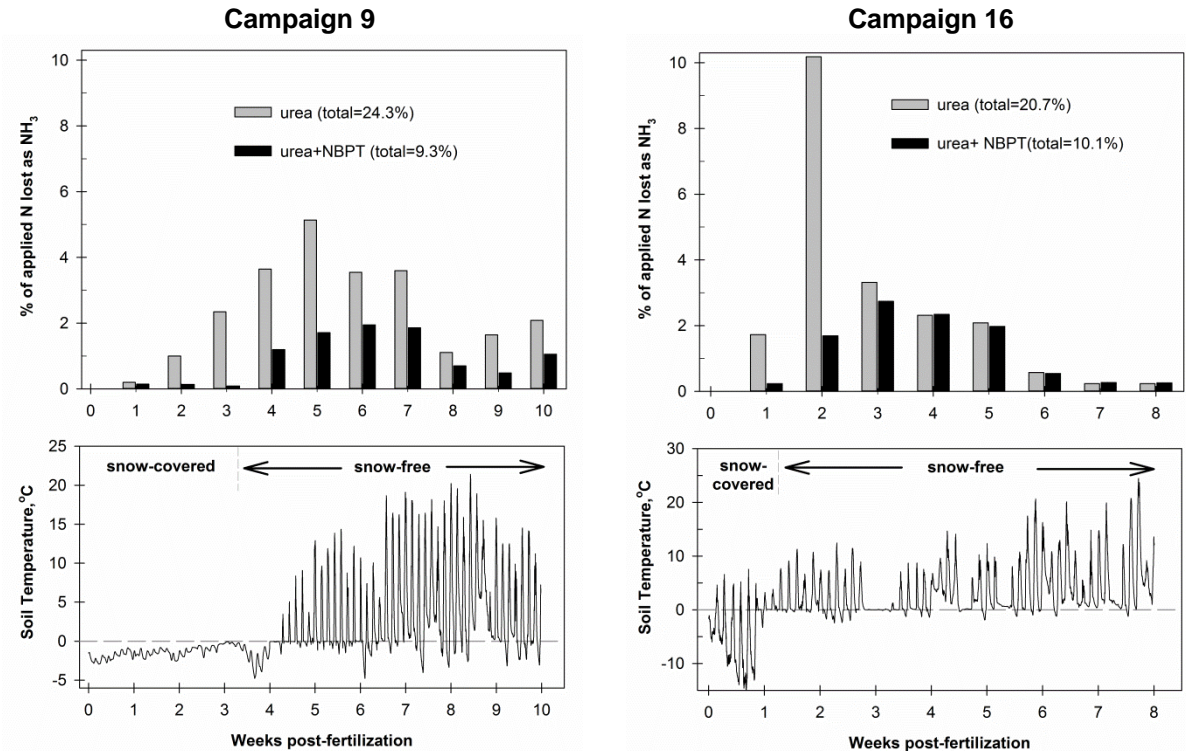


Fig 4. Cumulative NH_3 losses (% of applied N) from urea and urea-NBPT over each week following applications to field sites covered with modest snow packs at Campaigns 9 (15 mm water equivalent) and 16 (8 mm water equivalent).

Ammonia losses were moderated, or below the overall study mean (20.5% applied loss), by applying urea to dry soil surface. If the precipitation events that followed were light (<8 mm) and scattered losses were reduced to 10-20% of applied N. These conditions occurred during Campaigns 6, 7, 8, and 12. If the precipitation events were heavy (>18 mm) then losses were <10%. These conditions occurred during Campaigns 1, 2, and 11. The importance of soil water and precipitation on NH_3 loss is further provided by the contrasting results from Campaigns 2 and 5. Both trials were conducted in the same field, yet NH_3 losses during Campaign 5 (initial wet soil followed by a drying period) exceeded the losses during Campaign 2 (initial dry soil followed by a large, 24.9 mm precipitation event) by more than 10-fold

A previous review on NH_3 volatilization from fertilizers noted that large NH_3 losses are associated with an initially wet surface soil followed by several days of slow drying with little or no precipitation; and that N losses from urea are mitigated when sufficient rain or irrigation, typically 12.7 to 25.4 mm, occurs to move N into the soil profile (Hargrove, 1988). Our results are consistent with this review as the four largest NH_3 loss campaigns (30-44% of applied N) occurred when urea was applied to a wet or damp soil surface followed by scattered light precipitation events (<8 mm). Similarly, large NH_3 losses of 20.7 and 24.3% occurred in two campaigns where applications were made to soils covered with snow packs of modest depth. During these campaigns, the period of greatest emission activity occurring after the snowpack disappeared and the soil surface dried. Our results differ from other studies in two ways. First, NH_3 emissions from urea were observed over a long duration. In 9 of 13 trials it took 30 d or longer to accumulate at least 90% of the total NH_3 loss. This contrasts with reports from eastern

Canada where the rate of NH_3 volatilization from N fertilizer was found to decrease dramatically within 20 to 28 d following application (Ma et al., 2010). In Pennsylvania, Fox et al. (1996) reported that 90% of total NH_3 volatilized from urea and UAN took place within 10 d after application in two of three years. In Australia, Turner et al. (2010) reported emissions from urea peaked 2 d after fertilization and no emissions were observed after 16 d. Two reasons for the prolonged emissions were dry soil conditions and cold temperatures that slowed urea hydrolysis. A second unique aspect of this study was that cold mean daily soil temperatures (-2 to 5 °C) did not provide protection against realizing large NH_3 emissions. This is evident in the scatter diagram of mean NH_3 flux vs. mean daily soil temperature (Fig. 5). Time-integrated NH_3 flux for all sampling periods across the 13 campaigns was not correlated ($R^2=0.05$) with soil temperature at 1-cm depth. It is apparent from the scatter diagram that a significant fraction of the high NH_3 emission periods ($>30 \text{ g N ha}^{-1} \text{ h}^{-1}$ equivalent to $> 5 \text{ kg N ha}^{-1} \text{ wk}^{-1}$) occurred when soil temperatures averaged -2 to 5 °C. We are unaware of previous reports of significant NH_3 losses at these temperatures. Volatilization rates of NH_3 from urea can exhibit wide diurnal patterns with maximum flux activity occurring during midday. Hence, much of the NH_3 loss activity observed over the sampling periods may have been weighted to hours of the day when temperatures were above the daily average, or near the maximum. However, even with this consideration, it should be noted soil temperature only exceeded 5 °C for 5.3 h during the highest emission period ($134 \text{ g N ha}^{-1} \text{ h}^{-1}$ – Campaign 5) observed in this study.

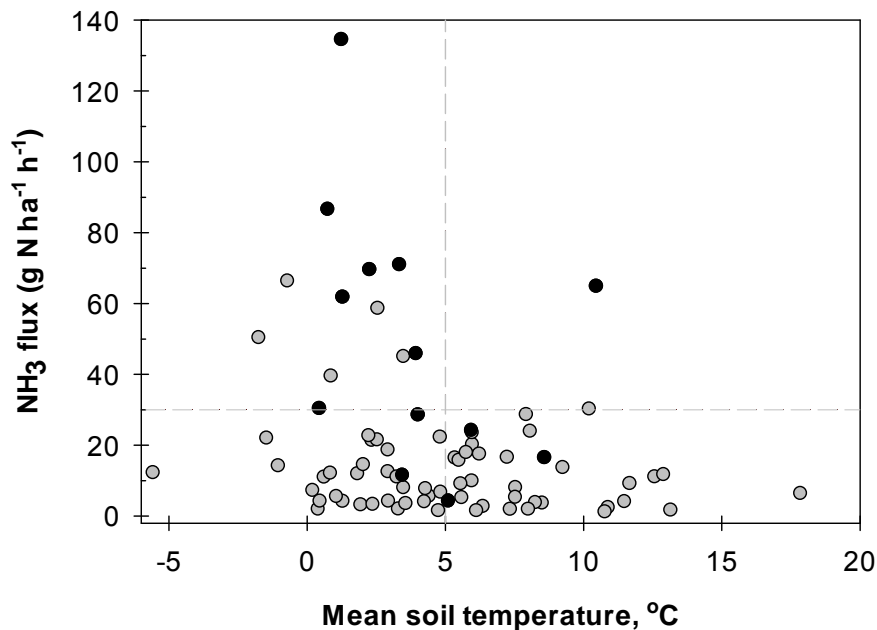


Fig. 5. Mean time-integrated NH_3 flux vs. soil temperature (1-cm) for all sampling periods where emissions exceeded background. Dark circles represent the maximum flux observed during Campaigns 1-13.

NH₃ volatilization mitigation by NBPT and urea transformations

The addition of the urease inhibitor NBPT to urea was effective in reducing cumulative NH₃ losses in all trials (Table 2). Total cumulative NH₃ losses averaged 7.2% of applied N with NBPT, or about 65% of the losses from untreated urea. The reduction in total cumulative loss by NBPT was as great as 88% during Campaigns 3 and 12 because NH₃ emissions for the entire campaign were largely confined to the first 14 and 20 d post-fertilization, respectively. Typically, the reduction in NH₃ loss by NBPT on acidic soils was limited to 2 wk following dissolution of the fertilizer granules, i.e. after two weeks post-fertilization NH₃ losses was similar for the NBPT treated and untreated urea. This was very evident in the high NH₃ loss trials (>20% applied N loss) (see Fig. 3 and 4). The benefit of NBPT persisted longer on calcareous soils (Campaign 9 - see Fig. 4), suggesting that degradation of NBPT and/or its metabolites may have occurred more slowly at high pH. We conducted a lab incubation study to validate these observations from the field. The results summarized in Fig. 6 clearly indicate that NBPT degradation is slower if CaCO₃ is present in the soil. This response is particularly apparent at 0.5 °C (0 °C indicated in figures) compared to 20°C. For example, NBPT fell to detection limits within 6 d in the Telstad-Joplin loam at 0.5 °C. If this same soil was spiked with CaCO₃, NBPT was still detectable up to 18 d into the incubation. A reduction in NBPT degradation should also impact urea hydrolysis as NBPT is a urease inhibitor. Soil analysis revealed that urea disappeared at rate of 6.0% and 8.3% per day without NBPT in the Telstad-Joplin loam and Tetstad-Joplin loam + CaCO₃ at 0.5 °C, respectively (Fig. 7). The corresponding rates of urea hydrolysis in these same soils were 2.7% and 0.5% per day with NBPT. Therefore, presence of NBPT reduced the rate of urea hydrolysis by a factor of 2 times at pH 5.5 (i.e. Telstad-Joplin loam) and 17 at pH 8.2.times.

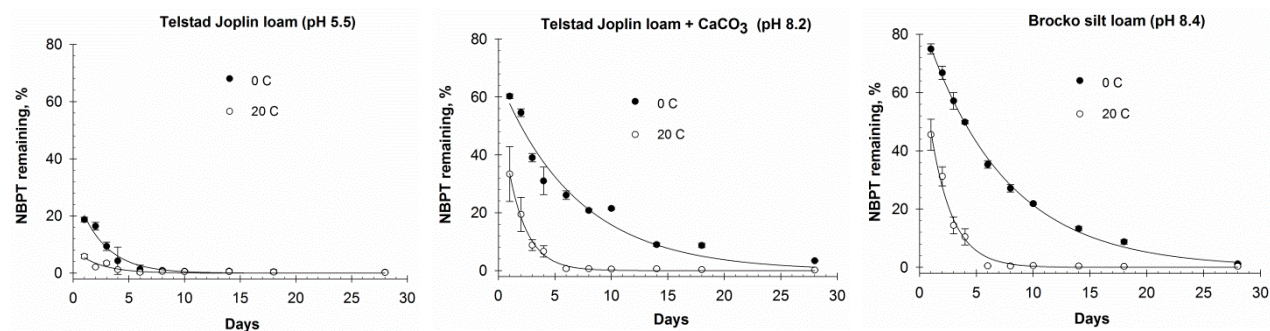


Fig 6. NBPT degradation in three soils and at two temperatures.

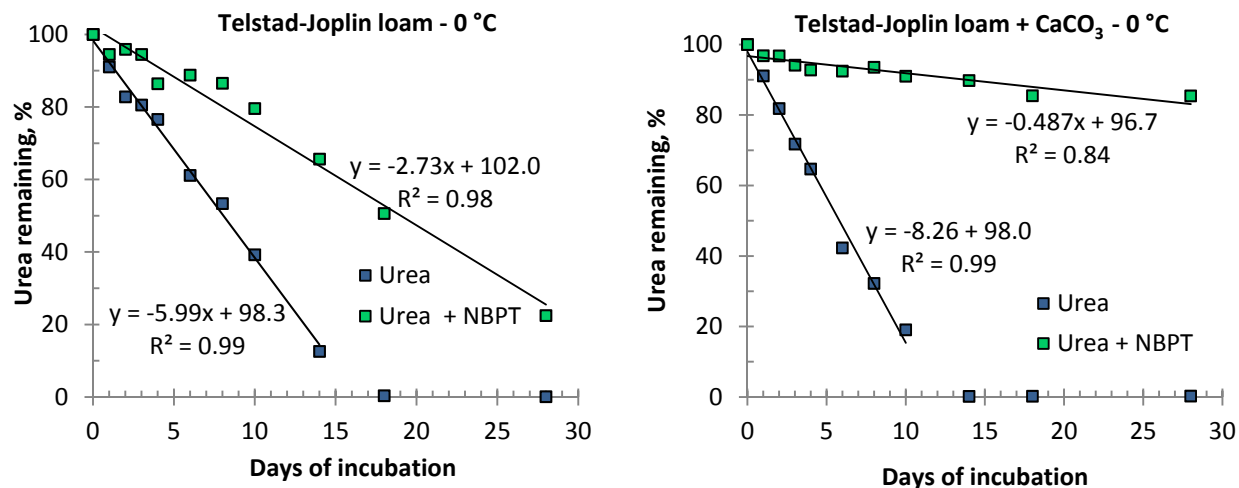


Fig. 7. Urea hydrolysis as affected by incubation time and NBPT in a Telstad-Joplin loam (pH 5.5) and Telstad-Joplin loam + CaCO₃ (pH 8.2).

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