Differences in the recovery of four different nitrogen containing fertilizers after two application seasons in pine plantations across the southeastern United States

1. Introduction

Loblolly pine (*Pinus taeda* L.) is the most widely planted tree species in the United States (USDA 2012), ranging from southeastern Oklahoma and east Texas to north Florida and southern New Jersey (Shultz 1997). Loblolly pine plantations are often intensively managed through site preparation, the planting of improved genetic stock, competition control, and fertilization (Fox et al. 2007a). These combined treatments often double pine growth, with many stands capable of producing more than $10 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ in total stem volume (Fox et al. 2007a). One primary constraint limiting growth in many southeastern pine plantations is an inadequate supply of plant available nutrients, specifically nitrogen (N) and phosphorous (P) (Allen 1987, Fox et al. 2007b). Low levels of plant available N in the soil are particularly common in these pine plantations which restricts leaf area production, decreases the photosynthetic capacity of the stand, and translates to low net primary productivity (Miller 1981, Linder 1987).

Nitrogen requirements for pine stands increase with age (Switzer and Nelson 1972, Wells and Jorgenson 1979, Fox et al. 2007a) and nutrient limitations to growth may occur as demand for nutrients by trees exceeds the available nutrient supply in the soil (Wells and Jorgenson 1979, Allen et al. 1990). At stand initiation, harvesting and site preparation activities increase soil N availability to plants through increased decomposition of soil organic matter and thus increased N mineralization (Vitousek and Matson 1985, Fox et al. 1986). As a stand ages, plant N availability declines in the soil because of increased N immobilization in the ecosystem (Birk and Vitousek 1986). To ameliorate reduced plant N availability, N fertilization may be required to maintain forest productivity (Miller 1981). On average, southeastern pine plantation

total stem volume growth increases $3 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ up to 8 years following a mid-rotation application of $224 \text{ kg ha}^{-1} \text{ N}$ plus $28 \text{ kg ha}^{-1} \text{ P}$ (Fox et al. 2007a). These results have led to the annual fertilization of between 300,000 ha to 350,000 ha annually of pine plantations in the southeastern United States between 2005 and 2015 (FPC 2015). However, certain sites do not exhibit a growth response or respond negatively to fertilization (Pritchett and Smith 1975, Martin et al. 1999, Amateis et al. 2000, Carlson et al. 2014) and these inconsistent fertilizer trial results have led to a wide range of responses in pine plantations (Rojas 2005, Fox et al. 2007a).

Despite the large quantity of N in forest soils supporting pine plantations in the southeastern United States, ranging from 2 Mg N ha^{-1} to 7 Mg N ha^{-1} , only a small fraction of this soil N ($< 2\%$) is immediately available for plant uptake (Fisher and Binkley 2000). Such large soil N stocks make it difficult to track the fate of N originating from fertilizer trials, which typically only adds 150 kg N ha^{-1} to 250 kg N ha^{-1} (Fisher and Binkley 2000, Flint 2007, Kiser and Fox 2014). One technique to distinguish the fate of fertilizer N in the environment is to use N containing fertilizers enriched with the stable isotope ^{15}N . These isotopically enriched fertilizers can be used to trace the fate of fertilizer N in the ecosystem to improve our understanding of the mechanisms driving the cycling of applied N (Hauck and Bystrom 1971, Mead and Pritchett 1975, Melin et al. 1983, Preston et al. 1990, Nadelhoffer and Fry 1994, Chang et al. 1996, Bubba et al. 1999, Currie and Nadelhoffer 1999, Nadelhoffer et al. 2004, Templar et al. 2012, Swanston and Preston 2014).

Although fertilization generally improves the growth of most southeastern pine plantations, N uptake by desired crop trees is generally less than 25% of the applied fertilizer N (Mead and Pritchett 1975, Johnson and Todd 1988, Albaugh et al. 2004, Blazier et al. 2006). Explanations for low fertilizer N uptake in field studies include: 1) fertilizer N loss from the system; 2) fertilizer N immobilization; and 3) application of fertilizer N asynchronous with seasonal plant N demand. An improved understanding of the mechanisms driving fertilizer N

cycling and retention in southeastern pine plantation ecosystems is required to enhance fertilizer nitrogen use efficiency (FNUE) to continue to increase productivity, economic viability, and reduce potential negative environmental impacts associated with fertilization in agroecosystems moving into the future (Zhang et al. 2015).

To address the economic and environmental issues associated with fertilization in agroecosystems, enhanced efficiency fertilizers (EEFs) were developed. The Fertilizer Institute (2015) defines EEFs as fertilizer products that reduce nutrient loss to the environment, increase plant nutrient availability by slowing nutrient release, and conversion of nutrients to those less susceptible to losses. Nitrogen containing EEFs are designed to address the specific problems associated with applied N: 1) increase crop uptake; 2) decrease leaching; 3) lower toxicity; 4) provide extended supply; 5) reduce volatilization and/or nitrification losses; and 6) lower application cost (Allen 1984, Hauck 1985). EEFs can broadly be divided into slow release (SRN), controlled release (CRN) and stabilized (SNF) N fertilizers (Azeem et al. 2014, AAPFCO 2015). SRN slowly releases fertilizer N as a result of microbial decomposition (Trenkel 1997). CRN is coated N that alters the rate, pattern and duration of N release (Chien et al. 2009). SNF contains chemical compounds that inhibit rapid N transformation to less stable forms (Shaviv 1996, 2005). The attributes of N containing EEFs may provide land managers increased flexibility to fertilize with N under a variety of conditions to maximize crop N uptake and improve FNUE (Hauck 1985, Goertz 1993, Azeem et al. 2014).

In southern forestry, pelletized urea (46-0-0) is the primary N fertilizer used because of its high N content and lowest overall cost per unit of applied N (Allen 1987, Fox et al. 2007b). Several chemical reactions occur after urea is applied in the environment (Hauck and Stephenson 1965, Ferguson et al. 1988). High losses of fertilizer N after urea fertilization can occur through ammonia (NH_3) volatilization and depend on the interaction of weather

conditions and edaphic site factors at the time of fertilization (Cabrera et al. 2005, Zerpa and Fox 2011, Elliot and Fox 2014, Raymond et al. 2016). To reduce NH_3 volatilization after urea application in southeastern pine plantations, fertilization has traditionally occurred in winter when temperatures are lower and precipitation is high. However, significant N loss through NH_3 volatilization can still occur during the winter (Engel et al. 2011, Elliot and Fox 2014), in addition to losses of fertilizer N by denitrification (Shrestha et al. 2014) and leaching (Binkely et al. 1995, Aust and Blinn 2004). Fertilizer N lost following fertilization translates to less fertilizer N remaining in the ecosystem and a lower FNUE (Galloway et al. 2003).

One SNF mechanism that can be added to urea prior to fertilization is a urease inhibitor (Gowariker and Krishnamurty 2009). Urease inhibitors reduce the amount of fertilizer N lost via NH_3 volatilization by temporarily binding to the urease enzyme (AAPFCO 2015) which is abundant in forest soils (Bremner and Douglas 1971, Bremner and Chai 1986, Sanz-Cobena et al. 2008). This action provides time for urea to dissolve and move into the soil where NH_3 volatilization losses are reduced by the soil buffering capacity. Several compounds inhibit urease activity, but few are chemically stable, effective at low concentrations, and are non-toxic when applied to urea when used in the environment (Hauck 1985, Azeem et al. 2014). The urease inhibitor N-(n-Butyl) triphosphoric triamide (NBPT) is currently the most widely used (Havlin et al. 2014), and urea impregnated with NBPT can significantly reduce fertilizer N losses via NH_3 volatilization (Engel et al. 2011, Raymond et al. 2016).

CRN products generally have a urea granule base that is coated with various compounds (i.e. sulfur (S), boron, (B), copper (Cu)) to allow a gradual, controlled urea dissolution and N release to the environment as the coatings degrade and crack (Chien et al. 2009). The urea used in these products may also be impregnated with urease inhibitors. CRN products may also include additional nutrient coatings to ameliorate site specific deficiencies.

Alternative CRN coatings are semi-permeable polymers which encapsulate urea granules but allow water movement into and out of the capsule, stimulating urea dissolution and allowing fertilizer N solution to move gradually into the environment by diffusion (Shaviv 2005).

Release of fertilizer N from polymer coated CRN depends on type of coating, soil moisture, temperature, relative humidity, precipitation, pH, and microbial activity (Christianson 1988, Shoji et. al. 2001, Shaviv 2005).

As previously stated, N fertilization generally occurs during winter or early spring months to reduce high losses from NH_3 volatilization and potential immobilization in various ecosystem components in southeastern pine plantations. Yet crop tree N uptake is generally low during these periods because of cooler soil temperatures (Bassirirad et al. 1993), lower root activity (Dong et al. 2001), low microbial activity (Blazier et al. 2005) and low evapotranspiration rates (Powers and Reynolds 1999), all which can contribute to low fertilizer N uptake (Plaza et a. 2004, Blazier et al. 2006). Conversely, tree N uptake is generally greatest during the spring and summer months (Bhat 1983, Blazier et al. 2006), and the application of fertilizer N during this period may be more synchronous to tree N demand as more foliage is produced over the growing season (Powers and Reynolds 1999). The ability to retain more fertilizer N in the ecosystem with a spring and/or summer fertilization may increase FNUE for these systems by improving synchronization of N fertilization with plant N demand and uptake (Blazier et al. 2006).

Nitrogen containing fertilizers enriched with the stable isotope ^{15}N to trace the uptake and ecosystem fate of fertilizer N, combined with enhanced efficiency fertilizer technology, will assist in improving our fundamental understanding of the cycling of fertilizer N in southeastern pine plantations. An increased understanding of fertilizer N cycling could improve economic and environmental fertilizer stewardship in southeastern pine plantations in the future. In this study, we compared fertilizer N uptake in loblolly pine ecosystems

following fertilization with urea with three enhanced efficiency fertilizers at two different application times (spring vs. summer). The statistical hypotheses for this research were:

H₀1: There were no differences in the total fertilizer N recovery between urea and the enhanced efficiency N containing fertilizers.

H₀2: There were no differences in total fertilizer N recovery between urea and the enhanced efficiency N containing fertilizers between a spring and summer fertilizer application.

H₀3: There were no differences in the total fertilizer N recovery between urea and the enhanced efficiency N containing fertilizers for ecosystem components.

2. Materials and Methods

2.1 Experimental Design and Site Description

This experiment used a split-plot complete-block design to test differences between five fertilizer treatments (main plots) at two different application seasons (split plot) following fertilization in mid-rotation pine plantations. Five sites were selected adjacent to plots in an existing network of forest thinning and fertilization studies in mid-rotation pine plantations across the southeastern United States (Figure 1). Each site served as a block and provided replication across the southeastern United States. The age of the stands ranged from 17 to 19 years and were thinned at 11 to 13 years. The main plots were the five fertilizer treatments (CUF, NBPT, PCU, urea, control- no fertilization) and the split plots were the two different application seasons (spring, summer). At each of the five sites, there was single replication of each of the five main plots. Selected climate, physical and stand characteristics are detailed in

Table 1. 2.2 Fertilizer Treatments

The five treatments used in this study were: 1) urea; 2) urea impregnated with N-(n-butyl) thiophosphoric triamide (NBPT); 3) urea impregnated with NBPT and coated with monoammonium phosphate (CUF); 4) urea coated with a polymer (PCU); and 5) a control treatment with no fertilizer N added. Urea (46-0-0) was the conventional N fertilizer

used because of its common use in southern forests. The size of the urea seed granules manufactured for each treatment ranged from 4 mm to 5 mm based on the manufacturing specifications for this study.

The enhanced efficiency fertilizers (EEFs) tested in this study were developed to reduce NH_3 volatilization and release fertilizer N slowly to the environment. The NBPT treatment (46-0-0) added N-(n-butyl) thiophosphoric triamide at a rate of 26.7% by weight to urea granules to inhibit urease activity. The CUF treatment (39-9-0) also added NBPT to urea granules, but added an aqueous binder solution of boron and copper sulfate to the urea+NBPT to slow environmental N release, with a final coating of monoammonium phosphate to provide P. The PCU (44-0-0) treatment encapsulated urea granules with a polymer coating containing pores designed to slowly release N (~80%) over 120 days. All N treatments were applied at an equivalent rate of 224 kg N ha^{-1} . Because the CUF treatment had P in a coating, P was applied with all other fertilizer treatment at the equivalent rate of 28 kg P ha^{-1} as triple superphosphate (TSP). The urea base for all treatments was enriched with the stable isotope ^{15}N (~370‰, 0.5 atom percent). All individual fertilizer treatments were broadcast applied by hand to individual 100m^2 circular plots described below.

2.3 Field Sampling

The plot centers of five 100 m^2 circular plots were installed between two co-dominant loblolly pine trees at five individual sites for each application season in areas of the site with similar stand, soil and landscape characteristics. The boundaries of all plots at each site were installed at a minimum distance of 30.48 m from each other and all existing fertilizer trials to reduce the risk of contamination to plots.

Prior to treatment application, plot measurements included: 1) height and diameter breast height (DBH) of all trees greater than 2.54 cm DBH, 2) forest floor (O horizon = O_i + O_e + O_a), 3), mineral soil from two depth increments (0-15 cm, 15-30 cm), 4) fine and coarse

roots, and 5) bulk density cores for the entire soil for each soil depth. The forest floor was sampled at 4 random locations in the plot with a circular sampler and composited. The mineral soil was sampled at 8 random locations in the plot with a push tube sampler and composited. Roots were sampled at 4 random locations in the plot with a bulk density corer down to 20 cm and composited. A single bulk density core was taken from the center of each plot. Upon completion of this sampling, two circular mesh traps (1 m²) were placed in each plot to sample litterfall at the end of the growing season after fertilization.

At each individual site, a single fertilizer N treatment was randomly applied to a 100m² circular plot on a single day for each application season (spring: March 26 to April 8, summer: June 18 to 30). After the first growing season post-fertilization, plots were sampled between November 1st and March 31st in the dormant season with the same procedures previously detailed. Litterfall from the entire growing season was collected once on a single date in the dormant season and composited from both litterfall traps in each individual plot.

Additionally, one central crop tree per plot was felled, divided into the following components and sampled: 1) foliage; 2) fine branches; 3) coarse branches; 4) dead branches; and 5) stem, with cookie samples from DBH, base of live crown (HLC), and mid-crown (HMC). The crop tree was chosen from the center of the plot to reduce the potential edge effect of the circular plot. Foliage, fine branches (fine branches = branches with foliage) and coarse branches (coarse branches = branches with no foliage) from different positions in the canopy were placed randomly in 3 separate piles to reduce the potential for over-representation of specific canopy areas in each pile. The foliage+fine branches were separated, in the field, from the coarse branches in each pile and weighed. One pile was randomly chosen and the foliage was separated from the fine branches and weighed. All cookies sampled from each height increment (DBH, HLC, HMC) were cut to a 2.54 cm

width. All weights for all tree components were weighed in the field on the same day as the tree was harvested to obtain appropriate green weights.

Although the understory was a minor component of the ecosystem for all plots at each site, when present, was divided into different strata and sampled. Sampling of the sapling stratum was by 2.54 cm diameter individual species categories. Shrubs, vines and herbaceous species were sampled from a randomly selected 3.13 m² wedge area of the plot. Individual shrub species were sampled in their entirety by species. Vines and herbaceous species were composite sampled in their respective strata. All green weights were weighed in the same manner as the harvested crop tree and the entire sample, when present, was returned to the laboratory for analysis.

2.4 Laboratory Processing

All biomass samples were dried by their respective components in a forced air oven at 60°C. Subsamples of bark, wood of the current year of growth after applied treatment (CGR), and the wood of previous growth prior to fertilizer treatment (PGR) were taken from the DBH stem cookies. Litterfall was additionally separated into pine needles, deciduous leaves, fine branches, coarse branches, bark and unidentifiable litterfall. The forest floor was sieved through a 6 mm sieve and each mineral soil depth increment was sieved through a 2 mm sieve. Roots were washed and sorted by live and dead roots into the general categories of loblolly pine and all other species (if present) into fine (<2 mm) and coarse (>2 mm) roots prior to drying.

After drying, all organic material samples were coarse ground in a Thomas® Model 4 Wiley Mill (Thomas Scientific, Sedesboro, NJ) to pass a 2 mm sieve. All organic samples were subsequently homogenized to a fine powder with a ball mill (Retsch® Mixer Mill MM 200, Haan, Germany) for 1 minute at 25 revolutions per second (rps), whereas all mineral soil samples were ball milled for 2 minutes at 25 rps. After ball milling, individual homogenized

samples were put in separate tin capsules and weighed on a Mettler-Toledo[®] MX5 microbalance (Mettler-Toledo, Inc., Columbus, OH, USA). These individually weighed samples were analyzed to determine the $\delta^{15}\text{N}$ (‰) values and total N concentration on a coupled elemental analysis-isotope ratio mass spectrometer (IsoPrime 100 EA-IRMS, Isoprime[®] Ltd., Manchester, UK). All instrumentation used during coarse grinding and ball milling was cleaned with ethanol to reduce cross sample contamination.

2.5. Calculation of Fertilizer N Recovery

The total fertilizer N recovered in each ecosystem component from the labeled ^{15}N fertilizer was calculated using a mass balance tracer technique adapted from Powlson and Barraclough (1993), Nadelhoffer and Fry (1994), Nadelhoffer et al. (1995), and Raymond et al. (2016). The natural abundance $\delta^{15}\text{N}$ values of individual ecosystem components was compared to the $\delta^{15}\text{N}$ values of the same individual ecosystem components at the end of a single growing season after N fertilization with each ^{15}N enriched fertilizer. The fertilizer N recovery for each individual ecosystem component was summed on a per plot basis to calculate total fertilizer N recovery for each individual fertilized plot. As an example, the calculated fertilizer N recovered for the single individual loblolly pine tree sampled (above + belowground biomass) was used to extrapolate loblolly pine fertilizer N uptake by the number of loblolly pine trees in the plot to provide a total fertilizer N recovery by loblolly pine trees for each individual plot. The difference between the amount of fertilizer N applied to the plot and the amount of fertilizer N recovered after a single growing season was considered lost from the system. The following equations were used:

$$[1] \text{ \% fertilizer N ecosystem component} = ((\delta^{15}\text{N}_{\text{final}} - \delta^{15}\text{N}_{\text{initial}})/(\delta^{15}\text{N}_{\text{labeled}} - \delta^{15}\text{N}_{\text{initial}})) * 100$$

Where $\delta^{15}\text{N}_{\text{initial}}$ is the natural ^{15}N abundance of the ecosystem component; $\delta^{15}\text{N}_{\text{labeled}}$ is the ^{15}N abundance of the added fertilizer; $\delta^{15}\text{N}_{\text{final}}$ is the final ^{15}N abundance of the ecosystem

component N pool. Equation [2] is the percent of N in the ecosystem component, Equation [3] to [5] was used to calculate the percent fertilizer recovery.

[2] $\%^{15}\text{N}$ from label in ecosystem component =

$$((\delta^{15}\text{N}_{\text{ecosystem component}} - \delta^{15}\text{N}_{\text{initial}})/(\delta^{15}\text{N}_{\text{fertilizer}} - \delta^{15}\text{N}_{\text{initial}}))$$

[3] Total N (g) = (Ecosystem component dry weight * %N) / 100

[4] N derived from fertilizer (g) in ecosystem component =

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

[5] %N derived from fertilizer in ecosystem component =

$$(\text{N from fertilizer (g) in ecosystem component} / ^{15}\text{N fertilizer (g)}) * 100$$

2.6 Statistical Analysis

Total fertilizer N recovery, expressed as a percentage of fertilizer N applied, was analyzed using a general linear model analysis of variance with SAS[®] 9.4 (SAS Institute Inc., Cary, NC). Percent data was arcsin transformed prior to analysis. Percent fertilizer N recovery (%) was the response variable for the model, fertilizer treatment (CUF, NBPT, PCU, urea, control) and season were fixed effects, and site was a random effect in the split plot analysis. Total fertilizer N recovery, expressed as a percentage of fertilizer N applied, for individual ecosystem components were also analyzed using a general linear model analysis of variance, except for analysis of individual mineral soil depth increments (0-15 cm, 15-30 cm) which were analyzed as a repeated measures analysis of variance. All levels of significance were set at $\alpha = 0.05$ and the $P > |t|$ values for the treatment means were tested. All post-hoc analysis was conducted with Tukey's HSD.

3. Results

There was a difference in total fertilizer N recovery, expressed as a percentage of fertilizer N applied, for the entire ecosystem between all EEFs (CUF = 78.7%, NBPT =

84.0%, PCU = 78.4%) and urea (52.0%), although there were no differences among individual EEFs (Figure 2, Tables 2 and 3). There were also differences in fertilizer N recovery for certain individual components of the crop trees among the different fertilizer treatments (Table 3). Fertilizer N recovery in the crop trees was greatest in the foliage, ranging from 9.1% to 14.8% of the N applied. Nitrogen recovery in the other components of the trees was less, averaging 1.5% for fine branches, 4.3% for coarse branches, 1.0% for bark, 1.8% in wood formed in the current year after fertilization and 3.9% in older wood. Fertilizer N recovery was higher in the fine roots, averaging 11.2%, but less in the coarse roots which averaged 1.7%. Fertilizer N recovered in the fresh litterfall was low, averaging 0.5%. In the soil, fertilizer N recovery in the forest floor averaged 2.8% among the fertilizer treatments. In contrast, fertilizer N recovery in the surface 0-15 cm of the mineral soil was greater for all EEFs (26.5% to 31.2%) compared to urea (13.4%). Fertilizer N recovery in the 15-30 cm mineral soil was lower than in the 0-15 cm depth, ranging from 5.1% to 8.3% for all fertilizer treatments. When ecosystem components were combined into the categories of tree (canopy + stem + roots) and soil (forest floor + 0-30 cm), only minor differences occurred among treatments (not significant) for the tree, with more fertilizer N recovered for all EEFs (35.5% to 42.5%) compared to urea (20.5%) in the soil (Tables 2 and 3).

Nitrogen concentrations (g kg^{-1}) in the foliage were greater overall in the fertilized treatments (Table 3). For fine branches, nitrogen concentrations in the foliage increased from 12.3 g kg^{-1} in the control to 14.1 g kg^{-1} to 15.0 g kg^{-1} for all fertilizer treatments. There was no difference between application season for N concentrations in the fine branches, although N concentrations were greater in the fertilized treatments (6.6 g kg^{-1} to 7.4 g kg^{-1}) compared to the control (5.1 g kg^{-1}) with no differences among the EEFs. Differences in N concentration between the control and the fertilizer treatments also occurred for the current growth ring the year after fertilization (CGR) where NBPT (1.9 g kg^{-1}) and urea (2.0 g kg^{-1})

were greater than CUF (1.5 g kg⁻¹), PCU (1.8 g kg⁻¹) and the control (1.4 g kg⁻¹), and in the forest floor where both CUF and PCU (9.4 g kg⁻¹) were greater than NBPT (8.6 g kg⁻¹) and urea (8.5 g kg⁻¹). For litterfall, urea (5.1 g kg⁻¹) had the lowest N concentration and was different from the control (7.8 g kg⁻¹).

The $\delta^{15}\text{N}$ (‰) values in all tree and soil components sampled were greater for the fertilizer treatments compared to the control (Table 3). The $\delta^{15}\text{N}$ values were greatest in the foliage (83.9‰ to 97.1‰), fine branches (74.2‰ to 86.9‰) and wood formed in the current growing season following fertilization (59.3‰ to 73.8‰). The $\delta^{15}\text{N}$ values in the current year litterfall was lower than foliage (29.7‰ to 33.8‰). Wood formed in years prior to the fertilization treatments had lower $\delta^{15}\text{N}$ values (30.7‰ to 36.6‰), whereas the $\delta^{15}\text{N}$ values were lowest in bark (15.0‰ to 18.9‰), fine roots (14.0‰ to 24.1‰) and coarse roots (8.5‰ to 15.8‰) with both CUF and NBPT greater than urea for the coarse roots. In the soil, the $\delta^{15}\text{N}$ values were greatest in the forest floor (65.0‰ to 89.6‰), comparable to the $\delta^{15}\text{N}$ values in the foliage. Conversely, the $\delta^{15}\text{N}$ value in the surface 0-15 cm mineral soil in fertilized treatments ranged from 10.2‰ to 18.0‰ compared to the control (4.0‰), and all EEFs were greater than urea. In the 15-30 cm mineral soil the $\delta^{15}\text{N}$ values ranged from 8.5‰ to 11.9‰, with the CUF, NBPT, PCU treatment being significantly greater than the control (5.5‰).

There were few differences for the amount of fertilizer N recovered, with no differences in $\delta^{15}\text{N}$ values or N concentration, for ecosystem components with the same fertilizer treatment between seasons of application (Figure 3, Tables 2, A.1 to A.3). For example, the NBPT treatment had a higher fertilizer N recovery for the coarse branches with the spring fertilization (7.3%) compared to the summer (2.6%), whereas the CUF treatment had a higher fertilizer N recovery for the stem in the spring (8.0%) compared to the summer (2.9%). Although there were few differences in the percentage of fertilizer N recovered for

ecosystem components between seasons, there was a trend among the primary components (tree, soil) between seasons for the EEFs collectively. In the tree, there was a higher percentage (not significant) of fertilizer N recovered in the spring (34.7% to 53.3%) compared to the summer (28.5% to 33.5%) for all EEFs. Conversely in the soil, there was a higher percentage (not significant) of fertilizer N recovered in the summer (40.2% to 50.0%) compared to the spring (29.6% to 37.6%) for all EEFs.

4. Discussion

Our first hypothesis that differences did not exist in total fertilizer N recovery among individual fertilizer treatments (CUF, NBPT, PCU, urea) was rejected. Total fertilizer N recovery was significantly greater for the enhanced efficiency fertilizers (CUF, NBPT, PCU) compared to urea in both application seasons. Although significantly more fertilizer N was recovered for EEFs collectively, differences generally did not exist among individual EEFs. The lower fertilizer N recovery for urea was likely due to larger NH_3 volatilization losses from the urea treatment compared to the EEFs (Raymond et al. 2016).

In a companion study, Raymond et al. (2016) used the same 5 sites in this study and found fertilizer N losses from NH_3 volatilization following fertilization with urea ranged from 26% to 49% compared to the same EEFs used in this research, which had fertilizer N losses ranging from 4% to 26%. Other studies have also reported large NH_3 volatilization losses following fertilization with urea in southeastern pine plantations (Kissel et al. 2004, Zerpa and Fox 2011). Elliot and Fox (2014) found NH_3 volatilization losses were approximately 50% less following fertilization in pine plantations for EEFs compared to urea. When the mean percent NH_3 volatilization loss reported by Raymond et al. (2016) are incorporated into the mass balance for each respective treatment, the percent recovery of the fertilizer N ranges from 90% to 100%. This evidence indicates that higher fertilizer N loss through NH_3

volatilization for urea compared to EEFs leads to a higher percent recovery of fertilizer N for the entire ecosystem for the EEFs compared to urea.

Although NH_3 volatilization loss after urea fertilization is likely to be the primary loss mechanism in these studies, leaching (Van Miegroet et al. 1994, Binkley et al. 1995, Lee and Jose 2005) and denitrification (Shrestha et al. 2014) are also potential pathways for fertilizer N loss. We indirectly tested the potential leaching loss of fertilizer N by sampling soil in the 15-30 cm mineral soil and analyzing for ^{15}N . If this loss pathway was significant, higher $\delta^{15}\text{N}$ values would potentially occur in the 15-30 cm mineral soil increment compared to the control, and the percent fertilizer N recovery in this soil depth increment could be large. In the 15-30 cm mineral soil, fertilizer N recovery for all treatments was less than 11%. This result, combined with the total mass balance calculation, indicates leaching for the year of fertilization from most sites was likely low. These results indicate that although some fertilizer N was moving deeper in to the soil, the percent loss for leaching was potentially a minor loss pathway during the initial growing season following fertilization.

Denitrification, another potential major loss pathway, was not measured. Recent work on fertilizer N loss from denitrification on similar sites used in this study found applied N losses via this pathway were low during spring and summer months (Shrestha et al. 2014), and is generally considered a minor loss pathway in aerobic forest soils (Bowden et al. 1991). If denitrification losses were a significant factor in these pine ecosystems, one would hypothesize that losses would be greater on poorly drained soils. Comparing N losses on well versus poorly drained soils in our study found no differences in percent recovery of fertilizer N due to soil drainage class (aerobic versus anaerobic systems), indicating denitrification was also likely a minor loss mechanism during the first growing season following fertilization.

The second hypothesis that no differences occurred in fertilizer N recovery among treatments between a spring and summer application was rejected. Statistically, significantly

more fertilizer N was recovered after the spring fertilization in only coarse branches of the NBPT treatment and the stem of the CUF treatment compared to the summer fertilization. Although these were the only two statistically significant differences that occurred among treatments, an obvious trend occurred for all treatments between the spring and summer fertilization (Figure 3, Table A.3.). When the fertilizer N recovery results are compared in Table A.3 for the same fertilizer treatment between seasons, a subtle shift in the ecosystem partitioning of the fertilizer N recovered occurs for all treatments. Although not statistically significant (NS), when the primary ecosystem components are viewed, a slightly greater proportion of fertilizer N is recovered after the spring fertilization for all treatments for the total tree (canopy + stem + roots) compared to summer. Conversely, a slightly greater proportion of fertilizer N is recovered after the summer fertilization for all treatments for the total soil (forest floor + 0-30cm mineral) compared to the spring. This subtle shift in ecosystem partitioning could partially be explained by the timing of the summer fertilization with soil water deficits in the surface soils of these ecosystems (Teskey et al. 1987, Albaugh et al. 2004, Blazier et al. 2006) It is important to note this shift because of the potential impact, particularly in soil recovery, that may influence the long term uptake and cycling of fertilizer N from the soil in these pine plantation systems of the southeastern United States

This subtle shift in ecosystem partitioning of an increasing amount of fertilizer N recovered with EEFs in the soil (spring+summer: 31% to 39%, spring: 24% to 38%, summer: 40% to 50%) compared to urea (spring+summer: 28%, spring: 22%, summer: 19%), raises two primary questions: 1) will the additional fertilizer N remaining in the soil with the EEF products after either a spring or summer fertilization become available to the desired crop trees; and 2) if the additional fertilizer N remaining in the soil with EEF products becomes immobilized and unavailable for crop tree uptake, can fertilizer N application rates be reduced while maintaining productivity?

Research in agricultural systems indicates the fertilizer N not taken up by plants during the growing season after fertilization is generally immobilized in the soil (Preston 1982), with minor amounts available in subsequent years. These results have also been shown to occur in forested systems where the majority of fertilizer N uptake occurs in the first growing season after fertilization, with significantly less occurring in subsequent years (Foster et al. 1985a, b, Hulm and Killham 1990, Nõmmik and Larsson 1992, Preston et al. 1994). Yet greenhouse bioassay studies using soil from long term ^{15}N tracer experiments have shown seedling uptake of ^{15}N labeled fertilizers from both forest floor and mineral soil sources a decade after fertilization (Swanston and Preston 2014). Additional long term research on the existing ^{15}N plots from this study will be required to appropriately address the question of whether the increased ^{15}N recovery in the soil will translate to a higher N availability for crop trees in these southeastern pine plantations in the future, or be immobilized or leached from the system. If future research for the ^{15}N plots used in this study find a majority of the fertilizer N immobilized in the soil for EEFs, as has been found with other studies using traditional N fertilizers in forest ecosystems, the primary question becomes whether fertilizer N application rates can be reduced by a percentage when compared to the traditional rates used in forestry while maintaining productivity. Results indicating either a higher fertilizer N availability for extended periods from the soil using EEFs and/or a reduction in fertilizer N application rates while maintaining productivity would assist in improving the FNUE efficiency of these pine plantation systems.

We rejected our third hypothesis that there was no difference in the ecosystem partitioning of fertilizer N among treatments. When individual ecosystem components (foliage, fine branches, forest floor, etc.) were analyzed separately, differences did occur among fertilizer treatments for certain individual ecosystem components. Regardless of the season or treatment, the majority of the fertilizer N was recovered in either the loblolly pine

trees or soil. These results are similar to those of other tracer studies in a variety of ecosystems (Preson et al. 1990, Preston and Mead 1994, Nadelhoffer et al. 1995, 2004, Tietema et al. 1998, Burke et al. 2002, Blanes et al. 2002), although many of these studies found a higher recovery of applied ^{15}N in the soil.

Previous studies in pine plantations indicate generally low fertilizer N uptake (<25%) by southeastern pine trees (Baker et al. 1974, Mead and Pritchett 1975, Melin et al. 1983, Ballard 1984, Johnson and Todd 1988, Johnson 1992, Li et al. 1999, Albaugh et al. 2004, Blazier et al. 2006). One probable explanation for low recoveries of fertilizer N in southeastern pine fertilizer trials is due to the use of urea for the fertilizer N source. As previously stated, large losses of fertilizer N can occur with urea through NH_3 volatilization (Raymond et al. 2016). If fertilizer N losses are substantial, this would equate to less fertilizer N remaining in the soil and translate to less potential N for uptake by the crop trees. As previously reported, fertilizer N recovery of urea by the entire loblolly pine tree (canopy + stem + roots) was calculated at 35% compared to EEFs which ranged from 33% to 50%, dependent on the season of application. A trend exists for increased fertilizer N recovery in loblolly pine trees for EEFs compared to urea, and differences in the partitioning of fertilizer N among ecosystem components between EEFs and urea is likely important in both the short and long term cycling of fertilizer N in these southeastern pine plantations.

A primary benefit of this research has been refining the understanding of the ecosystem fate for both traditional and enhanced efficiency forms of N containing fertilizers through the use of stable isotopes in mid-rotation pine plantations across the southeastern United States. Results from this study showed significant differences occur in ecosystem fertilizer N recovery between enhanced efficiency N containing fertilizers and urea across the entire southeastern U.S. where pine plantations are operationally fertilized and intensively managed. This area encompassed a large range of edaphic, climatic and stand variables.

Because of this broad range, the results from this study compliment other fertilizer N studies using urea in forest ecosystems focused on smaller regions or single sites. Additionally, the results from this research provide a level of confidence to managers that the enhanced efficiency products investigated in this study have high levels of ecosystem retention during the growing season for the year of fertilization when compared to urea, and should assist land managers in gradually improving fertilizer nitrogen use efficiency in pine plantations across the southeastern United States.

5. Conclusions

The primary conclusions from this research were: 1) significantly more fertilizer N was recovered from the ecosystem for all enhanced efficiency fertilizers compared to urea; 2) although there were differences in the ecosystem partitioning of fertilizer N among treatments between seasons of fertilizer application, more subtle trends occurred between seasons for primary ecosystem components (tree, soil); and 3) differences occurred in the ecosystem partitioning of fertilizer N among treatments. These results collectively indicate, regardless of the season of application, that there was an increase in fertilizer N remaining in the ecosystem for EEFs compared to urea. This finding may translate to increased fertilizer N availability over time for desired crop trees, and hence increase fertilizer nitrogen use efficiency when using enhanced efficiency N containing fertilizers compared to urea. Additional retention of fertilizer N in the soil for enhanced efficiency fertilizers may also increase fertilizer nitrogen uptake by the trees later in the rotation if this fertilizer N becomes available to the plants, but additional research will be required for this determination. These findings will help to continue to improve and refine fertilizer management in southeastern pine plantations. The results for the enhanced efficiency N containing fertilizers used in this study provide forest managers the flexibility to apply fertilizer N in either the spring or summer months in the

474 southeastern United States with the confidence that losses from the system will be lower
475 when compared to urea, potentially creating a more synchronous timing for fertilizer N
476 application to seasonal plant demand and increasing fertilizer nitrogen use efficiency for
477 these systems in the future.

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820 **Table 1.** Selected climate, physical and stand characteristics of 5 mid-rotation loblolly pine stands in the southeastern United States selected to evaluate ecosystem partitioning
821 of applied fertilizer N following the application of urea or enhanced efficiency N containing fertilizers enriched with ¹⁵N.

Site	Latitude	Longitude	Alt ¹ (m)	MAP ² (cm)	MAT ³ (°C)	Region	Soil Taxonomic Class	Soil Series	Soil Drainage Class	Trees plot ⁻¹	Trees ha ⁻¹	Mean Height (m)	Mean DBH (cm)	Basal Area (m ² ha ⁻¹)
VA	37.440087	78.662396	60	109	13	Piedmont	fine, mixed, subactive, mesic Typic Hapludults	Littlejoe	Well	13	1260	13.1	16.4	27.4
SC	33.869400	79.289300	2	125	17	Atlantic Coastal Plain	fine, kaolinitic, thermic Umbric Paleaquults loamy-skeleta	Byars	Very Poorly	5	540	15.7	25.9	29.4
AL1	33.233371	87.232384	48	125	17	Appalachian Plateau	l, mixed, subactive, thermic, Typic Dystrudepts	Montevallo	Well	4	400	19.2	24.4	19.0
AL2	31.659464	86.272045	34	145	26	Southern Coastal Plain	fine, smectitic, thermic, Typic Hapludults	Arundel	Well	6	600	18.1	20.5	20.5
AR	33.422310	91.732651	21	140	17	Western Gulf Coastal Plain	fine-silty, mixed, active, thermic, Typic Glossaqualfs	Calhoun	Poorly	6	620	15.4	20.7	21.3

¹ = Altitude. ² = Mean annual precipitation. ³ = Mean annual temperature.

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Table 2. The analysis of variance for differences in total ecosystem fertilizer N recovery (% of fertilizer N applied) of loblolly pine stands across the southeastern United States selected to evaluate ecosystem partitioning of applied fertilizer N of urea or enhanced efficiency N containing fertilizers enriched with ¹⁵N after combined seasonal application (spring + summer 2011). N = 10.

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F-Value	P-Value
Treatment	4	49169	12292	52	<0.0001
Season	1	17	17	0.1	0.8
Treatment*Season	4	447	112	0.5	0.8
Error	40	9517	238		
Total	49	59150			

Table 3. The mean $\delta^{15}\text{N}$ (‰), N concentration (g kg^{-1}), and fertilizer N recovery (% of fertilizer N applied) for individual ecosystem components of loblolly pine stands across the southeastern United States selected to evaluate ecosystem partitioning of applied fertilizer N of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after combined seasonal application (spring + summer 2011). Different letters represent significant differences at $\alpha = 0.05$ within each respective ecosystem component within columns. Numbers in parentheses represent the standard error of the mean. The N application rate was 224 kg N ha^{-1} . N = 10.

Ecosystem Component	Treatment	$\delta^{15}\text{N}$ (‰)	N concentration (g kg^{-1})	Fertilizer N Recovery (% fertilizer N applied)
Foliage	Control	-3.5a (0.5)	12.3a (0.4)	-
	CUF	96.5b (9.8)	14.1b (0.5)	11.7a (2.1)
	NBPT	97.1b (9.1)	15.0b (0.7)	14.8a (2.9)
	PCU	83.9b (9.3)	14.4b (0.4)	9.1a (0.9)
	Urea	92.7b (8.4)	14.7b (0.5)	12.2a (2.4)
Fine Branches	Control	-4.0a (0.78)	5.1a (0.2)	-
	CUF	86.9b (7.4)	7.4b (0.4)	1.7b (0.3)
	NBPT	83.3b (6.7)	7.3b (0.5)	1.9b (0.4)
	PCU	74.2b (7.2)	6.9b (0.3)	1.1a (0.2)
	Urea	75.1b (8.9)	6.6b (0.4)	1.2a (0.3)
Coarse Branches	Control	-2.4a (0.8)	3.4a (0.6)	-
	CUF	58.4b (5.7)	3.3a (0.4)	4.6a (0.8)
	NBPT	59.9b (6.3)	3.3a (0.3)	5.0a (1.0)
	PCU	52.8b (5.7)	3.3a (0.3)	3.5a (0.6)
	Urea	55.7b (3.1)	3.2a (0.2)	4.1a (0.6)
<i>Canopy (foliage+fine branches+coarse branches)</i>	Control	-	-	-
	CUF	-	-	18.0a (2.1)
	NBPT	-	-	21.6a (1.9)
	PCU	-	-	13.6a (1.7)
	Urea	-	-	17.5a (1.8)
Bark	Control	-2.8a (1.6)	2.1a (0.3)	-
	CUF	15.3b (3.4)	2.1a (0.2)	0.9a (0.4)
	NBPT	18.9b (4.7)	2.5a (0.3)	1.1a (0.3)
	PCU	16.0b (2.8)	2.5a (0.5)	1.1a (0.3)
	Urea	15.0b (2.3)	2.3a (0.1)	0.7a (0.1)
Growth ring year of sampling (CGR)	Control	-4.2a (0.60)	1.4a (0.2)	-
	CUF	73.8b (7.3)	1.5a (0.1)	1.7a (0.4)
	NBPT	72.9b (7.5)	1.9b (0.1)	1.9a (0.2)
	PCU	59.3b (8.1)	1.8a (0.1)	1.6a (0.3)
	Urea	70.5b (7.1)	2.0b (0.2)	2.1a (0.4)
Growth rings composited prior to year of sampling (PGR)	Control	-3.4a (1.1)	1.0a (0.1)	-
	CUF	35.7b (4.0)	1.1a (0.2)	3.4a (0.7)
	NBPT	36.6b (4.8)	1.3a (0.2)	5.0a (1.1)
	PCU	30.7b (4.3)	1.2a (0.2)	4.4a (1.0)
	Urea	31.6b (4.5)	0.9a (0.2)	2.7a (0.6)
<i>Stem</i>	Control	-	-	-
	CUF	-	-	5.5a (1.0)
	NBPT	-	-	7.7a (1.2)
	PCU	-	-	7.0a (1.0)
	Urea	-	-	5.3a (0.8)
Fine Roots (< 2mm)	Control	1.6a (1.9)	13.0a (0.8)	-
	CUF	24.1b (3.5)	12.3a (0.6)	11.1a (1.8)
	NBPT	19.8b (3.4)	12.8a (0.7)	12.6a (2.4)
	PCU	19.3b (3.2)	12.4a (0.6)	10.9a (1.3)
	Urea	14.0ab (1.5)	12.2a (0.6)	10.0a (1.8)
Coarse Roots (> 2mm)	Control	0.8a (0.7)	8.7c (0.6)	-
	CUF	15.8c (2.3)	7.9bc (0.5)	2.0a (0.5)
	NBPT	14.0c (1.4)	7.7b (0.5)	2.4a (0.8)
	PCU	12.2bc (1.8)	7.0b (0.2)	1.5a (0.5)
	Urea	8.5b (1.0)	6.8a (0.3)	0.8a (0.2)

Total Roots (<i>fine + coarse</i>)	Control	-	-	-
	CUF	-	-	13.2a (2.4)
	NBPT	-	-	14.0a (2.6)
	PCU	-	-	11.6a (2.0)
	Urea	-	-	10.8a (1.9)
Total Tree (<i>canopy + stem + total roots</i>)	Control	-	-	-
	CUF	-	-	37.2a (3.9)
	NBPT	-	-	43.4a (5.0)
	PCU	-	-	31.6a (2.8)
	Urea	-	-	33.4a (2.6)
Litterfall (current year of sampling)	Control	-2.2a (0.8)	7.8b (0.5)	-
	CUF	29.7b (7.2)	6.1ab (0.6)	0.5a (0.1)
	NBPT	31.0b (7.5)	5.9ab (0.8)	0.5a (0.2)
	PCU	33.8b (8.2)	6.0ab (0.6)	0.6a (0.2)
	Urea	28.9b (6.9)	5.1a (0.7)	0.3a (0.1)
Forest Floor	Control	-2.3a (0.4)	7.2a (0.6)	-
	CUF	77.5b (10.1)	9.4b (0.7)	2.8a (0.8)
	NBPT	71.5b (9.3)	8.6ab (0.5)	2.8a (0.8)
	PCU	89.6b (7.8)	9.4b (0.8)	3.4a (1.0)
	Urea	65.0b (9.1)	8.5ab (0.7)	2.0a (0.4)
Mineral Soil 0-15 cm	Control	4.0a (0.6)	1.2a (0.1)	-
	CUF	16.6c (1.9)	1.2a (0.2)	27.8b (3.8)
	NBPT	16.1bc (1.7)	1.1a (0.2)	26.5b (4.2)
	PCU	18.0c (2.8)	1.3a (0.3)	31.2b (4.2)
	Urea	10.2b (1.5)	1.2a (0.3)	13.4a (3.7)
Mineral Soil 15-30 cm	Control	5.5a (0.37)	0.7a (0.1)	-
	CUF	11.9b (2.3)	0.7a (0.1)	8.3a (3.3)
	NBPT	10.3b (1.0)	0.7a (0.2)	6.2a (1.3)
	PCU	11.2b (1.5)	0.9a (0.3)	7.9a (1.7)
	Urea	8.5ab (0.6)	0.9a (0.3)	5.1a (2.1)
Total Soil (<i>Forest Floor + Mineral Soil</i>)	Control	-	-	-
	CUF	-	-	38.9b (5.7)
	NBPT	-	-	35.5b (4.9)
	PCU	-	-	42.5b (5.3)
	Urea	-	-	20.5a (3.7)

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Figure 1. Location map of 5 mid-rotation loblolly pine stands across the southeastern United States selected to evaluate ecosystem partitioning of fertilizer N following the application of urea or enhanced efficiency N fertilizers enriched with ^{15}N .

Figure 2. The total fertilizer N recovery (% of fertilizer N applied) of the major ecosystem components (tree – canopy + stem + roots), litterfall, and soil (forest floor + 0 to 30 cm)) for 5 mid-rotation loblolly pine stands across the southeastern United States selected to evaluate ecosystem partitioning of fertilizer N of urea or enhanced efficiency N fertilizers enriched with ^{15}N . Data represents combined fertilizer application dates for spring (March-April 2011) and summer (June 2011). Different fonts represent comparisons among treatments between the same ecosystem components (soil, litterfall, tree). Different letters represent significant differences at $\alpha = 0.05$. The N application rate was 224 kg N ha^{-1} . N=10.

Figure 3. The total fertilizer N recovery (% of fertilizer N applied) of the major ecosystem components (tree – canopy + stem + roots), litterfall, O horizon (forest floor), mineral soil (0 to 30cm) and roots (fine+coarse) for 5 mid-rotation loblolly pine stands across the southeastern United States selected to evaluate ecosystem partitioning of fertilizer N of urea or enhanced efficiency N fertilizers enriched with ¹⁵N. Data represents fertilizer application dates for spring (March-April 2011) and summer (June 2011). Different letters represent significant differences at $\alpha = 0.05$ for total fertilizer N recovery for the entire ecosystem. The N application rate was 224 kg N ha⁻¹. N =5 for each season.