

Losses of fertilizer nitrogen after a winter fertilization in three managed pine plantations of the southeastern United States

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Abstract

Losses of fertilizer nitrogen (N) were compared between urea and urea treated with the urease inhibitor N-(n-Butyl) thiophosphoric triamide (urea + NBPT) after a surface application in winter at three thinned mid-rotation (age 15–20 years) loblolly pine plantations in Virginia, South Carolina, and Florida. Treatments were labeled with ¹⁵N and applied to open chamber microcosms in January and February 2016. Fifteen days after application, microcosms were removed from the field to determine fertilizer N lost from each microcosm. Losses following fertilization with urea (24% to 50%) were greater ($p \geq .05$) at all sites compared to urea + NBPT (12% to 22%). Fertilizer N losses were greater in Florida than in SC and Virginia although N loss following urea fertilization was still 25% in Virginia. The loss of fertilizer N was consistently lower on beds compared to interbeds for both urea (bed = 25%, interbed = 40%) and urea + NBPT (bed = 12%, interbed = 23%). This research highlights the value of using urea + NBPT to reduce fertilizer N losses after a winter application and the greater potential loss in the interbed on wetter sites.

1 | INTRODUCTION

Nitrogen (N) is a critical nutrient for plant biochemical-physical development and processes, including photosynthesis (Gough, Seiler, & Maier, 2004), essential plant compound production (Koricheva, Larsson, Haukioja, & Keinanen, 1998), foliar growth (Albaugh, Allen, Dougherty, Kress, & King, 1998; Vose & Allen, 1988), and root development (King et al., 2002). Although most terrestrial systems contain large amounts of N (Fisher & Binkley, 2000), only a small proportion is available for plant uptake, resulting in N limitations to productivity (Chapin, Vitousek, & Van Cleve, 1986). In managed forests, N limitations are ameliorated by the addition of N-containing fertilizers, generally as a

broadcast application of granular urea ((NH₂)₂CO) (Allen, 1987; Fox, Allen, Albaugh, Rubilar, & Carlson, 2007a).

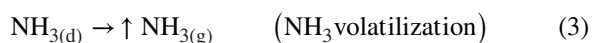
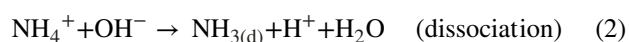
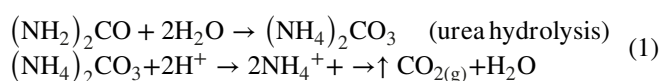
Urea application in managed loblolly pine (*Pinus taeda* L.) forests across the southeastern United States is an economically viable, sustainable silvicultural method to ameliorate N deficiencies due to a high N content, low processing cost, and high water solubility (Mulvaney & Bremner, 1981; Allen, 1987; Harre & Bridges, 1988). For example, a mid-rotation N application of 224 kg ha⁻¹ N with 28 kg ha⁻¹ phosphorous (P), another important co-limiting nutrient, generally translates to a growth increase of 3 m³ ha⁻¹ yr⁻¹ over 8 years (Carlson et al., 2014; Fox et al., 2007a). Although urea-N application generally increases productivity, there is large variation in the magnitude of response and may relate to the proportion and magnitude of fertilizer N lost from the ecosystem.

Loss of N following urea application depends on the complex interaction of environmental variables at the time of

Abbreviations: NBPT, N-(n-Butyl) thiophosphoric triamide.

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application (Raymond, Fox, Strahm, & Zerpa, 2016). Urea undergoes a series of reactions when applied to the surface of acidic, aerobic forest soils (Hauck & Stephenson, 1965). The first reaction, urea hydrolysis, has two stages (Equation [1]). In Stage 1, the urease enzyme facilitates the formation of ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$) (Conrad, 1942; Pettit, Smith, Freedman, & Burns, 1976; Marsh, Sims, & Mulvaney, 2005). In Stage 2, ammonium carbonate consumes hydrogen (H^+) ions, raising the pH, and ammonium (NH_4^+) ions form. In the second reaction, dissociation, NH_4^+ combines with a hydroxyl ion (OH^-) to form a dissolved ammonia ($\text{NH}_{3(d)}$) ion, an H^+ ion, and water (H_2O) (Equation [2]). After $\text{NH}_{3(d)}$ formation, $\text{NH}_{3(d)}$ can become a gas ($\text{NH}_{3(g)}$) and may be lost from the ecosystem to the atmosphere through diffusion, termed NH_3 volatilization (Equation [3]).



Edaphic variables correlated with urea-N loss via NH_3 volatilization include soil pH (Kissel et al., 2009), soil moisture (Kissel et al., 2004), soil temperature (Moyo, Kissel, & Cabrera, 1989), mineral soil versus forest floor substrate (Cabrera et al., 2005; Kissel et al., 2009; Zerpa & Fox, 2011), and soil buffering capacity (Fenn & Kissel, 1976; Ferguson, Koelliker, & Basel, 1984; Sunderlage & Cook, 2018). Climate variables correlated with applied urea-N loss via volatilization include relative humidity (Cabrera et al., 2005; Elliot & Fox, 2014), surficial wind speed (Kissel et al., 2004), precipitation (Craig & Wollum, 1982), and air temperature (Koelliker & Kissel, 1988). Although significant loss of fertilizer urea-N can occur with volatilization, tree canopies can absorb volatilized NH_3 and reduce applied urea-N losses (Nason, Pluth, & McGill, 1988). The quantification of urea-N loss from ecosystems through the volatilization pathway is difficult due to the interaction of these variables (Blazier, Hennessey, Dougherty, & Campbell, 2006). Urea applied in loblolly pine forests in the southeastern United States in cooler, wetter winter months would be expected to have lower urea-N losses due to rapid urea-N dissolution and movement into the soil (Black, Sherlock, & Smith, 1987; Cabrera et al., 2010; Ferguson & Kissel, 1986; Moyo et al., 1989; Paramasivam & Alva, 1997). However, large losses of urea-N can still occur under low temperatures, and winter application does not ensure low volatilization losses (Carmona & Byrnes, 1990; Engel, Jones, & Wallander, 2011).

In addition to N limitations, high water tables in poorly drained soils in the coastal plain of the southeastern United States create anaerobic conditions that also affect plantation

Core Ideas

- Urea + NBPT reduced fertilizer N loss (14.6%) after a winter fertilization compared to urea (30.0%).
- Reduced fertilizer N loss occurred from both bed and interbed areas when compared to urea.
- Losses in interbed areas (32.2%) were generally greater than bedded areas (17.6%).
- Fertilizer N loss with urea can be high even after a winter fertilization.
- Use of NBPT with urea in this situation can reduce fertilizer N loss in loblolly pine plantations.

forest productivity (McKee & Shoulders, 1970). Since the 1960s, bedding prior to planting has been a common site preparation treatment that creates rows of elevated microtopography in the stand that improve soil aeration (Allen, Fox, & Campbell, 2005; Burton, 1971; Fox, Jokela, & Allen, 2007b; Morris & Lowery, 1988; Smith, 1966) and leads to improved seedling survival and growth. Short-term height growth response to bedding for early forest development range from 0.9 to 2.7 meters (Gent, Allen, Campbell, & Wells, 1986; McKee & Shoulders, 1974; Terry & Hughes, 1975). The initial height gains results in increased volumes for mid-rotation pine plantation forests (Kyle et al., 2005). Soils in the raised bed are generally aerobic and unsaturated during most of the year while the interbed region can remain saturated and anaerobic. Because N application in plantation forests is through ground or aerial broadcast methods, the entire site is fertilized, not just bedded areas. On bedded sites, volatilization from the beds can be a large fertilizer N loss pathway due to aerobic, acidic conditions (Raymond et al., 2016). However, additional fertilizer N loss pathways such as leaching (Binkley, Burnham, & Allen, 1999), overland flow (Aust & Blinn, 2004), denitrification (Shrestha, Strahm, & Sucre, 2014) and dissimilatory N reduction (Minick, Pandey, Fox, & Subedi, 2016) may become more important under the saturated, anaerobic conditions found in the interbeds.

Enhanced efficiency N fertilizer products were developed to minimize N loss after fertilization (Azeem, KuShaari, Man, Basit, & Thanh, 2014; Goertz, 1993; Hauck, 1985) and can optimize plant N uptake (Shaviv, 1996). The compound N-(n-butyl) thiophosphoric triamide (NBPT) may be added to urea prior to application to reduce volatilization (Bremner & Douglas, 1971). Upon release from urea, NBPT binds with the urease enzyme prevalent in forest soils (Antisari, Marzadori, Gioacchini, Ricci, & Gessa, 1996; Bremner & Chai, 1986; Sanz-Cobena et al., 2008), delaying conversion of urea to ammonia and allowing time for urea to dissolve and move

into the soil where exchange sites can bind NH_4^+ (Bremner & Douglas, 1971). Although NBPT does reduce fertilizer N loss from loblolly pine plantations (Raymond et al., 2016), research has also shown soils with very low pH can lead to NBPT degradation and reduced efficacy (Engel, Towey, & Gravens, 2015; Forrester et al., 2016).

The primary objective of this research was to determine the efficacy of using urea treated with NBPT (urea + NBPT) compared to untreated urea to reduce fertilizer N loss in loblolly pine plantations in southeastern United States after a winter fertilization. Fertilizer N losses were also compared in bedded and non-bedded plantations. In the bedded plantations, fertilizer N losses were assessed in the beds and interbed areas. The hypotheses tested in this experiment were: (i) there was no difference in the amount of fertilizer N lost between urea and urea + NBPT after a winter fertilization, and (ii) there was no difference in the amount of fertilizer N lost on beds and interbeds after a winter fertilization.

2 | MATERIALS AND METHODS

2.1 | Study sites and treatments

This experiment used a complete block design, with each site serving as a block. Three mid-rotation (age 15–20 years) thinned loblolly pine forests with similar structural characteristics in Virginia, South Carolina, and Florida were selected along an ecophysiological gradient from an existing network of forest productivity trials to support long term research relating to thinning and fertilization along the eastern edge of southern loblolly pine plantations in the United States. Climatic data used in this study was obtained from the closest viable weather station to each site, whereas soil chemical and physical data were from analyses conducted at the Virginia Tech Soil Testing Laboratory.

The urea tested in this research was manufactured from the same material, a powdered 99% enriched ^{15}N urea. This initial powdered ^{15}N enriched urea was used to manufacture prilled urea with a falling curtain manufacturing process, where the 99% enriched ^{15}N urea was melted and sprayed onto a seed pellet multiple times to build up successive layers of ^{15}N urea until the desired granule size was obtained. The final mean ^{15}N urea enrichment for the prilled urea was 0.5 atom% and a mean delta value of 370‰. The granule size for the urea fertilizer treatment was approximately 5 mm, similar to that used in operational fertilization in forestry for the southeastern United States. The second fertilizer treatment, urea + NBPT, was treated with N-(n-Butyl) thiophosphoric triamide (NBPT). The NBPT was measured in the laboratory and stored in scintillation vials until application to the urea in the field at a rate typical for southern forestry of 1.25 kg Mg^{-1} . The previously measured NBPT was mixed with the urea in

a sealed plastic bag to ensure complete coating and impregnation of urea with NBPT, and then immediately applied to selected microcosms.

2.2 | Microcosm study design

Total fertilizer N loss was determined using open chamber microcosms (Marshall & Debell, 1980; May & Carlyle, 2005; Raymond et al., 2016). Microcosms were constructed from white Schedule 40 polyvinyl chloride pipe (PVC) with an inner diameter of 15.24 cm. Each microcosm was inserted vertically through the forest floor into the mineral soil to a depth of 30 cm, with a 2.5-cm lip extending above the forest floor. Six microcosms were installed at the flat-planted Virginia site whereas six microcosms were installed in both beds and interbeds at the South Carolina and Florida sites. At each site, three treatment replications were randomly applied by hand to the surface of the forest floor inside each microcosm on a single day in January or February 2016. Fifteen days after treatment application, individual microcosms containing soil were removed from the field. Covers were secured to the open ends of each intact microcosm, and the covered microcosms were placed on ice in coolers for transport to the laboratory. Microcosms were stored in a walk-in freezer with a constant temperature of -20°C until processed.

Once removed from the freezer, microcosms were thawed in a walk in refrigerator for 1 week and the soil in each microcosm was divided into three depth increments: forest floor (organic), 0–15 cm, and 15–30 cm (mineral soil). Forest floor samples were wet sieved through a 6 mm sieve, and mineral soil was wet sieved through a 2 mm sieve. After sieving, samples were dried in a forced air oven at 60°C for 1 week, and weighed. All sieved forest floor and sieved mineral soil samples were pulverized to a powder in a ball mill (Retsch GmbH Mixer Mill MM 200, Haan, Germany) for 2 min at 25 revolutions per second. After ball milling, 2 to 4 mg of forest floor sample and 40 to 45 mg of mineral soil subsamples were weighed in tin capsules on a Mettler-Toledo MX5 microbalance (Mettler-Toledo, LLC, Columbus, OH, USA). Between the weighing of each subsample, all instruments used during the weighing process were cleaned with an ethanol solution and allowed to dry to reduce contamination. All weighed subsamples in tin capsules were analyzed on a coupled elemental analysis-isotope ratio mass spectrometer (IsoPrime 100 EA-IRMS, Elementar Analysensysteme GmbH, Manchester, UK).

2.3 | $\delta^{15}\text{N}$ calculations

The recovery-loss of the fertilizer N treatments for each microcosm was determined by using an isotopic mass balance

TABLE 1 Climatic and site characteristics of three loblolly pine forests in the southeastern United States selected to evaluate fertilizer N loss following winter fertilization with urea and urea treated with NBPT

Site	Altitude m	Mean annual precipitation cm	Mean annual temperature °C	Physiographic region	Soil taxonomic class
Virginia	197	109	13	Southern Piedmont	Fine, mixed, subactive, mesic Typic Hapludults
South Carolina	2	125	17	Atlantic Coast Flatwoods	Fine, kaolinitic, thermic, Umbric Paleaquults
Florida	10	140	20	Atlantic Coast Flatwoods	Sandy, siliceous, thermic, Alfic Alaquods

equation adapted from Powlson and Barraclough (1993) and Nadelhoffer and Fry (1994) where:

$$M_{\text{FINAL}} = M_{\text{LABELED}} \frac{(\delta^{15}\text{N}_{\text{FINAL}} - \delta^{15}\text{N}_{\text{INITIAL}})}{(\delta^{15}\text{N}_{\text{LABELED}} - \delta^{15}\text{N}_{\text{INITIAL}})} \quad (4)$$

M_{FINAL} is the mass of the fertilizer N in the soil depth increment N pool; M_{LABELED} is the mass of the $\delta^{15}\text{N}$ recovered from the microcosm; $\delta^{15}\text{N}_{\text{INITIAL}}$ is the natural ^{15}N abundance of the soil depth increment pool; $\delta^{15}\text{N}_{\text{LABELED}}$ is the ^{15}N abundance of the added fertilizer N; and $\delta^{15}\text{N}_{\text{FINAL}}$ is the final ^{15}N abundance of the soil depth increment from the fertilizer N. Equations 4 through 8 were used to calculate the percentage of fertilizer N recovered from each microcosm. The percent (%) fertilizer N in each soil depth increment was calculated as:

$$\left[\frac{(\delta^{15}\text{N}_{\text{FINAL}} - \delta^{15}\text{N}_{\text{INITIAL}})}{(\delta^{15}\text{N}_{\text{LABELED}} - \delta^{15}\text{N}_{\text{INITIAL}})} \right] * 100 \quad (5)$$

The percentage (%) of ^{15}N from labeled material in each soil depth increment was calculated:

$$\left[\frac{(\delta^{15}\text{N}_{\text{SOILDEPTHINCREMENT}} - \delta^{15}\text{N}_{\text{INITIAL}})}{(\delta^{15}\text{N}_{\text{LABELED}} - \delta^{15}\text{N}_{\text{INITIAL}})} \right]$$

where $\delta^{15}\text{N}_{\text{SOILDEPTHINCREMENT}}$ is the ^{15}N abundance of individual soil depth increments.

The total N (g) for each soil depth increment was calculated as:

$$(\text{Soil depth increment dry weight} * \text{mg g}^{-1}\text{N}) / 100 \quad (6)$$

The amount of N derived from the fertilizer applied N treatment (g) in each soil depth increment was calculated as:

$$(\text{Total N} * \%^{15}\text{N derived from fertilizer N}) / 100 \quad (7)$$

2.4 | Statistical analysis

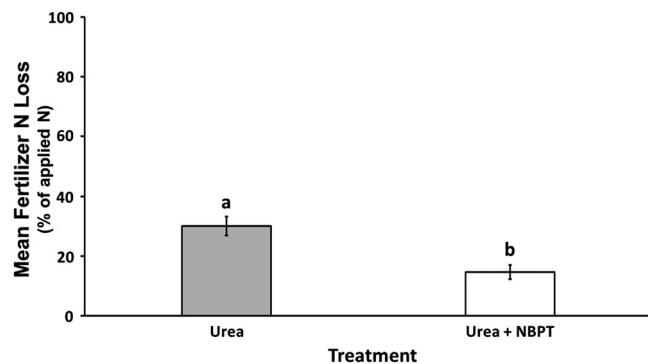
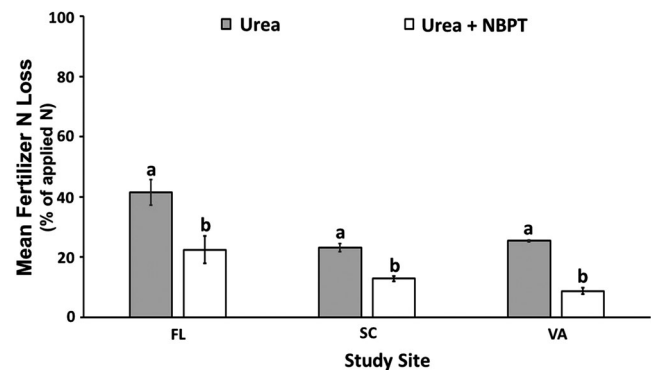
We tested whether differences occurred for fertilizer N loss between urea and urea + NBPT at the three locations. To test differences in fertilizer N loss at the three locations, we analyzed data from all microcosms at the nonbedded site (Virginia), and only microcosm data from the beds at the two bedded sites (South Carolina, Florida). Additionally, at the two bedded sites in South Carolina and Florida, we also tested whether there were differences in fertilizer N loss for microsites (bed vs. interbed). Therefore, the fertilizer N loss (%), expressed as a percentage of fertilizer N not recovered from an individual microcosm, was analyzed using an analysis of variance (PROC MIXED, SAS Institute, Cary, NC), with treatment and bed/interbed as main effects. Percent data were arc-sine transformed prior to analysis to conform to normal distribution. All data were plotted as means of raw data. If significant main effects or interactions were detected in the analysis of variance, these differences were additionally tested using the Tukey Honestly Significant Difference (HSD) test. All reported levels of significant are at $p \geq .05$.

3 | RESULTS

Selected climatic and site characteristics are detailed in Table 1. Climatic data used in Table 1 was from the closest viable weather station to each site. Selected soil chemical and physical properties are detailed in Table 2. Soil chemical and physical data used in Table 2 were from analyses conducted at the Virginia Tech Soil Testing Laboratory. The mean fertilizer N loss, expressed as a percentage of N applied to individual microcosms, was greater for urea compared to urea + NBPT (Figure 1). Fifteen days after N application, the mean fertilizer N loss from microcosms was 30.0% for urea and 14.6% for urea + NBPT. Losses for urea were greatest at the Florida site (41.5%), followed by South Carolina (23.1%) and Virginia (25.4%) (Figure 2). Losses for urea + NBPT followed a similar trend to urea among sites although losses were significantly lower at all three sites. For urea + NBPT, fertilizer N

TABLE 2 Physical and chemical soil characteristics of three loblolly pine forests in the southeastern United States selected to evaluate fertilizer N loss following winter fertilization with urea and urea treated with NBPT

Site	Depth	Bulk density	CEC	pH (H ₂ O)	P	K	Ca	Mg	Zn	Mn	Cu	Fe	B	Total N	Total C
	cm	g cm ⁻³	cmol(+) kg ⁻¹				mg kg ⁻¹ (Mehlich I extractable)							g kg ⁻¹	
Virginia	Forest Floor													15.3	315.9
	0–15	1.24	6.4	4.2	3.0	39.3	68.3	14.7	0.5	7.1	0.4	62.7	0.1	1.7	24.9
	15–30	1.32	5.3	4.3	2.3	29.7	44.0	11.7	0.3	4.0	0.6	16.9	0.1	0.5	7.1
	30–50	1.45	5.8	4.4	2.0	39.0	46.0	12.7	0.3	2.4	0.3	29.8	0.1	0.4	8.4
South Carolina	Forest Floor													7.1	252.6
	0–15	1.32	7.1	4.4	4.0	17.0	147.0	24.0	0.4	3.6	0.6	34.9	0.1	3.4	28.6
	15–30	1.35	7.8	4.5	6.0	18.0	217.0	30.0	0.7	2.2	0.5	34.7	0.1	1.2	27.6
	30–50	1.38	7.5	4.6	4.0	16.0	153.0	31.0	0.7	1.1	0.4	75.4	0.1	3.8	13.9
Florida	Forest Floor													5.2	189.4
	0–15	1.28	7.2	3.6	3.0	11.0	113.0	19.0	0.4	0.5	0.1	8.2	0.1	1.5	20.1
	15–30	1.28	4.4	3.7	2.0	8.0	67.0	10.0	0.1	1.7	0.1	7.4	0.1	0.4	5.2
	30–50	1.53	3.9	4.1	1.9	6.0	54.0	6.0	0.1	1.4	0.1	5.4	0.1	0.2	3.9

**FIGURE 1** Mean fertilizer N loss from microcosms, expressed as a percent of applied N, for three thinned mid-rotation loblolly pine forests in the southeastern United States selected to evaluate fertilizer N loss following the winter application of the ¹⁵N enriched treatments urea and urea treated with NBPT (urea + NBPT). Different letters are significant differences at $\alpha = .05$, and error bars are the standard error of the mean ($n = 3$)**FIGURE 2** Mean fertilizer N loss from microcosms, expressed as a percent of applied N, for three thinned mid-rotation loblolly pine forests in the southeastern United States (FL, Florida; SC, South Carolina; VA, Virginia) selected to evaluate fertilizer N loss following the winter application of the ¹⁵N enriched treatments urea and urea treated with NBPT (urea + NBPT). Analysis is between treatments for each individual site. Different letters are significant differences at $\alpha = .05$ at individual sites, and error bars are the standard error of the mean ($n = 3$)

losses decreased to 22.4% in Florida, 12.8% in South Carolina and 8.7% in Virginia.

At the two bedded sites in Florida and South Carolina, the mean fertilizer N losses was greater for interbeds compared to beds. Fifteen days after N application, the mean fertilizer N loss from microcosms was 32.3% for interbeds compared to 17.6% for beds. The addition of NBPT to the urea decreased the fertilizer N losses in both the beds and interbeds (Figure 3). Fifteen days after N application, the mean fertilizer N loss in the beds for urea + NBPT was 12.3% compared to 22.9% in the interbeds. For urea, fertilizer N loss in beds was 25.1% compared to 39.5% in the interbeds.

4 | DISCUSSION

We rejected our first hypothesis because fertilizer N losses were significantly lower for urea + NBPT (14.6%) compared to urea (30.0%). The mean fertilizer N loss for urea + NBPT ranged from 8.0–29.8% compared to 24.3–49.5% for urea 15 days after a winter application. This result is similar to other studies in forested systems that have found large losses of fertilizer N when urea was applied (Elliot & Fox, 2014; Kissel et al., 2009; Raymond et al., 2016). The use of

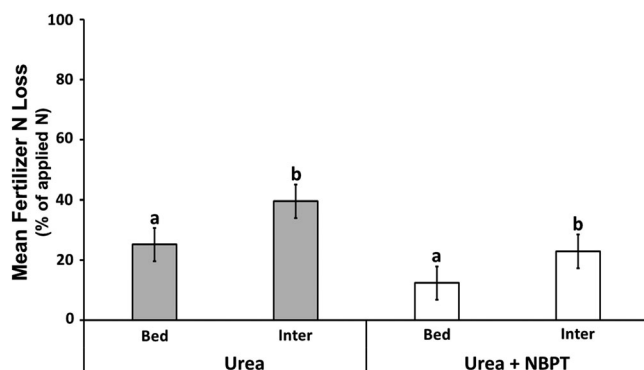


FIGURE 3 Differences in the mean fertilizer N loss, expressed as a percent of applied N, for urea and urea treated with NBPT, between beds and interbeds from microcosms, for two three thinned mid-rotation loblolly pine forests (South Carolina, Florida) in the southeastern United States selected to evaluate fertilizer N loss following the winter application of the ^{15}N enriched treatments urea and urea treated with NBPT (urea + NBPT). Different letters are significant differences at $\alpha = .05$ at individual sites, and error bars are the standard error of the mean ($n = 3$)

enhanced efficiency fertilizers significantly decreased fertilizer N losses in all of these studies.

Lower fertilizer N losses have been reported when urea was applied under wetter and cooler conditions (Cabrera et al., 2010; Ferguson & Kissel, 1986; Moyo et al., 1989) due to rapid urea dissolution (Black et al., 1987; Paramasivam & Alva, 1997) and is the rationale for applying urea in the winter in managed pine forests of the southeastern United States. It was thought that cooler and wetter conditions in the winter would lower the risk fertilizer N loss. However, this study demonstrates that large losses of urea-N still occurs after a winter fertilization in loblolly pine forests in the southeastern United States. A similar study in loblolly pine forests during the spring and summer months by Raymond et al. (2016) found comparable fertilizer N losses for urea N compared to enhanced efficiency products. Climatic conditions conducive to fertilizer N loss after urea application can occur any time of year across the southeastern United States. Large volatilization losses have been reported in agricultural systems even when temperatures are near freezing (Carmona & Byrnes, 1990; Engel et al., 2011). Yet volatilization losses observed in our study were lower when urea was treated with NBPT even in the winter. This finding suggests there is value in using urease inhibitors, such as NBPT, at all times of the year when fertilizing with urea. This is true from an ecological point of view, but the economical convenience must also be considered.

We rejected our second hypothesis because we observed significant differences in N loss on beds and interbeds. Fifteen days after N application, the mean fertilizer N loss from

microcosms was 32.3% for interbeds compared to 17.6% for beds. Volatilization can be a primary loss pathway for fertilizer N under the acidic, aerobic conditions present for a large portion of the year in forest soils supporting managed pine forests across the southeastern United States. The addition of the urease inhibitor NBPT to the urea fertilizer decreased fertilizer losses on both the bed and interbed areas in this study, suggesting volatilization was an important loss mechanism in both locations. In the present study although ammonia volatilization, nitrous oxide loss and nitrate leaching were not quantified, some considerations regarding those process are mentioned ahead. Fertilizer N loss was approximately 10% to 13% greater in the interbed areas compared to the beds, regardless of whether a urease inhibitor was applied. This finding suggests that loss mechanisms other than volatilization may be occurring on these poorly drained soils. Based on Natural Resource Conservation Service Soil Survey Geographic Database (NRCS SSURGO) drainage class data, and an estimate of loblolly pine coverage in the southeast (Zhang & Roy, 2017), we estimated that 35% of soils under loblolly pine are very poorly, poorly, or somewhat poorly drained. Potential mechanisms for loss from the microcosms include plant uptake, leaching, or denitrification. It is unlikely that plant uptake was a major component of fertilizer N loss from the microcosm because plant roots were severed when the microcosms were installed, and root uptake and growth is minimized during winter months. It is uncertain whether leaching or denitrification were significant loss pathways from the microcosms. Although the majority of ^{15}N recovered in individual microcosms was in the upper soil profile (forest floor, 0–15 cm) and not in the 15–30 cm soil depth increment, we did not specifically measure either leaching or denitrification in this experiment and are unable to distinguish which loss mechanism was more significant in relation to fertilizer N loss.

Denitrification is more likely to occur in the interbed locations on these poorly drained soils where anaerobic conditions would occur than on the elevated beds where surface soils remain aerobic (Kelting, Burger, & Edwards, 1998). A study in a similar managed forest in the southeastern United States by Shrestha et al. (2014) found losses due to denitrification after a winter N application were greater when compared to spring and summer months. Denitrification in aerobic forest soils are transient and stimulated by precipitation (Sexstone, Parkin, & Tiedje, 1985). Denitrification in aerobic soils is less than that observed in anaerobic and saturated soils (Zak & Grigal, 1991; Kuzyakov & Blagodatskay, 2015). Given the different microenvironments on beds and interbeds during winter months, it is probable that the majority of fertilizer N loss on beds is from volatilization (Raymond et al., 2016) whereas interbed fertilizer N loss is a combination of volatilization and denitrification (Kelting et al., 1998).

5 | CONCLUSIONS

Results from this study show fertilizer N losses following application of urea can be significant during winter in pine plantations in the southeastern United States. These losses can be reduced by using a urease inhibitor such as NBPT under these same conditions.

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REFERENCES

- Albaugh, T. J., Allen, H. L., Dougherty, P. M., Kress, L. W., & King, J. S. (1998). Leaf-area and above- and belowground growth responses of loblolly pine to nutrient and water additions. *Forest Science*, 44, 317–328.
- Allen, H. L. (1987). Forest fertilizers: Nutrient amendment, and productivity, and environmental impact. *Journal of Forestry*, 85, 37–46.
- Allen, H. L., Fox, T. R., & Campbell, R. G. (2005). What's ahead for intensive pine plantation silviculture in the South? *Southern Journal of Applied Physics*, 29, 62–69.
- Antisari, L. V., Marzadori, C., Gioacchini, P., Ricci, S., & Gessa, C. (1996). Effects of the urease inhibitor N-(n-butyl) phosphorothioic triamide in low concentrations on ammonia volatilization and evolution of mineral nitrogen. *Biology and Fertility of Soils*, 22, 196–201.
- Aust, W. M., & Blinn, C. R. (2004). Forestry best management practices for timber harvesting and site preparation in the eastern United States: An overview of water quality and productivity research during past 20 years. *Water Air Soil Pollution Focus (San Francisco, CA)*, 4, 5–36.
- Azeem, B., KuShaari, K., Man, Z. B., Basit, A., & Thanh, T. H. (2014). Review on materials and methods to produce controlled release coated urea fertilizer. *Journal of Controlled Release*, 181, 11–21.
- Binkley, D., Burnham, H. & Allen, H. L. (1999). Water quality impacts of forest fertilization with nitrogen and phosphorous. *Forest Ecology and Management*, 121, 191–213.
- Black, A. S., Sherlock, R. R., & Smith, N. P. (1987). Effect of timing of simulated rainfall on ammonia volatilization from urea applied to soil of varying moisture content. *Journal of Soil Science*, 38, 679–687.
- Blazier, M. A., Hennessey, T. C., Dougherty, P., & Campbell, R. (2006). Nitrogen accumulation and use by a young loblolly pine plantation in southeast Oklahoma: Effects of fertilizer formulation and date of application. *Southern Journal of Applied Physics*, 30, 66–78.
- Bremner, J. M., & Douglas, L. A. (1971). Inhibition of urease activity in soils. *Soil Biology and Biochemistry*, 3, 297–307.
- Bremner, J. M., & Chai, H. S. (1986). Evaluation of N-butyl phosphorothioic triamide for retardation of urea hydrolysis in soil. *Communications in Soil Science and Plant Analysis*, 17, 337–351.
- Burton, J. D. 1971. Prolonged flooding inhibits growth of loblolly pine seedlings. US For. Serv. Res. Note SO-124. New Orleans, LA: Southern Forest Exp. Stn., 4 p.
- Cabrera, M. L., Kissel, D. E., Vaio, N., Craig, J. R., Rema, J. A., & Morris, L. A. (2005). Loblolly pine needles retain urea fertilizer that can be lost as ammonia. *Soil Science Society of America Journal*, 69, 1525–1531.
- Cabrera, M. L., Kissel, D. E., Craig, J. R., Qafoku, N. P., Vaio, N., Rema, J. A., & Morris, L. A. (2010). Relative humidity controls ammonia loss from urea applied to loblolly pine. *Soil Science Society of America Journal*, 74, 543–549. <https://doi.org/10.2136/sssaj2009.0220>.
- Carlson, C. A., Fox, T. R., Allen, H. L., Albaugh, T. J., Rubilar, R. A., & Stape, J. L. (2014). Growth responses of loblolly pine in the south-east united states to midrotation applications of nitrogen, phosphorus, potassium, and micronutrients. *Forest Science*, 60, 157–169.
- Carmona, G.C., & Byrnes, B. H. (1990). Temperature and low concentration effects of the urease inhibitor N-(n-butyl) thiophosphoric triamide (NBPT) on ammonia volatilization from urea. *Soil Biology and Biochemistry*, 22, 933–937. [https://doi.org/10.1016/0038-0717\(90\)90132-](https://doi.org/10.1016/0038-0717(90)90132-).
- Chapin, F. S., Vitousek, P. M., & Van Cleve, K. (1986). The nature of nutrient limitation in plant-communities. *American Naturalist*, 127, 48–58. <https://doi.org/10.1086/284466>.
- Conard, J. P. (1942). The occurrence and origin of urease like activities in soils. *Soil Science*, 54, 367–380.
- Craig, J., & Wollum, A. (1982). Ammonia volatilization and soil nitrogen changes after urea and ammonium nitrate fertilization of *Pinus taeda* L. *Soil Science Society of America Journal*, 46, 409–414. <https://doi.org/10.2136/sssaj1982.03615995004600020040x>.
- Elliot, J. R., & Fox, T. R. (2014). Ammonia volatilization following fertilization with urea or ureaform in a thinned loblolly pine plantation. *Soil Science Society of America Journal*, 78, 1469–1473. <https://doi.org/10.2136/sssaj2013.12.0512n>.
- Engel, R., Jones, C., & Wallander, R. (2011). Ammonia volatilization from urea and mitigation by NBPT following surface application to cold soils. *Soil Science Society of America Journal*, 75, 2348–2357. <https://doi.org/10.2136/sssaj2011.0229>.
- Engel, R.E., & Towey, Bradley, & Gravens, Emily (2015). Degradation of the urease inhibitor NBPT as affected by soil pH. *Soil Science Society of America Journal*, 79, 1674–1683. <https://doi.org/10.2136/sssaj2015.05.0169>.
- Fenn, L. B., & Kissel, D. E. (1976). The influence of cation exchange capacity and depth of incorporation on ammonia volatilization from ammonium compounds applied to calcareous soils. *Soil Science Society of America Journal*, 40, 394–398.
- Ferguson, R. B., Kissel, D.E., Koelliker, J. K., & Basel, W. S. (1984). Ammonia volatilization from surface applied urea: Effect of hydrogen ion buffering capacity. *Soil Science Society of America Journal*, 48, 578–582.
- Ferguson, R. B., & Kissel, D. E. (1986). Effects of soil drying on ammonia volatilization from surface-applied urea. *Soil Science Society of America Journal*, 50, 435–490.
- Fisher, R. F., & Binkley, D. (2000). *Ecology and management of forest soils*. New York: Wiley.
- Forrestal, P. J., Harty, M., Carolan, R., Lanigan, G. J., Watson, C. J., Laughlin, R. J., ... Richards, K. G. (2016). Ammonia emissions from urea, stabilized urea and calcium ammonium nitrate: Insights into loss abatement in temperate grassland. *Soil Use and Management*, 32, 92–100.
- Fox, T. R., Allen, H. L., Albaugh, T. J., Rubilar, R., & Carlson, C. A. (2007a). Tree nutrition and forest fertilization of pine plantations in the southern United States. *Southern Journal of Applied Forestry*, 31, 5–11.
- Fox, T. R., Jokela, E. J., & Allen, H. L. (2007b). The development of pine plantation silviculture in the southern United States. *Journal of Forestry*, 105, 337–347.
- Gent, J. A., Allen, H. L., Campbell, R. G., & Wells, C. G. (1986). Magnitude, duration, and economic analysis of loblolly pine growth

- response following bedding and phosphorus fertilization. *Southern Journal of Applied Forestry*, 10, 124–128.
- Goertz, H. M. 1993. Controlled Release Technology. Kirk-Othmer Encyclopedia of chemical technology, Vol.7. *Controlled Release Technology (Agricultural)*, 251–274.
- Gough, C. M., Seiler, J. R., & Maier, C. A. (2004). Short-term effects of fertilization on loblolly *Pinus taeda* pine (L.) physiology. *Plant, Cell and Environment*, 27, 876–886. <https://doi.org/10.1111/j.1365-3040.2004.01193.x>.
- Harre, E. A., & Bridges, J. D. 1988. Importance of urea fertilizers. In Bock, B. R., et al. (Eds.), *Ammonia volatilization from urea fertilizers*. Bull. Y- 206. Muscle Shoals, AL: National Fertilizer Development Center, Tennessee Valley Authority. 15 pp.
- Hauck, R. D. (1985). Slow release and bio-inhibitor-amended nitrogen fertilizers. In Engelstad O. P. (Ed.), *Fertilizer technology and use* (3rd Ed, pp. 293–322). Madison, WI: ASA.
- Hauck, R. D., & Stephenson, H. F. (1965). Nitrification of nitrogen fertilizers. Effect of nitrogen source, size, and pH of the granule, and concentration. *Journal of Agricultural and Food Chemistry*, 13, 486–492.
- Kelting, D. L., Burger, J. A., & Edwards, G. S. (1998). Estimating root respiration, microbial respiration in the rhizosphere, and root-free soil respiration in forest soils. *Soil Biology and Biochemistry*, 30, 961–968.
- King, J. S., Albaugh, T. J., Allen, H. L., Buford, M., Strain, B. R., & Dougherty, P. (2002). Below-ground carbon input to soil is controlled by nutrient availability and fine root dynamics in loblolly pine. *New Phytologist*, 154, 389–398.
- Kissel, D., Cabrera, M., Vaio, N., Craig, J., Rema, J., & Morris, L. (2004). Rainfall timing and ammonia loss from urea in a loblolly pine plantation. *Soil Science Society of America Journal*, 68, 1744–1750. <https://doi.org/10.2136/sssaj2004.1744>.
- Kissel, D., Cabrera, M. L., Vaio, N., Craig, J. R., Rema, J. A., & Morris, L. A. (2009). Forest floor composition and ammonia loss from urea in a loblolly pine plantation. *Soil Science Society of America Journal*, 73, 630–637. <https://doi.org/10.2136/sssaj2007.0377>.
- Koelliker, J. K., and Kissel, D. E. 1988. Chemical equilibria affecting ammonia volatilization. In: Bock, B.R., et al. (Eds.), *Ammonia volatilization from urea fertilizers*. (pp. 37–52) Bull. Y- 206. Muscle Shoals, AL: National Fertilizer Development Center, Tennessee Valley Authority.
- Koricheva, J., Larsson, S., Haukioja, E., & Keinanen, M. (1998). Regulation of woody plant secondary metabolism by resource availability: Hypothesis testing by means of meta-analysis. *Oikos*, 83, 212–226.
- Kuzyakov, Y., & Blagodatskay, E. (2015). Microbial hotspots and hot moments in soil: Concept and review. *Soil Biology and Biochemistry*, 83, 184–189.
- Kyle, K. H., Andrews, L. J., Fox, T. R., Aust, W. M., Burger, J. A., & Hansen, G. H. (2005). Long-term impact of drainage, bedding, and fertilization on growth of loblolly pine (*Pinus taeda* L.) in the Coastal Plain of Virginia. *Southern Journal of Applied Forestry*, 29, 205–214.
- Marsh, K. L., Sims, G. K., & Mulvaney, R. L. (2005). Availability of urea to autotrophic ammonia-oxidizing bacteria as related to the fate of ^{14}C and ^{15}N labeled urea added to soil. *Biology and Fertility of Soils*, 42, 137–145. <https://doi.org/10.1007/s00374-005-0004-2>.
- Marshall, V., & Debell, D. (1980). Comparison of four methods of measuring volatilization losses of nitrogen following urea fertilization of forest soils. *Canadian Journal of Soil Science*, 60, 549–563.
- May, B. M., & Carlyle, J. C. (2005). Nitrogen volatilization from urea fertilizer in mid-rotation *Pinus radiata* plantations in southeastern Australia. *Australian Forestry*, 68, 20–26. <https://doi.org/10.1080/00049158.2005.10676222>.
- McKee, W. H. Jr., & Shoulders, E. (1970). Depth of water table and redox potential of soil affect slash pine growth. *Forest Science*, 16, 399–402.
- McKee, W. H. Jr., & Shoulders, E. (1974). Slash pine biomass response to site preparation and soil properties. *Soil Science Society of America, Proceedings*, 38, 144–148.
- Minick, K. J., Pandey, C. B., Fox, T. R., & Subedi, S. (2016). Dissimilatory nitrate reduction to ammonium and N_2O flux: Effect of soil redox potential and N fertilization in loblolly pine forests. *Biology and Fertility of Soils*, 52, 601–614. <https://doi.org/10.1007/s00374-016-1098-4>
- Morris, L. A., & Lowery, R. F. (1988). Influences of site preparation on soil conditions affecting stand establishment and tree growth. *Southern Journal of Applied Forestry*, 12, 170–178.
- Moyo, C. C., Kissel, D., & Cabrera, M. L. (1989). Temperature effects on soil urease activity. *Soil Biology and Biochemistry*, 21, 935–938. [https://doi.org/10.1016/0038-0717\(89\)90083-7](https://doi.org/10.1016/0038-0717(89)90083-7).
- Mulvaney, R. L., & Bremner, J. M. (1981). Controls of urea transformations in soils. In Paul, E. A., & Ladd, J.N. (Eds.), *Soil biochemistry* (pp. 153–196). New York: Marcel Dekker.
- Nadelhoffer, K. J., & Fry, B. (1994). Nitrogen isotope studies in forest ecosystems. In Lajtha, K., & Michener, R. H. (Eds.), *Stable isotopes in ecology and environmental science* (pp. 22–44). New York, NY: Blackwell Scientific Publications.
- Nason, G. E., Pluth, D. J., & McGill, W. B. (1988). Volatilization and foliar recapture of ammonia following spring and fall application of nitrogen-15 urea to a Douglas-fir ecosystem. *Soil Science Society of America Journal*, 52, 821–828.
- Paramasivam, S., & Alva, A. K. (1997). Leaching of nitrogen forms from controlled release fertilizers. *Communications Soil Science and Plant Analysis*, 28, 1663–1674.
- Pettit, N. M., Smith, A. R. J., Freedman, R. B., & Burns, R. G. (1976). Soil urease: Activity, stability and kinetic properties. *Soil Biology and Biochemistry*, 8, 479–484.
- Powlson, D. A., & Barraclough, D. (1993). Mineralization and assimilation in soil-plant systems. In Knowles, R., & Blackburn, H. (Eds.), *Nitrogen isotope techniques* (pp. 209–221). San Diego, CA: Academic Press.
- Raymond, J. E., Fox, T. R., Strahm, B. D., & Zerpa, J. (2016). Ammonia Volatilization following nitrogen fertilization with enhanced efficiency fertilizers and urea in loblolly pine plantations of the southern United States. *Forest Ecology and Management*, 376, 247–255.
- Sanz-Cobena, A., Misselbrook, T. H., Arce, A., Mingot, J. I., Diez, J. A., & Vallejo, A. (2008). An inhibitor of urease activity effectively reduces ammonia emissions from soil treated with urea under Mediterranean conditions. *Agriculture, Ecosystems and Environment*, 126, 243–249.
- Sexstone, A. J., Parkin, T. B., & Tiedje, J.M. (1985). Temporal response of soil denitrification rates to rainfall and irrigation. *Soil Science Society of America Journal*, 49, 99–103.
- Shaviv, A. (1996). Plant response and environmental aspects as affected by rate and pattern of nitrogen release from controlled release N fertilizers. In Van Cleemput, O. (Ed.), *Progress in nitrogen cycling studies* (pp. 285–291). Netherlands: Kluwer.

- Shrestha, R., Strahm, B. D., & Sucre, E. B. (2014). Nitrous oxide fluxes in fertilized *Pinus taeda* plantations across a gradient of soil drainage classes. *Journal of Environmental Quality*, 43, 1823–1832.
- Smith, L. F. (1966). Site preparation and cultivation improve survival and growth of planted slash pine. *Tree Plant. Notes*, 64, 12–15.
- Sunderlage, B., & Cook, R. L. (2018). Soil property and fertilizer additive effects on ammonia volatilization from urea. *Soil Science Society of America Journal*, 82, 253–259.
- Terry, T. A., & Hughes, J. H. (1975). The effects of intensive management on planted loblolly pine (*Pinus taeda* L.) growth on poorly drained soils of the Atlantic Coastal Plain. In Bernier, B., & Winget, C. H. (Eds.), *Forest soils and forest land management* (pp. 351–377). Canada: Les Presses de L'Universite Laval Quebec.
- Vose, J. M., & Allen, H. L. (1988). Leaf area, stemwood growth, and nutrition relationships in loblolly pine. *Forest Science*, 34, 547–563.
- Zerpa, J. L., & Fox, T. R. (2011). Controls on volatile NH_3 losses from loblolly pine plantations fertilized with urea in the southeast USA. *Soil Science Society of America Journal*, 75, 257–266. <https://doi.org/10.2136/sssaj2010.0101>.
- Zak, D., & Grigal, D. (1991). Nitrogen mineralization, nitrification, and denitrification in upland and wetland ecosystems. *Oecologia*, 88, 189–196.
- Zhang, H. K., & Roy, D. P. (2017). Using the 500 m MODIS land cover product to derive a consistent continental scale 30 m Landsat land cover classification. *Remote Sensing of Environment*, 197, 15–34.

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