Use of Stable Isotopes to Trace the Fate of Applied Nitrogen in Forest Plantations to Evaluate Fertilizer Efficiency and Ecosystem Impacts

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ABSTRACT

This study assessed five fertilizer treatments (control – no fertilizer, urea, urea treated with N-(n-butyl) thiophosphoric triamide (NBPT), coated urea + NBPT (CUF), polymer coated urea (PCU)) during two application seasons (spring, summer) to: 1) compare fertilizer nitrogen (N) losses (*see* Chapter 2); 2) evaluate temporal N uptake patterns of loblolly pine (*see* Chapter 3); and 3) evaluate fertilizer N cycling and partitioning in a loblolly pine ecosystem (*see* Chapter 4).

Chapter 2 results showed enhanced efficiency fertilizers (EEFs) significantly reduced ammonia (NH_3) volatilization losses compared to urea. Mean NH_3 volatilization after spring fertilization ranged from 4% to 26% for EEFs versus 26% to 40% for urea, and 8% to 23% for EEFs versus 29% to 49% for urea in summer. Chapter 3 results showed an increase in timing and development of foliage in fertilized compared to unfertilized plots. In addition, the cumulative N uptake by loblolly pines increased over the entire growing season from N originating from fertilizer and natural sources. Chapter 4 results showed greater fertilizer N recovery for EEFs in both spring and summer (80%, 70-80% respectively) compared to urea (60%, 50% respectively) with most fertilizer N recovered from mineral soil (20% to 50%) and loblolly pines (10% to 50%).

Three primary conclusions come from this research: 1) EEFs reduce NH_3 volatilization after N fertilization compared to urea regardless of application timing and weather conditions (*see* Chapter 2); 2) N uptake by loblolly pines increases over the entire growing season after N fertilization (*see* Chapter 3); more fertilizer N remains in the ecosystem with EEFs compared to urea with most fertilizer N remaining in the soil (*see* Chapter 4). From these findings, we hypothesize that the EEFs in this study: 1) reduce ammonia volatilization which 2) translates to an increase in fertilizer nitrogen remaining in the loblolly pine plantation system that 3) increases the amount of plant available nitrogen for an extended period into the stand rotation and 4) increases fertilizer nitrogen use efficiency (FNUE) for all enhanced efficiency fertilizers investigated in this study compared to the conventional form of fertilizer N used in forestry, urea.

Dedication

To my wife Anja Whittington.

Without her understanding, patience and support at every level,

this process would have been less enjoyable and less meaningful.

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Chapter 1. Introduction

1.1. Justification

Fertilizers, primarily containing nitrogen (N) and phosphorous (P), have been used for several decades in loblolly pine (*Pinus taeda* L.) plantations in the southern United States to improve productivity by ameliorating widespread nutrient deficiencies of N and P occurring in these southern forest soils (Allen 1987, Fischer and Binkley 2000, Richter et al. 2000, Allen et al. 2005, Fox et al. 2007a, b). Consequently, over 500,000 ha of forested plantations are fertilized annually with N and P in the southern United States (Albaugh et al. 2004). Other nutrients, such as potassium (K) and/or micronutrients, may also be limiting or co-limiting but are applied on a site specific basis when these deficiencies are observed (Albaugh et al. 1998, 2004, Sampson and Allen 1999, Jokela and Martin 2000, Vogel and Jokela 2011, Carlson et al. 2014).

Despite significant gains in forest productivity from fertilization, a large degree of uncertainty exists in the fate of the fertilizer N in these plantation systems (Fox et al. 2007a). The majority of fertilizer N applied in southern pine plantations is urea ($CO(NH_2)_2$) (Allen 1987). Urea is the preferred source of N because of a high N content (46%) and overall low cost per unit (Allen 1987, Fox et al. 2007b). However, potentially large losses of fertilizer N can occur following fertilization with urea due to ammonia (NH_3) volatilization, with losses generally correlated with weather conditions at the time of application (Kissel et al. 2009, Zerpa and Fox 2011, Elliot and Fox 2014). Losses due to NH_3 volatilization in acidic forest soils are highly variable and range from 5% to 90% (Boomsma and Pritchett 1979, Craig and Wollum 1982, Kissel et al. 2004, Kissel et al. 2009, Zerpa and Fox 2011, Elliot and Fox 2014).

The amount of urea N lost from the system by NH_3 volatilization decreases the amount of fertilizer N potentially available to crop trees (loblolly pines) and likely contributes to low rates of fertilizer N uptake (Elliot and Fox 2014). Additional loss pathways of fertilizer N include denitrification (Shrestha et al. 2014) or leaching (Vitousek and Matson 1985a, Binkley et al. 1999, Meason et al. 2004, Aust and Blinn 2004). Although field experiments show crop tree fertlizer N uptake ranges from 25% to 50% (Baker et al. 1974, Mead and Pritchett 1975, Heilman et al. 1990, Johnson and Todd 1988, Li et al. 1991, Albaugh et al. 1998, 2004, Blazier et al. 2006), laboratory pot studies show plant uptake of fertilizer N ranges from 50% to near 100% in lodgepole pine (*Pinus contorta* Dougl. ex. Loud.) (Amponsah et al. 2004), Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) (Pang 1985), and black walnut (*Juglans nigra* L.) (Salifu et al. 2009). Conversely, in a 3 year old loblolly pine plantation in Oklahoma fertilized with urea and diammonium phosphate, fertilizer N recovery ranging from 6% to 25% (Blaizer et al. 2006). Mead et al. (2008) found on average 14.5% of fertilizer N was recovered 10 years after application in a 38 to 39 year old Douglas-fir stand in British Columbia. Heilman at al. (1990) found higher N recoveries, averaging 30% in 7 to 9 year old Douglas-fir forests in Washington. Unfortunately, none of these represent southeastern loblolly pine mid-rotation fertilization conditions.

The remaining fertilizer N not lost from the system or taken up by the crop trees is partitioned among other ecosystem components including the understory vegetation (competing volunteers, deciduous trees, shrubs, vines, herbaceous species), litter, forest floor (organic horizon), and mineral soil (Birk and Vitousek 1986, Albaugh et al. 2004). The cumulative effect of reduced fertilizer N uptake by desired crop trees translates to lower fertilizer nitrogen use efficiency (FNUE), defined as the ratio between the amount of fertilizer N removed with a crop and the amount of fertilizer N applied (Havlin et al. 2014). An improved understanding of the proportion of fertilizer N lost from the system, incorporated by crop trees and partitioned in ecosystem components is hindered by our inability to quantify the cycling of fertilizer N and its ultimate fate in forest ecosystems.

Although many loblolly pine plantations respond positively to the application of N and/or other nutrients, some do not (Pritchett and Smith 1975, Martin et al. 1999, Amateis et al. 2000, Carlson et al. 2014). Some stands may not respond to N additions because large amounts are lost from the system, immobilized in the soil by microbial communities or on exchange sites, or the site may not be N deficient due to high rates of mineralization from decomposing organic matter. Additionally, stands may not respond because deficiencies of elements other than N create nutrient imbalances that limit growth (Vogel and Jokela 2011, Carlson et al. 2014) and would respond to fertilization if the required nutrients could be properly identified and effectively applied.

The inability to accurately determine sites specific responsive to fertilization influences forest management decisions. Until our understanding of the mechanisms and interactions governing the movement of fertilizer N through loblolly pine plantations on a site-specific basis improves, the ability to accurately predict response will be limited. The goal of this research is to enhance our fundamental understanding of the fate of fertilizer N in loblolly pine plantations to improve the economic feasibility of fertilization and reduce potential negative environmental impacts of fertilizer N loss from the system. This research used N containing fertilizers enriched with the stable isotope ^{15}N in a series of pulse-chase experiments. We compared the fate and cycling of conventional (urea) and enhanced efficiency N containing fertilizers (coated Urea + NBPT (CUF), urea + NBPT (N-(n-Butyl) thiophosphoric triamide) (NBPT), polymer coated urea

(PCU)) in loblolly pine plantations. This research will help determine the feasibility of using enhanced efficiency N containing fertilizers to improve fertilizer nitrogen use efficiency.

1.2. Literature Review

1.2.1. The Nitrogen Cycle in Forest Ecosystems

Nitrogen is generally the most limiting element to primary productivity in terrestrial ecosystems because it is an essential component of key molecules such as deoxyribonucleic acid (DNA), ribonucleic acid (RNA), amino acids, proteins, enzymes that facilitate biochemical reactions, and photosynthesis (Miller 1981, Chapin et al. 1986, Vitousek and Howarth 1991, Lambers et al. 2005). Reduced N availability to trees restricts leaf area production, and forest plantations with low leaf area do not achieve optimal growth (Linder 1987).

Although terrestrial systems contain large quantities of N, only a small fraction is generally plant available at any given time (Likens and Bormann 1995, Fisher and Binkley 2000). While the atmosphere contains the largest pool of N (~79%), it exists as unreactive dinitrogen gas (N_2) and is not available for uptake by vascular plants. The strong triple bonds of N_2 must be broken and N must then be synthesized into plant available forms in biochemical reactions which include ammonium (NH_4^+), ammonia (NH_3), volatile organic compounds (VOCs), nitrate (NO_3^-), organic forms of nitrogen (e.g. urea), or from fertilizer production (Galloway et al. 2004, Lambers et al. 2005). In most forests, the primary N cycling pathways are biological fixation, mineralization, immobilization, nitrification, and denitrification (Fisher and Binkley 2000) (Figure 1.1).

Bonds of atmospheric N_2 can be broken by lightning, fertilizer production, nonsymbiotic (free living microbes) or symbiotic (microbes inside roots nodules of plants) microbial mediated dinitrogen fixation (nitrogen fixation) (Sylvia et al. 2005). Nitrogen fixation is the conversion of N_2 to ammonia (NH_3). Nonsymbiotic N fixation is low for most terrestrial ecosystems and confined to substrates high in energy but low in N, such as coarse woody debris (Groffman and Rosi-Marshall 2013). Conversely, symbiotic N fixation can be high where the symbiosis between plant and N fixing soil microbes provides a competitive advantage (Vitousek et al. 2002, Groffman and Rosi-Marshall 2013). Nitrogen fixation may decline with ecosystem succession but can still be important for N inputs to the system over extended periods (Grant and Binkley 1987, Vitousek et al. 2002).

Nitrogen mineralization is the transformation of organic N to NH_4^+, and immobilization is the incorporation of assimilation of NH_4^+ or NO_3^- into organic components. These processes are considered the primary sources of plant available N in most forested ecosystems (Miller et al. 1979, Likens and Bormann 1995, Groffman and Rosi-Marshall 2013). Nitrogen mineralization is partly controlled by soil microbial requirements for carbon (C) and N during substrate utilization, where C and N are incorporated into microbial biomass or lost through respiration. Adequate N in substrates used by soil microbes will generally translate to an accumulation of N in the soil that increases plant available N (Vitousek and Matson 1984, 1985b, Hart et al. 1994, Scott and Binkley 1997, Finzi et al. 1998, Piatek and Allen 1999, Vitousek et al. 2002). Yet if N is limiting in substrates utilized by soil microbes, N will be removed from mineral soil pools to satisfy microbial demand and immobilized, reducing plant available N (Vitousek and Matson 1985b, Davidson et al. 1990, Groffman et al. 1993, Scott and Binkley 1997).

Nitrification, the conversion of NH_4^+ to nitrite (NO_2^-) then nitrate (NO_3^-), is limited by soil NH_4^+ concentrations, because nitrifying microbes (bacteria and archaea) are generally poor competitors for NH_4^+ compared to heterotrophic microbes (Ward 2011, Norman and Barrett 2014, Norman et al. 2015). During nitrification N may be lost from the system due to high NO_3^- mobility (leaching). Nitrification is generally greater in systems with high N availability such as annually fertilized agriculture fields or recently disturbed ecosystems (Groffman and Rosi-Marshall 2013). Although NO_3^- may be lost from upland systems by nitrification, it can be transformed to N_2 or other gaseous N compounds by denitrification in anaerobic environments (Schimel and Bennett 2004, Ward 2011).

Denitrification is a form of anaerobic respiration utilizing N compounds along a redox gradient and sequence of electron acceptors that produce reduced products (Sylvia et al. 2005). Complete denitrification is the conversion of NO_3^- to N_2 with intermediary compounds of NO_2^-, nitric oxide (NO) and nitrous oxide (N_2O) (Sylvia et al. 2005). Dependent on the environmental conditions and microbial communities, incomplete denitrification may occur with intermediary compounds released from the system (Vitousek and Matson 1984, 1985b, Vitousek and Howarth 1991).

1.2.2. Productivity of Intensively Managed Pine Plantations

Forests provide numerous functions and services to society including clean water and air, carbon sequestration, biodiversity, food for human consumption, commodities for economic development, and diverse recreational opportunities (Nabuurs et al. 2007). Wood produced in forests is a primary economic commodity serving as a raw material for industries, especially in the southern United States (Howard 2003). Demands for both traditional forest products, such as pulp and timber, and new products for emerging markets such as the bioenergy industry, are increasing (Howard 2003). How to balance increasing demand for wood with non-commodity services and values produced by forests is a key question facing modern society (Fox 2000).

Most forests in the United States regenerate naturally, are extensively managed, and have low productivity (Fox et al. 2007b). The productivity of these extensively managed forests is not sufficient to produce the raw resources required to supply competing societal demands for forest products, and larger areas of natural forests will be required to meet commodity demands (Sedjo 2001). Yet high growth rates of intensively managed tree plantations can supply large quantities of wood and assist in meeting increasing demand for raw materials (Sedjo 2001, WWF 2015).

In the United States, plantation forests are primarily concentrated in the South (Oswalt et al. 2014) occupying 12.8 million ha of mostly loblolly pine (Albaugh et al. 2007). Intensive silvicultural management can significantly increase the productivity of these forests, from less than 2 $m^3\ ha^{-1}\ yr^{-1}$ to more than 10 $m^3\ ha^{-1}\ yr^{-1}$ (Fox et al. 2007b). Although improved growth rates have been achieved by intensive silviculture management, theoretical models and empirical field trials indicate productivity of 20 $m^3\ ha^{-1}\ yr^{-1}$ for 15 year rotations are possible in these southern pine plantations (Allen et al. 2005, Fox et al. 2007b). Increases in future productivity must include site-specific silvicultural prescriptions to ameliorate growth limiting factors in an economically viable and environmentally responsible manner.

1.2.3. Assessment methods for nutrient deficiencies in loblolly pines

Numerous methods have been developed to assess loblolly pine nutrient status and evaluate potential response to fertilization (Bowen and Nambier 1984, Carter 1992). The simplest method is visual observation (Stone 1968) but is of limited value because obvious symptoms (chlorosis) can be due to multi-nutrient deficiencies (Vogel or Jokela 2011) or pathogens (USDA 1989). Foliar analysis including nutrient concentrations, critical nutrient levels (Tisdale et al. 1985, Havlin et al. 2014), nutrient ratios in Diagnosis of Recommendation Integrated System (DRIS) (Hockman and Allen 1990, Sypert 2005), and graphical analysis (Haase and Rose 1985) improve on visual assessment, but also may be of limited value if sampling season and edaphic factors are not integrated (Carter 1992). Soil sampling and nutrient addition experiments (Binkely and Hart 1989) also provide insight but may require large sample sizes to address soil heterogeneity. Integrated soils research has led to the development of soil groupings based on similar parent materials within physiographic regions that correlate well with specific nutrient deficiencies, like the Cooperative Research in Forest Fertilization (CRIFF) soil groupings in the southern United States (Fisher and Garbett 1980, Jokela and Long 2000, Villanueva 2015), or groupings in the Pacific Northwest (Littke et al. 2014). Field or greenhouse bioassays using seedlings can also be valuable, but results may be confounded by artificial laboratory conditions and may be difficult to extrapolate to the field (Addoms 1937, Hobbs 1947, Morrison 1974, Mead and Pritchett 1975, Birk and Vitousek 1986). The most reliable, but expensive, assessment of nutrient deficiencies and fertilizer N response are fertilizer field trials (Miller 1981, Carter 1992, Albaugh et al. 2004, Fox et al. 2007a).

1.2.4. Nitrogen Fertilization of Pine Plantations

Low availability of plant available soil nutrients, primarily N (Albaugh et al. 1998, Sampson and Allen 1999, Jokela and Martin 2000, Fox et al. 2007b) and P (Pritchett and Comerford 1982, Gent et al. 1986, Fox et al. 2007b) reduce productivity of many pine plantations across the southern United States. Low nutrient availability reduces leaf area production and decreases photosynthetic capacity, and stands with suboptimal leaf area do not grow at their maximum potential (Linder 1987, Fox et al. 2007b). Fertilization of these systems can increase soil nutrient availability, translating to increased leaf area and hence improved stand productivity. Empirical results from fertilization field trials in intensively managed loblolly pine plantations show most nutrient limitations can be ameliorated with fertilization (Fox et al. 2007a).

Nutrient limitations for plants in most terrestrial ecosystems develop when the nutrient demand exceeds the nutrient supply (Miller 1981, Chapin et al. 1986, Allen et al. 1990). In forest plantations, stand N uptake progresses in a sigmoidal pattern, increasing with stand development as plant N demand becomes unbalanced with plant N availability (Figure 1.2) (Switzer and Nelson 1972, Wells and Jorgenson 1975). Plant N availability in the soil can increase after site disturbances, such as forest harvesting because conditions (temperature, moisture, aeration) favor decomposition of the forest floor, coarse woody debris, and root turnover (Vitousek and Matson 1985, Fox et al. 1986). As the stand age progresses, N is increasingly immobilized in pools with varying turnover times, and the environmental conditions conducive to high N availability decrease. The plant available N required to maintain maximum growth gradually shifts until plant demand for N exceeds the supply of plant available N in the soil (Miller 1981). As the stand transitions to plant available N limitation, N fertilization is required to maintain optimal tree growth (Figure 1.2).

Fertilization studies using 224 kg ha^{-1} N plus 30 kg ha^{-1} P on loblolly pine plantations in the southern United States show a mean fertilization growth response of 3 m^3 ha^{-1} yr^{-1} over 8 years (Fox et al. 2007a) which has led to the fertilization of over 500,000 ha of these systems annually (Albaugh et al. 2007). Despite a positive response for most pine plantations to N + P fertilization in the South, certain sites fail to respond or respond negatively (Pritchett and Smith

1975, Martin et al. 1999, Amateis et al. 2000, Carlson et al 2014). These inconsistent fertilization results in southern pine plantations have led to a range in growth of 0 m^3 ha^{-1} yr^{-1} to 10 m^3 ha^{-1} yr^{-1} (Fox et al. 2007b).

Several empirical field trials indicate less than 50% of fertilizer N is taken up by loblolly pine trees (Mead and Pritchett 1975, Albaugh et al. 1998, Blazier et al. 2006). The remaining fertilizer N is partitioned into other ecosystem components with varying turnover timescales that include: 1) loss from the system (ammonia (NH_3) volatilization, leaching, nitrification, denitrification); 2) understory (volunteer loblolly pine, deciduous tree/shrub/vine competition, herbaceous species); 3) litterfall; 4) forest floor; 5) soil microbial community; and/or 6) mineral soil (Figure 1.1 and 1.3) (Birk and Vitousek 1986, Albaugh et al. 2004). The fertilizer N remaining in the system may become available to the loblolly pines in the future (Meason et al. 2004), but uncertainty remains concerning mechanisms controlling fertilizer N turnover from ecosystem components. The high variability and uncertainty involved with understanding crop N tree uptake, losses, pathways and mechanisms of fertilizer N cycling may contribute to the variability in growth response observed in fertilizer studies.

1.2.5. Cycling of Fertilizer Nitrogen in Pine Plantations

The most common fertilizer N source used in southern loblolly pine plantations is pelletized urea ($CO(NH_2)_2$) which is surface applied via aerial or ground broadcast methods (Allen 1987). Urea is preferred because of a high N content (46% N) and ease of transport-storage-application, translating to the lowest overall cost per pound of any type of fertilizer N (Gould et al. 1986, Allen 1987, Harre and Bridges 1988, Fox et al. 2007b). Other fertilizer N forms besides urea may be used, but their use is dependent primarily on associated costs (storage, delivery, application, etc.) and regional availability (Havlin et al. 2014, IPNI 2015, PSU 2015) (Table 1.1). Yet fertilizer N cycling in loblolly pine plantations is uncertain, and large losses can occur following urea application which translate to reduced fertilizer nitrogen use efficiency (Figure 1.3).

In the acidic forest soils supporting southern loblolly pine plantations, a urea granule undergoes a series of chemical reactions (Hauck and Stephenson 1965). Urea hydrolysis is the initial chemical reaction and is facilitated by the extracellular enzyme urease that is prevalent in forest soil (Conrad 1942, Pettit et al. 1976, Marsh et al. 2005). This initial hydrolysis reaction produces ammonium carbonate that dissociates to ammonium (NH_4^+) and bicarbonate (HCO_3^-). The bicarbonate consumes hydrogen (H^+) ions near the dissolving urea granule which raises the surrounding pH. The ammonium ions (NH_4^+) can be converted to NH_3 from the elevated pH near the urea granule and lost to the atmosphere by NH_3 volatilization (Koelliker and Kissel 1988).

Urea N losses via NH_3 volatilization in pine plantations are highly variable, but can be rapid and significant (Nômmik 1973, Kissel et al. 2004, Cabrera et al. 2010, Zerpa and Fox 2011, Elliot and Fox 2014). Losses from NH_3 volatilization after urea fertilization range from less than 10% (Boomsma and Pritchett 1979, Craig and Wollum 1982), 10% to 40% (Nômmik 1973, Zerpa and Fox 2011), to greater than 50% (Kissel et al. 2004, Kissel et al. 2009, Elliot and Fox 2014). Although factors influencing NH_3 volatilization following urea fertilization are well researched in agriculture (Volk 1970, Black et al. 1987, Kissel et al. 1988), less research has focused on forests (Volk 1970, Nômmick 1973, Kissel et al. 2004, Elliot and Fox 2014). Results from studies primarily in agroecosystems on NH_3 volatilization losses are correlated with soil pH (Ernst and Massey 1960, Cabrera et al. 1991, Kissel et al. 2009), soil moisture (Clay et al. 1990, Kissel et al. 2004), type of mineral soil substrate (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011), relative humidity (Cabrera et al. 2005), soil temperature (Ernst and Massey 1960, Clay et al. 1990, Moyo et al. 1989), surficial wind speed (Watkins et al. 1972, Kissel et al. 2004), precipitation (Craig and Wollum 1982, Kissel et al. 2004) and air temperature (Gould et al. 1973, Craig and Wollum 1982, Koelliker and Kissel 1988). A forest floors (organic horizon), present in forests but not agriculture systems, can also effect NH_3 volatilization (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011) because urea N after dissolution may enter voids in decomposing pine needles and be volatilized as environmental conditions change (Cabrera et al. 2005).

Urea N lost from after fertilization from NH_3 volatilization is difficult to quantify because fertilizer N losses are caused by the interaction among many factors. Because larger NH_3 volatilization losses generally occur with higher temperatures and relative humidity (Craig and Wollum 1982, Ferguson and Kissel 1986, Moyo et al. 1989), urea is usually applied during the winter in the South under cooler, wetter conditions to reduce the likelihood of high NH_3 volatilization losses (Ferguson and Kissel 1986, Moyo et al. 1989, Cabrera et al. 2010). Under these conditions, urea dissolution and movement of fertilizer N into the soil increases (Black et al. 1987, Paramasivan and Alva 1997). Large urea losses via NH_3 volatilization have occurred under even under low temperatures (Carmona and Byrnes 1990, Engel et al. 2011). For example, Kissel et al. (2004) observed low NH_3 volatilization in August when NH_3 volatilization is typically high because urea was applied on the same day as a significant precipitation event, but high NH_3 volatilization (45% to 58%) was observed during the same period under simulated, minor precipitation events. These highly variable results for NH_3 volatilization after urea fertilization highlight the difficulty in predicting the magnitude of urea N loss from NH_3 volatilization after N fertilization at any time of the year.

When environmental conditions are not conducive to NH_3 volatilization after urea dissolution, NH_4^+ enters the soil which is enhanced by precipitation. The NH_4^+ that moves into the soil is by soils with a high H^+ buffering capacity (high organic matter, clay, silt) (Fenn and Kissel 1976, Ferguson et al. 1984). Once in the soil, NH_4^+ may: 1) undergo nitrification to form nitrate (NO_3^-); 2) become immobilized by soil microbes; 3) taken up by plants (loblolly pines, understory competition); or 4) be leached from the surface soil (Figure 1.1 and 1.3). Because most pine plantations are N deficient and fertilized intermittently, fertilizer N cycling is tightly coupled in processes 1 to 3 with 4 (leaching) a minor long term loss (Binkely et al. 1999).

1.2.6. Enhanced Efficiency Fertilizers

Urea applied during cooler, wetter months in pine plantations of the southern United States reduces the potential for NH_3 volatilization (Allen 1987, Allen et al. 2005, Fox et al. 2007a). During winter, lower mean temperatures and higher mean precipitation increases the probability of urea dissolution and movement into the soil, hence reducing NH_3 volatilization loss. Yet conditions following winter urea fertilization in the South can still favor high rates of NH_3 volatilization (high relative humidity, temperatures, wind speeds, low precipitation). Additionally, winter urea application may be asynchronous to seasonal plant N demand, decreasing FNUE (Blazier et al. 2006). One alternative to urea application in cooler, wetter conditions to minimize NH_3 volatilization is the use of slow or controlled release fertilizers, termed enhanced efficiency fertilizers (EEFs) (Hauck 1995, Azeem et al. 2014, IPNI 2015).

To improve FNUE in agroecosystems, slow release (SRN), controlled release (CRN) and stabilized (SNF) N fertilizers were developed (Azeem et al. 2014, Fertilizer Institute 2015). CRN is fertilizer N that is coated or encapsulated to alter the rate, pattern and duration of N release

(Chien et al. 2009). SRN are N fertilizer formulations that gradually release fertilizer N slowly as a result of microbial decomposition (Trenkel 1997). SNF is fertilizer N that is treated with chemical compounds that inhibit rapid N transformation to less stable forms (Shaviv 1996, 2005). The SRN, CRN and SNF products can all be broadly categorized as enhanced efficiency fertilizers (EEFs) (AAPFCO 2015). The Fertilizer Institute (2015) defines EEFs as fertilizer products that reduce nutrient loss to the environment while increasing plant nutrient availability by slowing nutrient release or conversion of the nutrient to forms less susceptible to losses. The EEF N containing fertilizers are designed to: 1) increase N uptake by the crop; 2) decrease N leaching; 3) lower toxicity; 4) provide extended N supply; 5) reduce NH_3 volatilization and/or nitrification losses of N; and 6) lower application cost (Allen 1984, Hauck 1985). Therefore, EEFs can reduce fertilizer N loss and provide greater flexibility for fertilization under a variety of weather conditions that enhance fertilizer N uptake by the crop (Hauck 1985, Goertz 1993, Azeem et al. 2014). Although EEFs were developed to address FNUE issues in agroecosystems, similar issues related to FNUE exist for southern pine plantations. Implementing EEF technology into forest fertilization programs in southern pine plantations may improve FNUE and increase the productivity of these systems. Table 1.2 highlights several EEFs used primarily in turf grass management, horticulture, and high value agricultural products and was primarily adapted from Havlin et al. (2014). If manufacturing costs of these products decreases in the future, many may become more applicable to large scale agricultural and forestry (Trenkel 2010).

One SNF mechanism to reduce urea N loss is to add urease inhibitors to urea prior to fertilization. Urease inhibitors are substances which temporarily inhibit the hydrolytic action on urea by the urease enzyme, resulting in less urea N lost through NH_3 volatilization (AAPFCO 2015). These products generally reduce urease activity for two to three weeks after fertilizer application (Gowariker and Krishnamurty 2009, Havlin et al. 2014) although under certain conditions effectiveness can be longer (Engel et al. 2011). Urease is abundant in soils and produced by numerous soil organisms and plants (Bremner and Douglas 1971, Bremner and Chai 1986, Antisari et al. 1996, Sanz-Cobena et al. 2008). When urease inhibitors are released to the environment, they bind to the urease enzyme, and reduce the enzyme activity to provide time for urea to dissolve and move into the soil. Once urea is in the soil, loss via NH_3 volatilization is reduced because the soil buffers the pH. Many compounds inhibit urease activity, but few of these compounds are chemically stable when added to urea, effective when applied at low concentrations and/or are non-toxic when applied in the environment (Hauck 1985, Azeem et al. 2014). The most widely used urease inhibitor is N-(n-Butyl) triphosphoric triamide (NBPT) (Havlin et al. 2014). Urea impregnated with NBPT can reduce losses of fertilizer N via NH_3 volatilization significantly compared to untreated urea (Engel et al. 2011, Raymond et al. 2016) and may translate to improved productivity for desired species (Engel et al. 2011).

The CRN products generally have a urea granule base which is initially coated with a compound (i.e. sulfur (S), boron, (B), copper (Cu)) and additionally sealed with a wax-like substance to fill imperfections (Chien et al. 2009). Fertilizer N release from CRN depends on the thickness and coating quality (Allen 1984). As the coating degrades in the environment, cracks develop in the coating allowing for moisture to enter the product, and urea dissolution is initiated. Urea N release to the environment is slowed by the coating compared to uncoated urea. The substrate coating of CRN may reduce urease activity, and CRNs may also be impregnated with urease or nitrification inhibitors to slow loss mechanisms as urea N is released to the environment. Additionally, CRN products may include other nutrients in the coatings to address additional nutrient deficiencies.

An alternative CRN approach encapsulates fertilizer N (generally urea) in a semi-permeable polymer coating. The coatings have small pores which allow water to enter the capsules. As water enters the CRN product, urea dissolution occurs, the capsule expands, and the resulting internal pressure of the capsule forces fertilizer N solution into the environment through diffusion (Shaviv 2005). Because of the low mass of the polymer coatings, these products do not significantly reduce the N percentage in the product as may occur with the aforementioned CRN products (Chien et al. 2009). Release patterns for polymer coated CRN products are related to the type of coating, soil moisture, temperature, relative humidity, precipitation, pH, and microbial activity (Christianson 1988, Shoji et. al. 2001, Shaviv 2005).

1.2.7. Use of Stable Isotopes to Trace Nitrogen in Forest Ecosystems

Stable isotopes, specifically ^{15}N, can be used to assess of nutrient deficiencies and quantify fertilizer N uptake (Hauck 1968). Fertilizers enriched with stable isotopes (^{15}N) can trace the ultimate fate of applied N (Walker 1958, Hauck 1968, Powlson and Barraclough 1993) and integrate ecosystem processes (Robinson 2001, Dawson et al. 2002, Templar et al. 2012). Numerous ^{15}N tracer studies were initially conducted in agroecosystems (Hauck and Bystrom 1971), but ^{15}N tracer studies have also improved the understanding of N dynamics in natural (Tietema et al. 1998, Currie and Nadelhoffer 1999, Dinkelmeyer et al. 2003, Nadelhoffer et al. 2004, Templar et al. 2012) and plantation (Walker 1958, Hauck 1968, Mead and Pritchett 1975, Melin et al. 1983, Clinton and Mead 1994, Chang et al. 1996, Bubb et al. 1999, Mead et al. 2008, Werner 2013) forests. The use of stable isotopes is often combined with other analytical methods to improve the understanding of nutrient status and fertilizer responsiveness (Miller 1981, Birk and Vitousek 1985, Sypert 2005).

Fertilizers labeled with the stable isotope ^{15}N can be traced through the ecosystem and improve the understanding of the fate and cycling of N in forests (Peterson and Fry 1987, Knowles and Blackburn 1993, Nadelhoffer and Fry 1994, Dawson et al. 2002, Weatherall 2005, Templar et al. 2012). The productivity of most forests is limited by N despite relatively large quantities of total N in the soil (Fisher and Binkley 2000). This is because only small amounts of plant available N exist at any point in time in the ecosystem (Chapin et al. 1986, Vitousek and Matson 1985, Fisher and Binkley 2000). The majority of N, ranging from 2 Mg ha^{-1} to 7 Mg ha^{-1}, is in the forest floor and mineral soil (Cole and Rapp 1981, Fahey et al. 1985, Likens and Bormann 1995, Fisher and Binkley 2000). Because of the large quantity of natural soil N, it is difficult to distinguish between the N that occurs naturally in the soil and the N that originates from the fertilizer (Nômmik 1990, Kiser and Fox 2012).

Response to fertilizer N is often assessed through changes in ecosystem metrics such as foliar N concentration, leaf area index (LAI), C:N ratios, nutrient ratios, tree diameters, rooting profiles, photosynthetic rates, soil respiration, soil solution measurements, and gross-net N mineralization. Although these metrics provide valuable information on system responses to fertilizer N, it can be difficult to discern pathways, mechanisms and partitioning of fertilizer N within the ecosystem. Because of the difficulty in distinguishing N originating from the natural environment or fertilizer, fertilizers can be enriched with stable isotopes (^{15}N) to improve the understanding of the effects of fertilization on ecosystem processes.

Elements naturally occurring in the environment have one or more stable isotopes with slightly different molecular weights due to a difference in neutrons (Fry 2006). The ratio of different isotopes in a compound is measured with an isotope ratio mass spectrometer (IRMS). Current analytical techniques allow for the simultaneous measurement of the isotopic composition of several elements (C, N, O, H, S as examples) on an IRMS that can be coupled with an elemental analyzer for nutrient concentration analysis on a single sample (Sulzman 2007).

During the IRMS analysis, the isotopic ratios of the compound are compared to a standard ratio, and the difference between these ratios is expressed as the del value (δ) or per mil (‰) (Fry 2006, Sulzman 2007) through the formula:

[1.1] $\delta^{15}N = ((R_{sample}/R_{standard})-1) * (1000)$

The R_{sample} is the ratio of the heavier isotope to lighter isotope of the sample, and the $R_{standard}$ is the ratio of the heavier isotope to lighter isotope of the standard (Fry 2006, Sulzman 2007). The standard used for N stable isotope analysis is atmospheric N_2 gas which has a $\delta^{15}N$ of 0. Samples enriched in the heavier isotope will have positive δ value whiles samples enriched with the lighter isotope will have negative δ values.

Two naturally occurring stable isotopes exist for N, ^{14}N (99.63%) and ^{15}N (0.3663%) (Sulzman 2007). Although both isotopes behave similarly in the environment, certain kinetic and biological reactions favor the lighter isotope (^{14}N) in reaction products, which enriches remaining substrate(s) in the heavier isotope (^{15}N) in a process termed fractionation (Fry 2006). Fractionation can create distinct isotopic signatures in ecosystem components, with $\delta^{15}N$ values ranging from -15 to +2, with increasingly negative values observed in vegetation and positive values in deeper soil depths (Nadelhoffer and Fry 1994, Ben-David et al. 1998).

Nitrogen additions to ecosystems enriched with ^{15}N improve the precision and accuracy of tracing fertilizer N movement through the ecosystem by measuring ^{15}N accumulation in each ecosystem component (tree, competing vegetation, soil, etc.) (Powlson and Barrachlough 1993). This ^{15}N tracer technique has been used in many ecosystems to improve the understanding of N cycling including agriculture (Hauck 1973, Hauck and Bremner 1976), forests (Nadelhoffer and Fry 1994, Templar et al. 2012), stream ecology (Simon et al. 2003), oceanography (Kline 1999), and food webs (Ben-David et al.1998, Setälä et al. 2002). Yet due to the high expense of producing ^{15}N labeled substrates, their use is generally limited to smaller field and laboratory experiments.

Two techniques using ^{15}N as a tracer in forest ecosystem research are: 1) using small amounts (few kg N ha^{-1}) of heavily enriched (60-90 Atom Percent- AP) ^{15}N labeled fertilizer; or 2) larger amounts (few hundred kg N ha^{-1}) of lightly enriched (2.5-10 AP) ^{15}N labeled fertilizer. Forest ecosystem research has used both techniques, from highly enriched, low N concentration additions (Koopmans et al. 1996, Seely and Lajtha 1997, Tietema et al. 1998, Perakis and Hedin 2001) to slightly enriched, higher N concentration additions (Currie and Nadelhoffer 1999, Dinkelmeyer et al. 2003, Nadelhoffer et al. 2004). Perakis et al. (2005) applied a gradient of enriched $^{15}NH_4{}^{15}NO_3$ to an ecosystem, ranging from 0.2 kg N ha^{-1} 99 AP to 640 kg N ha^{-1} 2.5 AP in an attempt to optimize tracer techniques. Their results found that both slightly and highly enriched substrates were detectable as tracers through the ecosystem and the decision of which technique to use should be based more on the specific research question.

This study will use fertilizers enriched with ^{15}N (0.5 AP, ~370‰) to trace the fate of fertilizer N in intensively managed mid-rotation loblolly pine plantations across the southern United States. The ^{15}N enrichment is 20% greater than the natural abundance range for forest ecosystem components, ranging from 0.3626 AP to 0.3718 AP (Nadelhoffer and Fry 1994). This study balances the tracer technique suggested by Nadelhoffer and Fry (1994) by using moderate enrichments of 0.5 AP and high N concentrations (urea, 46% N) while simulating the standard operational fertilizer N application rate (224 kg N ha^{-1}) in southern industrial pine plantations.

1.3. Objectives

The objective of this study is to assess the fate of fertilizer N through the use of four fertilizer formulations labeled with the stable isotope ^{15}N in mid-rotation loblolly pine plantations across the southern United States. Specific focus will be on N uptake by loblolly pines. The four fertilizer formulations to be assessed include: 1) urea; 2) monoammonium phosphate (MAP) coated urea + NBPT (CUF); 3) urea + NBPT (NBPT); and 4) a homogenous polymer coated controlled release fertilizer (PCU). All fertilizer N formulations will be applied at the industry standard of 224 kg ha^{-1} N combined with 28 kg ha^{-1} P. Two application seasons, spring (March-April) and summer (June) will be assessed. Ammonia volatilization will be measured following fertilization. Results from this study will provide information on the fundamental cycling of fertilizer N in loblolly pine plantations across the southern United States to improve fertilization techniques that will translate to improved economic efficiencies and environmental stewardship concerning fertilization in these systems.

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Figures

Figure 1.1. *The nitrogen cycle in forested ecosystems. Adapted from Fisher and Binkley (2000), Dawson et al. (2002), Sylvia et al. (2005), Groffman and Rosi-Marshall (2013).*

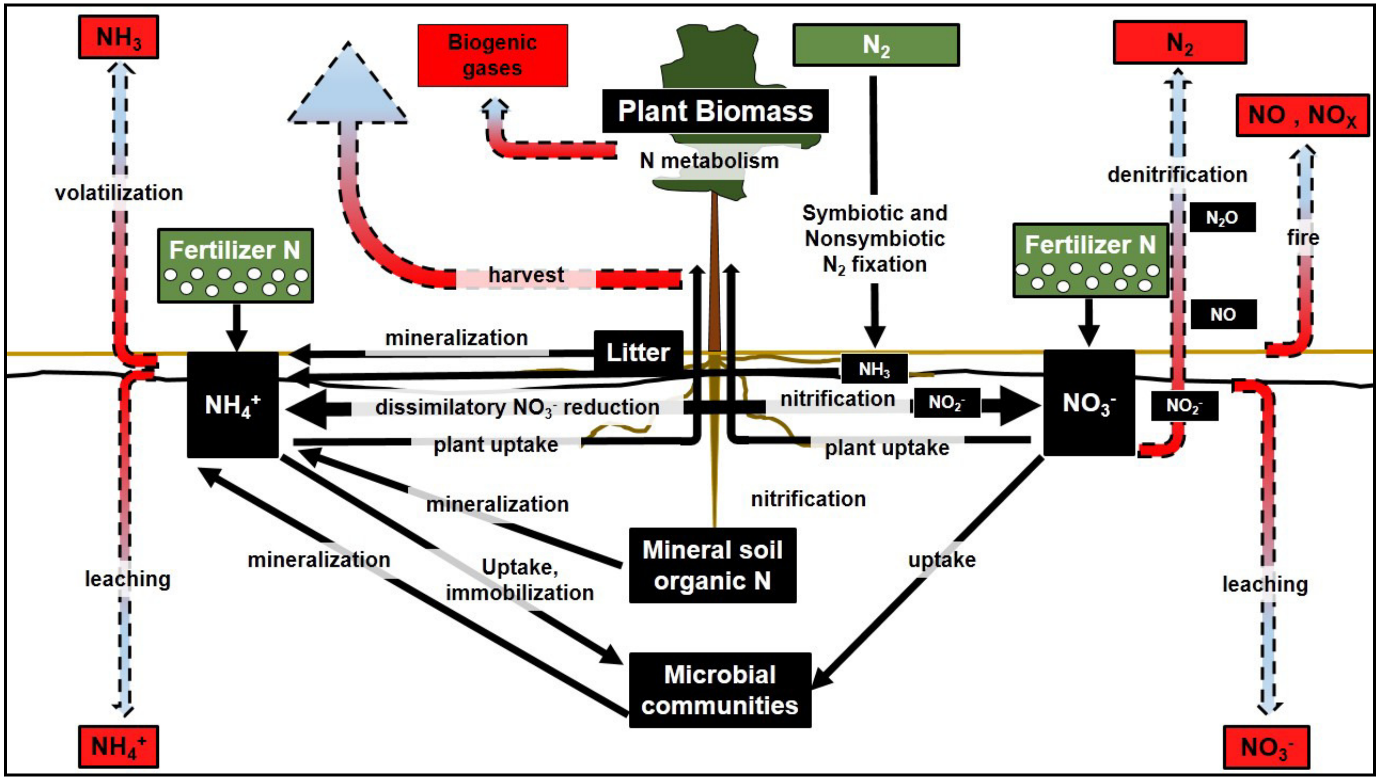


Figure 1.2. *Soil nitrogen supply and tree nitrogen demand during a typical rotation for loblolly pine in the southern United States. Adapted from Allen (1990) and Fox et al. (2007a).*

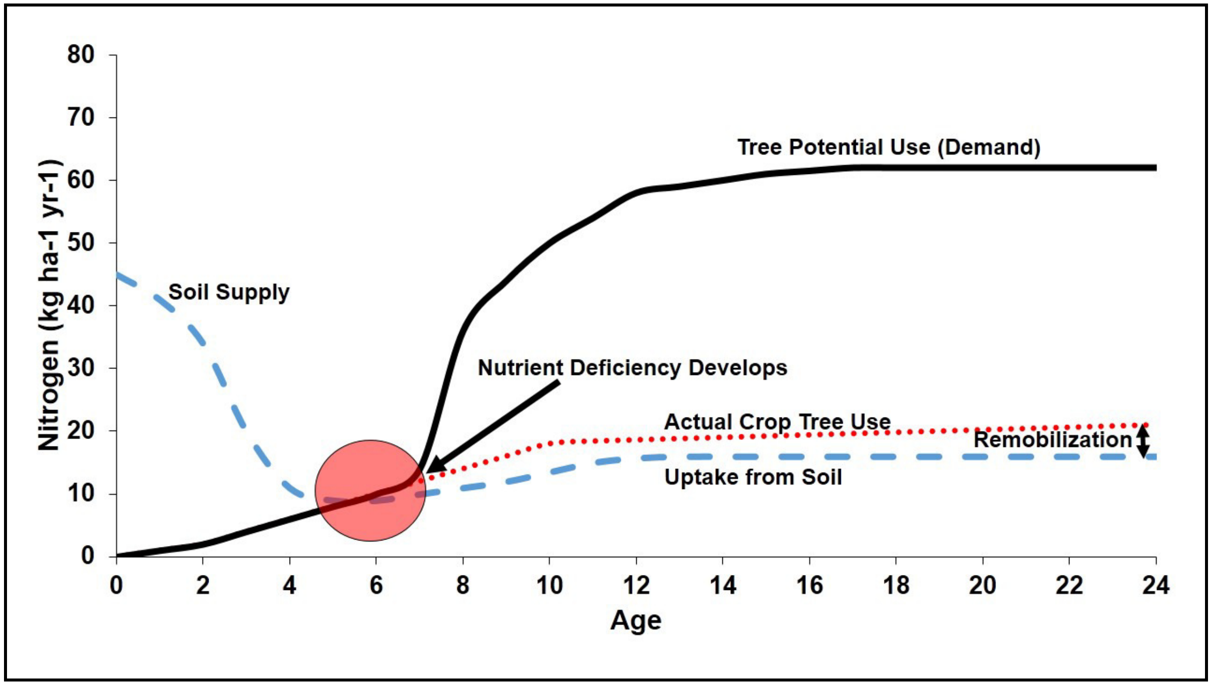
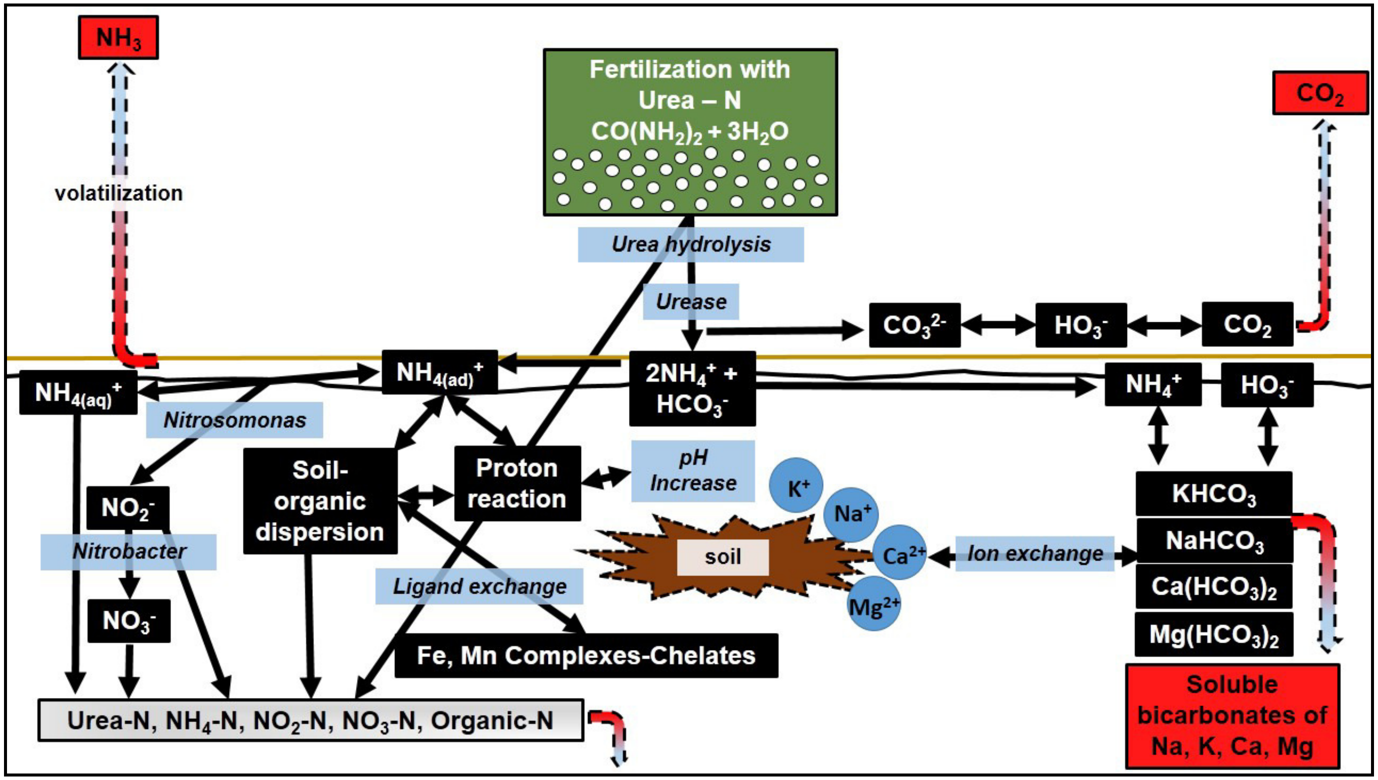


Figure 1.3. *The fertilizer nitrogen cycle in a coniferous forested ecosystem. Adapted from Crane (1972).*



Tables

Table 1.1. *Common types of nitrogen containing fertilizers for use in turf grass management, horticulture, agriculture and forestry. Top portion of list has general applications to forestry. Information summarized from tables specific to N containing fertilizers in Havlin et al. (2014), IPNI (2015) and PSU (2015).*

Nitrogen Fertilizer Source	**N** (%)	**P_2O_5** (%)	**K_2O** (%)	**Ca** (%)	**Mg** (%)	**S** (%)	**Cl** (%)	**Application Form**
Urea ($CO(NH_2)_2$, (NH_2CONH_2))	45-46	0	0	0	0	0	0	Solid
Ammonium nitrate (NH_4NO_3)	33–34	0	0	0	0	0	0	Solid
Ammonium sulfate $(NH_4)_2SO_4$	21	0	0	0	0	24	0	Solid
Diammonium phosphate (DAP) $(NH_4)_2HPO_4$	18-21	46-54	0	0	0	0	0	Solid
Monoammonium phosphate (MAP) $NH_4H_2PO_4$	10-12	48-61	0	2	2	1-3	0	Solid
Compound fertilizers	10, 12, 17, 21	10, 12, 17, 7	10, 12, 17, 14	0	0	0	0	Solid
Ammonium polyphosphate $[NH_4 PO_3]_n$	10	34	0	0	0	0	0	Liquid
Potassium nitrate (KNO_3)	13	0	44	0.5	0.2	0.2	1.2	Solid
UAN (urea-ammonium nitrate) (Urea + NH_4NO_3 + H_2O)	28-32	0	0	0	0	0	0	Liquid
UAP (urea-ammonium phosphate)	21-38	13-42	0	0	0	0	0	Solid
Urea phosphate ($CO(NH_2)_2 \cdot H_3PO_4$)	17	43-44	0	0	0	0	0	Solid
Urea sulfate ($CH_6N_2O_5S$)	30-40	0	0	0	0	6-11	0	Solid
Anhydrous ammonia (NH_3)	82	0	0	0	0	0	0	Liquid-Gas
Ammonium bicarbonate (NH_4HCO_3)	21-23	0	0	0	0	0	0	Solid
Ammonium chloride (NH_4Cl)	25-26	0	0	0	0	0	66	Solid
Ammonium thiosulfate ($H_8N_2O_3S_2$)	12	0	0	0	0	26	0	Liquid
Calcium ammonium nitrate ($5Ca(NO_3)_2 \cdot NH_4 \cdot NO_3 \cdot 10H_2O$)	15-27	0	0	9-19	0	0	0	Solid-Liquid
Ammonium polyphosphate ($H_{n+2}P_nO_{3n+1}$)	10-11	34-37	0	0	0	0	0	Liquid
Calcium nitrate (CAN) ($Ca(NO_3)_2$)	15	0	0	34	0	0	0	Solid
Sodium nitrate ($NaNO_3$)	16	0	0	0	0	0	0.6	Solid

Table 1.2. *Common types of enhanced efficiency fertilizer products for use with nitrogen containing fertilizers in agriculture, horticulture, and turf grass management. Certain products have current applications to forestry. Information summarized from tables specific to slow and controlled release fertilizers found in Hauck (1985), Shaviv (1996), Trenkel (2010), Azeem et al. (2014), Havlin et al. (2014), IPNI (2015) and PSU (2015). Primary resource was Table 4-23 in Havlin et al. (2014).*

Enhanced Efficiency Fertilizer	N Source	Common Name(s)	N Content (%)	Inhibition Duration (weeks)
Coated				
Sulfur coated urea	Urea	SCU	30-42	4-12
		Enspan	39	
		CUF		
Polymer and/or S coated urea	Urea	PolyPlus Poly-S TriKote	38-42	6-16
		XCU	41-43	
Polymer or resin coated urea	Urea	Polyon, Osmocote,	38-44	8-14
		Meister	25-46	
		Agriform Multicote		
		Escote		
		Prokote		
		ESN		
		Nutrisphere		
Insoluble Organic N				
Urea formaldehyde	Ureaforms Methylene urea Methylol urea Polymethylene urea	Nitroform	38	10-30+
		FLUF	18	6-10
		Folocron	29	
		GP-4340	30	
		Nutralene	40	7-12
		Nydrolene		
		Nitamin		
		Resi-Grow	30	6-10
		CoRoN	12 or 38	7-9
Isobutylidene diurea	Isobutylidine urea	IBDU	31	10-16
Triazone	Triazone/urea	N-Sure, Nitamin TriSert, Formolene	28-33	6-10

Enhanced Efficiency Fertilizer	N Source	Common Name(s)	N Content (%)	Inhibition Duration (weeks)
Crotonylidene diurea	Urea/crotonaldehyde	Crotodur, CDU, Triabon	34	6-12
Melamine	2,4,6 triamino- 1, 3, 5-triazine	Nitrazine	50-60	6-12
DCD	Dicyandiamide	DCD Ensan	1.6 0	4-8
DMPP	3,4-dimethpyrazole phosphate	DMPP ENTEC	0 12-26	6-8 12
Thiosulfate	Ammonium or calcium thiosulfate	ATS CaTS	12 0	2-3
DCD + NBPT	Dicyandiamide + N-(n-butyl) thiophosphoric triamide	Agrotain Plus HYDREXX	0 0	6-8
DCD + NBPT + urea		UMAXX UFLEXX SuperU	47 46	8-12 6-8
Polymer	Maleic-itaconic copolymer	Nutrisphere	0	6
Polymer + urea		SSN	46	6-12
Inhibitors				
Nitrapyrin	2-chloro-6-trichloromethyl pyridine	N-Serve Stay-N 2000	0 0	2-6
NBPT	N-(n-butyl) thiophosphoric triamide	Agrotain SuperU	0 46	2-3

Chapter 2. Ammonia volatilization following nitrogen fertilization with enhanced efficiency fertilizers and urea in loblolly pine (*Pinus taeda* L.) plantations of the southern United States

Abstract

Ammonia (NH_3) volatilization losses following surface application of urea and three enhanced efficiency nitrogen (N) containing fertilizers (EEFs) were compared in six thinned mid-rotation loblolly pine stands (*Pinus taeda* L.) across the southern United States. All fertilizer treatments were labeled with ^{15}N (~370 permil, 0.5 AP) and applied during two different seasons (spring, summer) in 2011 to open chamber microcosms. Individual microcosms were sampled 1, 15 and 30 days after fertilization to estimate remaining ^{15}N. Losses of fertilizer N were determined using a mass balance calculation. Significantly less N loss occurred following fertilization with EEFs compared to urea after all sampling days for both seasons. Because root uptake was eliminated in the microcosms and there was no leaching of ^{15}N below the microcosms, the most likely loss pathway of the ^{15}N from the microcosms was NH_3 volatilization. There were generally no differences among the individual EEFs. Following spring application, the mean NH_3 volatilization during the 30 day experiment ranged from 4% to 26% for the EEFs compared to 26% to 40% for urea. In summer, mean NH_3 volatilization for EEFs ranged from 8% to 23% compared to 29% to 49% for urea. This research highlights the potential of EEFs to reduce loss of fertilizer N in forest systems, potentially increasing fertilizer N use efficiency in these pine plantations.

2.1. Introduction

The productivity of many loblolly pine (*Pinus taeda* L.) plantations in the southern United States is limited by low levels of plant available soil nutrients, especially nitrogen (N) and phosphorous (P) (Allen 1987). These N deficiencies are common because N is required in larger quantities compared to other nutrients for the formation of foliar tissue and photosynthetic enzymes of cellular structure (Miller 1981, Chapin et al. 1986). Nitrogen deficiencies can generally be ameliorated through fertilization (Fox et al. 2007a, Carlson et al. 2014). In the South, loblolly pine plantations generally respond positively to a mid-rotation fertilization with a mean growth increase of 3 m^3 ha^{-1} over 8 years following the application of 224 kg ha^{-1} N plus 30 kg ha^{-1} of P (Fox et al. 2007a). Consequently, fertilization has become an important silvicultural tool to improve forest productivity (Allen 1987, Fox et al. 2007b). Despite these systems being N deficient, less than 50% of applied N fertilizer is usually utilized by loblolly pines, with some studies indicating a much lower percentage (Baker et al. 1974, Mead and Pritchett 1975, Johnson and Todd 1988, Li et al. 1991, Albaugh et al. 1998, 2004, Blazier et al. 2006).

The most common N fertilizer used in loblolly pine plantations in the South is pelletized urea ($CO(NH_2)_2$) which is usually surface applied via aerial or ground broadcast methods (Allen 1987). Urea is used because of its high N content (46% N) and ease of transport-storage-application, translating to the lowest overall cost per pound of applied N (Gould et al. 1986, Allen 1987, Harre and Bridges 1988, Fox et al. 2007b). In the acidic forest soils of the South that support loblolly pine plantations, urea undergoes a series of chemical reactions that can lead to ammonia (NH_3) volatilization losses (Hauck and Stephenson 1965).

[2.1] $(CO(NH_2)_2) + H^+ + 2H_2O \xrightarrow{\text{urease}} 2NH_4^+ + HCO_3^-$ (hydrolysis)

[2.2] $NH_4^+ \longrightarrow NH_{3(d)} + H^+$ (dissociation)

[2.3] $NH_{3(d)} \longrightarrow NH_{3(g)}$ (NH_3 volatilization)

The initial reaction, urea hydrolysis, is facilitated by the extracellular enzyme urease which originates from plant and animal residues and microbial activities and is common in forest soils (Conrad 1942, Pettit et al. 1976, Marsh et al. 2005). Urea hydrolysis produces ammonium carbonate which dissociates into ammonium (NH_4^+) and bicarbonate (HCO_3^-). The bicarbonate consumes hydrogen (H^+) ions near the dissolving urea granule which raises the surrounding pH. At high pH, the ammonium ions (NH_4^+) dissociate and are converted to ammonia (NH_3) which can be volatilized and lost to the atmosphere.

The losses of fertilizer N by NH_3 volatilization after application of urea in plantation forests can be rapid and significant (Nômmik 1973, Kissel et al. 2004, Cabrera et al. 2010, Zerpa and Fox 2011, Elliot and Fox 2014). Losses range from less than 10% (Boomsma and Pritchett 1979, Craig and Wollum 1982), 10% to 40% (Nômmik 1973, Zerpa and Fox 2011) to more than 50% (Kissel et al. 2004, Kissel et al. 2009, Elliot and Fox 2014). The losses due to NH_3 volatilization in pine plantation systems are similar to the NH_3 volatilization losses observed in agriculture, which can range from 25% to 47% when urea is applied to the soil surface (Scharf and Alley 1988).

The factors that influence NH_3 volatilization after urea fertilization in agricultural systems are well studied (Volk 1959, Black et al. 1987, Kissel et al. 1988) whereas less research has focused on forested systems (Volk 1970, Nômmik 1973, Kissel et al. 2004, Elliot and Fox 2014). Ammonia volatilization is affected by soil pH (Ernst and Massey 1960, Cabrera et al. 1991, Kissel et al. 2009), soil moisture (Clay et al. 1990, Kissel et al. 2004), mineral soil substrate (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011), relative humidity (Cabrera et al. 2005), soil temperature (Ernst and Massey 1960, Clay et al. 1990, Moyo et al. 1989), surficial wind speed (Watkins et al. 1972, Kissel et al. 2004), precipitation (Craig and Wollum 1982, Kissel et al. 2004) and air temperature (Gould et al. 1973, Craig and Wollum 1982, Koelliker and Kissel, 1988). The organic (O) horizon (forest floor) in forest soils can also have a significant effect on NH_3 volatilization (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011). Soils with a high H^+ buffering capacity (high organic matter, clay, silt) generally have lower NH_3 volatilization losses of fertilizer N compared to those soils with a lower buffering capacity (sand) (Fenn and Kissel 1976, Ferguson et al. 1984).

Urea applied under cooler, wetter conditions generally has low NH_3 volatilization losses (Ferguson and Kissel 1986, Moyo et al. 1989, Cabrera et al. 2010) due to rapid urea dissolution and movement into the soil (Black et al. 1987, Paramasivan and Alva 1997). Higher temperatures and relative humidity stimulate urease activity and increase NH_3 volatilization (Craig and Wollum 1982, Ferguson and Kissel 1986, Moyo et al. 1989). Elevated wind near the soil boundary layer (Kissel et al. 2004) can also exacerbate losses. Higher rates of NH_3 volatilization also occur with higher pH values (Koelliker and Kissel 1988). The ammonium ions may enter the soil if a precipitation event occurs soon after fertilization which decreases NH_3 volatilization losses (Kissel et al. 2004). The amount of urea N lost from the system due to NH_3 volatilization is difficult to accurately predict because the loss of fertilizer N is driven by the interaction among many of these factors. Large losses of fertilizer N through NH_3 volatilization have occurred even under low temperatures (Carmona and Byrnes 1990, Engel et al. 2011). Conversely, Kissel et al. (2004) observed low NH_3 volatilization during August when urea was applied on a day with significant precipitation but high NH_3 volatilization (45% to 58%) under simulated, minor precipitation events.

Enhanced efficiency fertilizers (EEFs) have been developed to minimize losses through NH_3 volatilization (Hauck 1985, Goertz 1993, Azeem et al. 2014). Enhanced efficiency fertilizers that reduce NH_3 volatilization can be divided into two broad categories (Azeem et al. 2014). In the first, a chemical additive, such as N-(n-butyl) thiophosphoric triamide (NBPT) impregnates the urea granule which reduces urease activity near the urea granule (Bremner and Douglas 1971, Bremner and Chai 1986, Antisari et al. 1996, Sanz-Cobena et al. 2008). Reducing urease activity allows the urea granule to dissociate and slowly move into the soil, thus reducing NH_3 volatilization losses (Bremner and Douglas 1971). A second EEF method is to coat urea granules with a physical barrier, as with a sulfur (S) or a polymer coating. This approach slows dissolution of the urea granule so that it is released to the environment in a more constant, gradual rate. This may reduce NH_3 volatilization losses and create release rates more synchronous with plant demand during the year (Shaviv 1996, Blazier et al. 2006).

The primary objective of this research was to determine the effectiveness of three enhanced efficiency fertilizers compared to urea in reducing fertilizer N volatilization losses in mid-rotation loblolly pine plantation systems in the South. We compared NH_3 volatilization losses following fertilization in two different seasons (spring, summer). Two statistical hypotheses were tested in this experiment:

Ho1: There are no differences in NH_3 volatilization between urea and enhanced efficiency fertilizers.

Ho2: There are no differences in NH_3 volatilization for treatments between seasonal applications (spring, summer).

2.2. Materials and Methods

2.2.1. Experimental Design

The experiment used a split plot complete block design to test differences in NH_3 volatilization losses. Four fertilizer treatments (main plots) were applied at two different seasons, spring versus summer (split plot) following fertilization in mid-rotation loblolly pine plantations. The fertilizer treatments were the main plots with a single replication of each treatment combination (fertilizer source and season of application) at each individual site. The six sites served as blocks and provided replication. The split plot was the application date (spring, summer).

2.2.2. Fertilizer Treatments

The four fertilizer treatments were: 1) urea; 2) urea impregnated with N-(n-Butyl) thiophosphoric triamide (NBPT); 3) urea impregnated with N-(n-Butyl) thiophosphoric triamide and coated with monoammonium phosphate and a proprietary binder (CUF); and 4) polymer coated urea (PCU). Urea (46-0-0) was the conventional N fertilizer used because of its common use in southern forests. The enhanced efficiency fertilizers (EEFs) tested in this study were developed to reduce NH_3 volatilization and release fertilizer N slowly to the environment. In the NBPT treatment, urea (46-0-0) granules were impregnated with N-(n-butyl) thiophosphoric triamide at a rate of 26.7% by weight to inhibit urease activity. In the CUF treatment (39-9-0), urea granules were impregnated with NBPT and coated with an aqueous binder containing a boron and copper sulfate solution to slow the release of N to the environment. A coating of monoammonium phosphate was then added to provide P. The PCU (44-0-0) treatment had a polymer coating covering urea granules with pore openings designed to slowly release N (~80%) over a 120 day period. The application rate for all treatments was equivalent to 224 kg N ha^{-1}. Because the CUF treatment contained P in the monoammonium phosphate coating, P was added to the other fertilizer treatment at the equivalent rate of 28 kg P ha^{-1} using triple superphosphate (TSP). The urea for all treatments was enriched with the stable isotope ^{15}N (~370‰, 0.5 AP) (Nadelhoffer and Frye 1994). All individual fertilizer treatments for both studies were applied by hand to individual microcosms.

2.2.3. Site Descriptions

Six sites were selected adjacent to plots in an existing network of forest thinning and fertilization studies in mid-rotation loblolly pine plantations across the South (Figure 2.1). Selected site characteristics are detailed in Tables 2.1 and 2.2 with selected soil chemical and physical data in Table 2.3. Daily mean weather data for study sites was obtained from the Oak Ridge National Laboratory (ORNL) DAYMET website (http://daymet.ornl.gov/) and nearest weather stations from the Weather Underground website (http://www.wunderground.com/).

2.2.4. Experimental Method

Ammonia volatilization losses were determined using an open chamber microcosm methodology detailed by Marshall and Debell (1980) and May and Carlyle (2005). Each microcosm was constructed from a white Schedule 40 polyvinyl chloride pipe (PVC) with an inner diameter of 15.24 cm (Figure 2.2). Each microcosm was inserted vertically into the soil through the entire organic (O) horizon (Oi + Oe + Oa) into the mineral soil to a depth of 30 cm which severed roots. A 2.5 cm portion of the microcosm remained above the O horizon. The O and mineral soil horizons inside the microcosm were minimally disturbed during insertion.

Immediately prior to each treatment application to individual microcosms, two fertilizer granules were randomly sampled and placed in labeled scintillation vials for ^{15}N analysis that was used in the overall mass balance calculation for each individual treatment determination of total ^{15}N recovery. The four fertilizer treatments were applied to the surface of the O horizon in each microcosm. The fertilizer N treatments were applied to randomly selected microcosms on two separate dates. All fertilizer applications in a specific season were made on a single day at individual sites. The spring application dates ranged from March 26 to April 8, 2011, and the summer application dates ranged from June 18 to June 30, 2011. At each site one microcosm for each fertilizer treatment was randomly selected and removed without disturbing the soil inside the microcosm on 1, 15 or 30 days after fertilizer application. An additional mineral soil sample was taken immediately below the microcosm at each sampling date to determine if fertilizer ^{15}N had leached below the microcosm. After removal, a plastic cover was secured to the top of the microcosm and plastic wrap to the bottom, and the intact microcosms were placed on ice in a cooler and transported to the laboratory for processing.

2.2.5. Laboratory Processing

All microcosms were stored in a walk-in freezer with a constant temperature of -20.0°C until processed. Upon removal from the freezer, microcosms were thawed and the soil was divided into four depth increments: O horizon (Oi + Oe + Oa) and the mineral soil increments of 0-10 cm, 10-20 cm, and 20-30 cm. Each depth increment from each microcosm was wet sieved, placed in a labeled double paper bag and dried in a forced air walk-in oven with a constant temperature of 60°C for 1 week. After drying, O horizon samples were sieved through a 6 mm sieve whereas mineral soil samples were sieved through a 2 mm sieve. All sieved O horizon and mineral sieved solid soil samples were ground to a fine powder in a ball mill (Retsch® Mixer Mill MM 200, Haan, Germany) for 2 minutes at 25 rps to ensure proper homogenization. Between 40 mg to 45 mg of the ball milled samples were weighed in a tin capsule on a Mettler-Toledo© MX5 microbalance (Mettler-Toledo, Inc., Columbus, OH, USA). The $^{15}N/^{14}N$ isotopic ratio and total N of individual samples were analyzed on a coupled elemental analysis-isotope ratio mass spectrometer (IsoPrime 100 EA-IRMS, Isoprime© Ltd., Manchester, UK). All instruments prior to isotopic-elemental analysis were cleaned with an ethanol solution and allowed to dry after each sample was processed to reduce potential contamination. The quantity of fertilizer N recovered in each depth increment was determined from the $^{15}N/^{14}N$ isotope ratio and total N using the method discussed by Nadelhoffer and Fry (1994). Fertilizer recovered in each depth increment in the microcosm was summed to determine the total recovery in the microcosm. The fertilizer N not recovered was attributed to NH_3 volatilization loss, and expressed as a percentage of the applied fertilizer N.

2.2.6. Weather Data

Daily weather data, including mean daily temperature and total daily precipitation during the first 15 days of the experiment was downloaded from the nearest weather station to each site through the Oak Ridge National Laboratory DAYMET website (http://daymet.ornl.gov/) and the Weather Underground (http://wunderground.com).

2.2.7. Calculations

The individual total fertilizer N recovery for each depth increment in each microcosm was determined as detailed below, and all depth increments for each individual microcosm was summed to obtain a total fertilizer recovery for each individual microcosm. The individual depth increment in each microcosm was calculated using individual soil depth mass, N concentration, and ^{15}N (‰) adapted from Powlson and Barraclough (1993), Nadelhoffer and Fry (1994) and Nadelhoffer et al. (1995). Equation [3.1] calculated the ratio of fertilizer ^{15}N to natural abundance ^{15}N:

Equation [3.1] was used to calculate the individual soil depth increment component N percent that originated from the fertilizer N:

[3.1] %N from fertilizer in individual soil depth increment =

$$((\delta^{15}N_{final} - \delta^{15}N_{initial}) / (\delta^{15}N_{labeled} - \delta^{15}N_{initial})) * (100)$$

Equations [3.2] to [3.5] determined the percent recovery of fertilizer N in individual soil depth increments:

[3.2] % ^{15}N from labeled treatment in individual soil depth increment =

$$((\delta^{15}N_{component} - \delta^{15}N_{pretreatment}) / (\delta^{15}N_{fertlizer} - \delta^{15}N_{pretreatment})) * (100)$$

[3.3] Total N (g) = (Individual soil depth increment component dry weight * %N) / (100)

[3.4] N originated from fertilizer (g) in individual soil depth increment component =

((Total N * %^{15}N originated from fertilizer) / (100))

[3.5] % N originated from fertilizer in individual soil depth increment =

[(N from fertilizer (g) in individual soil depth increment)/(^{15}N fertilizer (g))] * (100)

2.2.8. Statistical Analysis

Ammonia volatilization loss (%), expressed as a percentage of fertilizer N not recovered from an individual microcosm, was analyzed using a repeated measure analysis of variance (ANOVA) using PROC MIXED in SAS® 9.4 (SAS Institute, Cary, NC). Several alternative spatial covariate structures were tested, and the optimal structure was the unstructured covariate structure based on Akaike's Information Criteria (AIC). Percent data were arcsin transformed prior to analysis (Gomez and Gomez 1984). Ammonia volatilization loss (%) was the response variable for the model, day (1, 15, 30) was the repeated measure, fertilizer treatment (CUF, NBPT, PCU, Urea) and season (spring, summer) were fixed effects, and site (VA, ALN, AR, ALS, SC, NC) was a random effect. (Littell et al. 2006). Levels of significance were set at $\alpha = 0.05$ and the $P > |t|$ values for the treatment means for each day and season were tested using the SLICE function in PROC MIXED. The post-hoc analysis used was Tukey's HSD.

Pearson's correlations were conducted between treatments and weather variables to assess relationships with NH_3 volatilization loss. Maximum daily relative humidity and mean daily relative humidity were calculated from dewpoint and temperature data (Iduchov and Eskridge 1996), mean daily critical relative humidity (CRH) for urea dissolution was calculated using the procedure in Elliot and Fox (2014). The number of days when mean daily relative humidity was greater than the CRH for each sampling interval (1, 15, 30 days after fertilization) were determined. Pearson's correlation coefficients were calculated for mean NH_3 volatilization from urea and the selected weather data 1 day and 15 consecutive days after fertilization for spring, summer and spring + summer application seasons combined to assess relationships between NH_3 volatilization from urea and weather variables. Strong correlations were defined as $0.5 < |r|$, moderate correlations as $0.3 < |r| < 0.5$, and small correlations as $0.1 < |r| < 0.3$.

2.3. Results

2.3.1. Differences in NH_3 Volatilization Among Fertilizer Treatments – Spring

Mean NH_3 volatilization losses following urea application in the spring were greater than losses from the EEFs after 1, 15 and 30 days after fertilizer application (Table 2.6; Figure 2.4). There were no significant differences among the individual EEFs 1 and 15 days after fertilization, but mean NH_3 volatilization losses following CUF application were less than mean NH_3 volatilization losses from NBPT and PCU after 30 days. Mean NH_3 volatilization losses, expressed as a percent of the fertilizer N added, after 1 day were 3.9% for CUF, 9.6% for NBPT, 8.2% for PCU and 25.9% for urea. Fifteen days after fertilization, the mean NH_3 volatilization was 13.7% for CUF, 20.0% for NBPT, 15.3% for PCU, and 35.2% for urea. Thirty days after fertilization, the mean NH_3 volatilization was 15.2% for CUF, 23.5% for NBPT, 21.5% for PCU and 40.0% for urea.

2.3.2 Differences in NH_3 Volatilization Among Fertilizer Treatments – Summer

Mean NH_3 volatilization losses following urea fertilization in the summer were significantly greater after 1, 15 and 30 days then mean NH_3 volatilization losses from the EEFs (CUF, NBPT, PCU) except after 1 day, where there was no difference between CUF and urea (Table 2.6; Figure 2.4). There were no significant differences in mean NH_3 volatilization among the individual EEFs after 1 and 15 days, but after 30 days losses from PCU were less than CUF. Mean NH_3 volatilization losses, expressed as a percent of fertilizer N added, were 18.8% for CUF, 6.7% for NBPT, 11.0% for PCU and 28.5% for urea after 1 day. After 15 days, the mean NH_3 volatilization was 16.6% for CUF, 14.2% for NBPT, 11.4% for PCU, and 45.4% for urea.

After 30 days, the mean NH_3 volatilization was 23.3% for CUF, 21.4% for NBPT, 11.8% for PCU and 48.7% for urea.

2.3.3. NH_3 Volatilization Among Fertilizer Treatments – Spring + Summer

When the data was combined for the spring and summer application (spring + summer), mean NH_3 volatilization losses were significantly greater following fertilization with urea than the EEFs after 1, 15 and 30 days (Table 2.6; Figure 2.4). There were no significant differences among the individual EEFs 1 and 15 days after fertilization, but mean NH_3 volatilization losses were greater following fertilization with NBPT than PCU after 30 days. Mean NH_3 volatilization losses, expressed as a percent of fertilizer N added, were 11.3% for CUF, 8.7% for NBPT, 9.6% for PCU and 27.3% for urea after 1 day. The mean NH_3 volatilization was 15.2% for CUF, 17.1% for NBPT, 13.9% for PCU, and 40.3% for urea after 15 days. After 30 days, the mean NH_3 volatilization was 19.2% for CUF, 22.4% for NBPT, 16.7% for PCU and 44.4% for urea.

2.3.4 Correlation Between NH_3 Volatilization From Urea and Weather Data

Correlations between weather data and cumulative mean NH_3 volatilization loss, expressed as a percentage of N applied, following fertilization with urea for spring, summer and spring + summer data were generally poor (Table 2.7, *Appendix Figure A.2.1.*). After the spring fertilization, a strong negative correlation occurred with total daily precipitation (-0.512), moderate positive correlations with maximum daily temperature (0.312) and mean daily temperature (0.303), and a moderate negative correlation when the critical relative humidity exceeded the relative humidity (-0.303). After the summer fertilization, strong positive correlation occurred with mean daily relative humidity, and moderate positive correlations with maximum daily relative humidity (0.375) and minimum daily relative humidity (0.375). When the spring and summer data was combined, only a moderate negative correlation occurred with total daily precipitation (-0.311).

Ammonia volatilization losses from urea 15 days following the spring application were strongly correlated with maximum daily temperature (0.579), mean daily temperature (0.808) and cumulative days when daily mean relative humidity exceeded critical relative humidity (0.697). Moderate negative correlations occurred with maximum daily relative humidity (-0.406), and a positive correlation with mean daily relative humidity (0.470). Following summer application, no strong correlations were observed. Moderate positive correlations existed with maximum daily relative humidity (0.403) and mean daily relative humidity (0.322). When data for mean NH_3 volatilization losses after 15 days following spring and summer applications were combined, a strong positive correlation existed with the cumulative days when mean daily relative humidity was greater than the critical relative humidity (0.573), and moderate positive correlations existed with maximum daily temperature (0.406) and cumulative precipitation (0.386).

2.4. Discussion

Our study focused on assessing NH_3 volatilization following N fertilization with urea and enhanced efficiency N containing fertilizers in loblolly pine plantations in the southern United States. We correlated weather variables associated with NH_3 volatilization loss of the fertilizer N (Ernst and Massey 1960, Craig and Wollum 1982, Cabrera et al. 2005). By installing a study across the entire region where loblolly pine plantations are operationally grown, we hoped to improve the understanding of the magnitude of fertilizer N loss across the region rather than focus on a detailed study in a single location or smaller area as done by others (Volk 1970, Nômmik 1973, Kissel et al. 2004, Elliot and Fox 2014). This study tested two primary hypotheses: 1) whether differences existed in NH_3 volatilization between urea and enhanced efficiency fertilizers; and 2) whether differences existed in NH_3 volatilization among treatments between application seasons.

Because tree roots were severed when the microcosms were inserted into the soil, N uptake by the trees was eliminated. Therefore, we assume that the primary loss mechanism for the applied N from the open chamber microcosm was NH_3 volatilization to the atmosphere (Figure 2.2). However, the two additional potential loss pathways that could occur from the microcosms are leaching and denitrification. We tested for potential leaching loss of fertilizer N by sampling soil directly below each microcosm on each date and analyzing the sample for ^{15}N. If this loss pathway was important, samples immediately below the bottom of the microcosm (>30 cm soil increment) should have had elevated ^{15}N signatures. The ^{15}N signature (‰) for the >30 cm increment was similar to the natural abundance background ^{15}N values at each site (Table 2.4). We were unable to detect any of the fertilizer ^{15}N in the >30 cm depth increment after any sampling date (1, 15, 30) following the spring or summer fertilization with any fertilizer (CUF, NBPT, PCU, urea). This result indicates that leaching was an undetectable loss pathway in this study (Figure 2.3).

We did not directly measure denitrification losses in this study. Recent work examining denitrification on similar sites used in this study found that denitrification losses were relative low during spring and summer months (Shrestha et al. 2014). Denitrification occurs in anaerobic environments when nitrate functions as the terminal electron acceptor during the oxidation of soil organic matter. Microcosms in this study were all located in aerobic soil. Even at the sites with poorly drained soils, microcosms were located on top of the beds where soils were aerobic (Kelting et al. 1998, Shrestha et al. 2014). We compared N losses on well versus poorly drained soils in our study and found no differences in percent recovery of fertilizer N due to soil drainage class. Since losses were similar on both types of soil, denitrification was a minor loss mechanism in this study. Although a small amount of fertilizer N loss may have occurred through both denitrification and leaching, we concluded that NH_3 volatilization was the primary loss pathway for N in our microcosm systems.

Our first hypothesis that no differences in NH_3 volatilization existed between urea and enhanced efficiency fertilizers was rejected. Ammonia volatilization losses were significantly lower for enhanced efficiency fertilizers (CUF, NBPT, PCU) compared to urea following both a spring and summer application. Significantly less NH_3 volatilization occurred with all enhanced efficiency fertilizers, and when differences were present among individual enhanced efficiency fertilizers they were small. Yet with urea, NH_3 volatilization losses were large following fertilization in both spring and summer, ranging from 25% to 30% of fertilizer N lost after a single day. Losses following fertilization with EEFs were much less compared to urea. Ammonia volatilization losses continued to increase through time for all fertilizers. The incremental additional loss through 15 days and 30 days after fertilization were greater for urea than the EEFs. The result for these fertilizer treatments agree with those of other studies which have generally found the largest losses for NH_3 volatilization for urea occurring between 1 day and 7 days after application (Craig and Wollum 1982, Clay et al. 1990, Kissel et al. 2009, Elliot and Fox 2014).

The major loss pathway of the fertilizer N for this study was assumed to be through NH_3 volatilization. Urea hydrolysis, facilitated by urease, produces ammonium carbonate which dissociates into ammonium (NH_4^+) and bicarbonate (HCO_3^-). The bicarbonate consumes hydrogen (H^+) ions near the dissolving urea granule which raises the surrounding pH (Hauck and Stevenson 1965). In this situation the ammonium ions (NH_4^+) can be converted to ammonia (NH_3) which can be lost to the atmosphere. Higher rates of precipitation immediately following fertilization with urea, in excess of 1.27 cm, can dissolve the urea and move it into the soil, reducing NH_3 volatilization (Kissel et al. 2004). Sites for this study had minor (less than 12 mm) or no precipitation events within the first few days following fertilization. Minor precipitation events coinciding with warm temperatures following urea fertilization can exacerbate losses through the NH_3 volatilization pathway (Kissel et al. 2004) which occurred in our study and likely caused large losses for urea. Conversely, studies have shown large precipitation events after fertilization with urea can move fertilizer N into the soil profile rapidly (Kissel et al. 2004). Once the fertilizer N moves into the soil profile, it becomes more difficult for NH_4^+ conversion to NH_3 due to the soil buffering capacity (Ferguson et al. 1984) and mass flow of $NH_{3(g)}$ to the atmosphere. Two enhanced efficiency fertilizers we used in this study, CUF and NBPT, were developed to reduce urease activity for a short period (approximately 2 weeks), allowing time for precipitation to move the urea into the soil and thereby reducing losses from NH_3 volatilization. For the PCU treatment, water from the minor precipitation events may have been moving into the PCU granules, or N release may have just been initiated from the granule with incomplete dissolution of the urea granule occurring at the time of the analysis.

We rejected our second hypothesis that there were no differences in NH_3 volatilization between application seasons (spring versus summer). There was a significant interaction effect between treatment and season indicating larger NH_3 volatilization losses following the summer application (Table 2.5). Losses from urea were greater in the summer compared to spring. Sites generally experienced higher temperatures, more days when relative humidity exceeded critical relative humidity, and few large precipitation events. As previously discussed, these weather conditions increase the chances for NH_3 volatilization from urea because the rate of urease activity increases (Koelliker and Kissel 1988, Moyo et al. 1989, Kissel et al. 2004). When there is insufficient precipitation to move dissolved urea into the soil, NH_3 can be lost to the atmosphere, or losses may be exacerbated by minor precipitation events (Kissel et al. 2004).

Differences among the EEFs were small and somewhat variable. For day 1 in the summer, losses from the CUF treatment were greater compared to spring, but similar after 15 days and 30 days. For the NBPT and PCU treatments, losses were less each day for the summer compared to the spring. When reviewing site specific data, there were more frequent, smaller precipitation events during the summer compared to the spring at most sites. Although this can increase NH_3 volatilization from urea (Kissel et al. 2004), this may assist in gradually moving the EEFs into the soil due to their release mechanisms. The CUF and NBPT treatments generally have a two week effectiveness period for minimizing urease activity (Shaviv 1996). The PCU treatment is formulated to allow moisture into the polymer coating which permits gradual urea dissolution. As moisture enters the PCU granule, it expands and dissolved urea-N is forced through the polymer coating, gradually releasing fertilizer N to the environment. For the summer application period, more frequent, smaller precipitation events may have increased in the release and movement of N into the soil for PCU.

An additional question for this research was the effect of weather on NH_3 volatilization for urea. Similar to others (Cabrera et al. 2005, Cabrera et al. 2010, Elliot and Fox 2014) we found a strong correlation between NH_3 volatilization from urea and the number of days which relative humidity exceeded the calculated critical relative humidity during the first days following fertilization (Elliot and Fox 2014). When relative humidity exceeds the threshold of critical relative humidity, urea hydrolysis occurs and can lead to NH_3 volatilization (Cabrera et al. 2005).

The amount of urea N lost from the system due to NH_3 volatilization is difficult to accurately predict because the loss of fertilizer N is affected by many factors. High surficial wind speed (Watkins et al. 1972, Kissel et al. 2004), low precipitation (Craig and Wollum 1982, Kissel et al. 2004) and high air temperature (Gould et al. 1973, Craig and Wollum 1982, Koelliker and Kissel 1988) increase NH_3 volatilization. Urea applied under wetter, cooler periods tends to reduce NH_3 volatilization losses (Ferguson and Kissel 1986, Moyo et al. 1989, Cabrera et al. 2010) due to rapid urea dissolution (Black et al. 1987, Paramasivan and Alva 1997) which moves N into the soil compared to urea remaining on the soil surface. This is a primary reason why urea is applied during winter months in loblolly pine plantations in the South. However, large losses of fertilizer N through NH_3 volatilization have occurred even under low temperatures (Carmona and Byrnes 1990, Engel et al. 2011). High temperatures and high relative humidity can stimulate urease activity increasing NH_3 volatilization (Craig and Wollum 1982, Ferguson and Kissel 1986, Moyo et al. 1989). Conversely, Kissel et al. (2004) observed low NH_3 volatilization during August when urea was applied on the same day as a significant precipitation event, but high volatilization (45% to 58%) under simulated, minor precipitation events. These weather variables were also positively correlated with large NH_3 volatilization losses in our study.

Interactions of weather variables with edaphic factors may also increase NH_3 volatilization and can include increases with soil pH (Ernst and Massey 1960, Koelliker and Kissel 1988, Cabrera et al. 1991, Kissel et al. 2009), soil moisture (Clay et al. 1990, Kissel et al. 2004), type of mineral soil substrate (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox

2011), low soil buffering capacity as in sand or soils with low organic matter (Fenn and Kissel 1976, Ferguson et al. 1984), and soil temperature (Ernst and Massey 1960, Clay et al. 1990, Moyo et al. 1989, He et al 1999). Although similarities exist between agriculture and forest systems, important differences occur which may also affect NH_3 volatilization losses between the two systems. One important difference is the presence of an organic (O) horizon (forest floor) in forest soils which can have a significant effect on NH_3 volatilization (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011). Although we did not measure many of these factors, future research should continue to investigate their interactions to refine our understanding of NH_3 volatilization losses of urea from forest ecosystems.

Based on our findings, the primary fertilizer N loss pathway in this study was from NH_3 volatilization. In acidic, aerobic forest soils, denitrification is considered to be a minor loss pathway for N (Bowden et al. 1990) except in anaerobic soils. Although anaerobic conditions may have existed in the interbed areas of three of our sites, all the microcosms were installed in the bedded areas which were likely aerobic (Kelting et al. 1998). Interbed areas of a site can have extended periods of anaerobic conditions, and the pathway of fertilizer N loss may increasingly shift towards denitrification (Shrestha et al. 2014). Because fertilizers are generally applied through surface broadcast methods, and in essence 50% of the site may be comprised of interbeds, understanding loss mechanisms of fertilizer N in these areas is important. Additionally, the organic horizon is generally thicker in the interbed compared to the bed, and this potentially influences the amount of fertilizer N loss that can occur (Cabrera et al. 2005). Our analysis of fertilizer N loss based on the grouping of well drained (aerobic) compared to poorly drained soils did not find any differences, indicating soil drainage classification for the bedded areas of the sites were not a controlling variable for fertilizer N loss. If losses were different between these two soil groupings, losses of fertilizer N by denitrification in the poorly drained sites would need to be considered.

The remaining potential loss pathway from the microcosms was leaching. Measurements immediately adjacent and outside the bottom opening of the microcosm found no detectable increase in the ^{15}N values in the soil depth of >30 cm compared to natural abundance values of a control. From these findings, we conclude the majority of fertilizer N loss was by the NH_3 volatilization pathway. If other losses did occur from the system, they were below the detectable limits of our methods. We did not measure NH_3 volatilization from unfertilized control plots for a comparison with treatment plots due to traditionally low observed values in forest ecosystems by others (Marshall and DeBell 1980, Kissel and Cabrera 1988, Elliot and Fox 2014).

A primary benefit of this study is the results are drawn from an extensive geographic area, encompassing the entire region where loblolly pine plantations are operationally fertilized. Because this study does encompass such an extensive region, and hence large range of climatic and edaphic variables, a synthesis of significant factors helps to support the results specific to NH_3 volatilization after N fertilization of other studies in forest ecosystems that are an assessment of a single or smaller regional sites. Additionally, the open chamber system design of the microcosm does not have the potential limitations of closed chamber systems that can manipulate pressure, temperature or wind speed, although the results of this study still should be viewed as an index and not absolute values. This study provides continued evidence that although weather conditions can be important in determining NH_3 volatilization losses from urea, they do not translate to the same losses for enhanced efficiency fertilizers. These results provide evidence to forest managers that enhanced efficiency fertilizers can significantly reduce NH_3 volatilization losses of fertilizer N across the entire range of southern loblolly plantations, regardless of the season of application. The use of enhanced efficiency N containing fertilizers may translate to improved forest productivity and fertilizer N use efficiency in southern loblolly pine plantations in the future.

2.5. Conclusions

The primary results for this research were that significantly lower NH_3 volatilization losses occurred between all forms of N containing enhanced efficiency fertilizers (CUF, NBPT, PCU) over extensive ecophysiographic regions of the southern United States in mid-rotation loblolly pine stands compared to the conventional current standard of forest fertilization, urea. These results indicate that enhanced efficiency N containing fertilizers can reduce NH_3 volatilization losses over a significant range of weather and edaphic factors compared to urea. These findings may translate to improved fertilizer N use efficiency in southern loblolly pine plantation systems by retaining more fertilizer N in the system, with the possibility of reducing application rates of N in the future. These findings are important to the continued direction of improving productivity in southern loblolly pine plantations. Using enhanced efficiency fertilizers may also provide the ability for forest managers to apply fertilizer N in a more synchronous manner to plant demand under conditions where large NH_3 volatilization losses occur with urea, continuing to improve fertilizer N use efficiency in these systems. Viewed collectively, these results indicate that NH_3 volatilization can be decreased significantly using any of the enhanced efficiency fertilizers in this study (CUF, NBPT, PCU) when compared to urea under a diverse range of climatic and site variables for pine plantations in the southern United States. These results provide forest managers a level of certainty that if NH_3

volatilization is a concern at the site, enhanced efficiency N containing fertilizers may assist in retaining more fertilizer N in the system, translating to a potentially higher fertilizer N use efficiency over the rotation of the stand.

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Figures

Figure 2.1. *Location map for sites selected in the southern United States to evaluate ammonia volatilization following fertilization with urea and enhanced efficiency fertilizers enriched with* ^{15}*N after a spring and summer fertilizer application.*

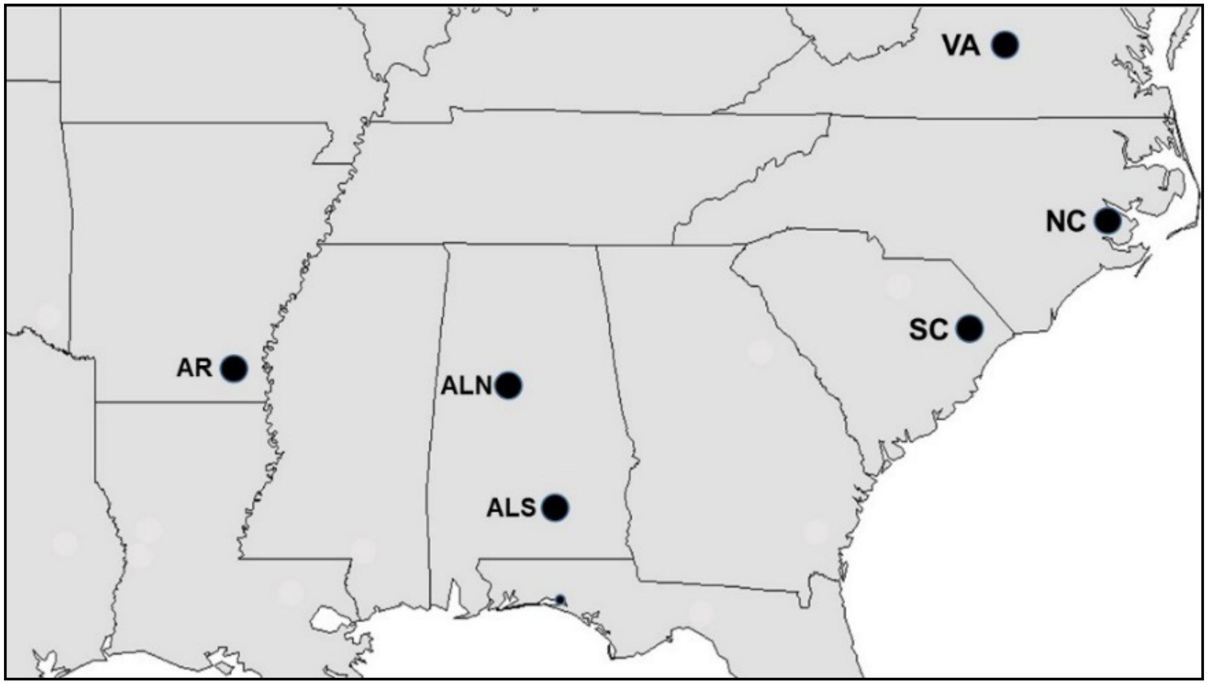


Figure 2.2. *Microcosm diagram for ammonia volatilization study in the southern United States selected to evaluate ammonia volatilization following fertilization with urea and enhanced efficiency fertilizers enriched with ^{15}N after a spring and summer application.*

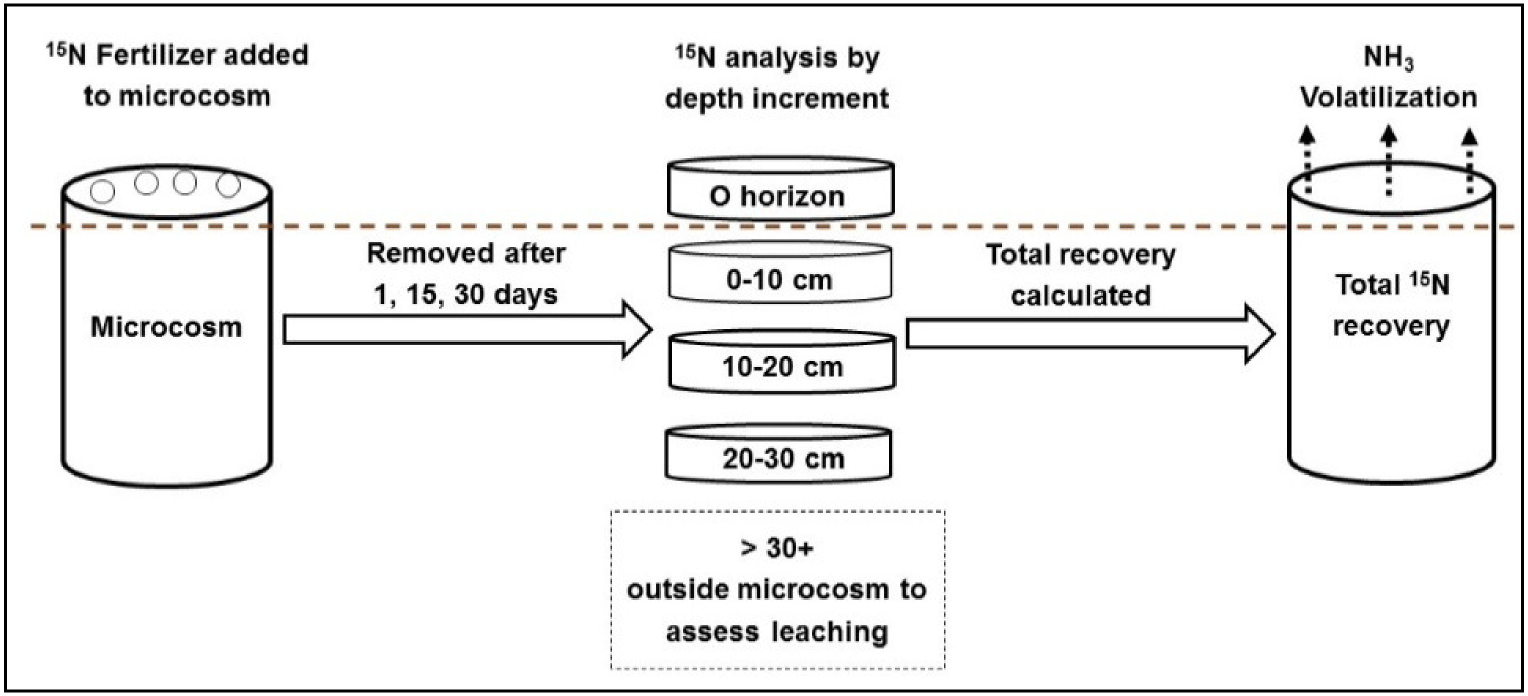


Figure 2.3. *The mean percent recovery of applied* ^{15}*N enriched fertilizers in microcosms, by soil depth increment, for spring and summer application after 30 days. The >30 cm increment is immediately below the microcosm. Error bars are the standard error of the mean.*

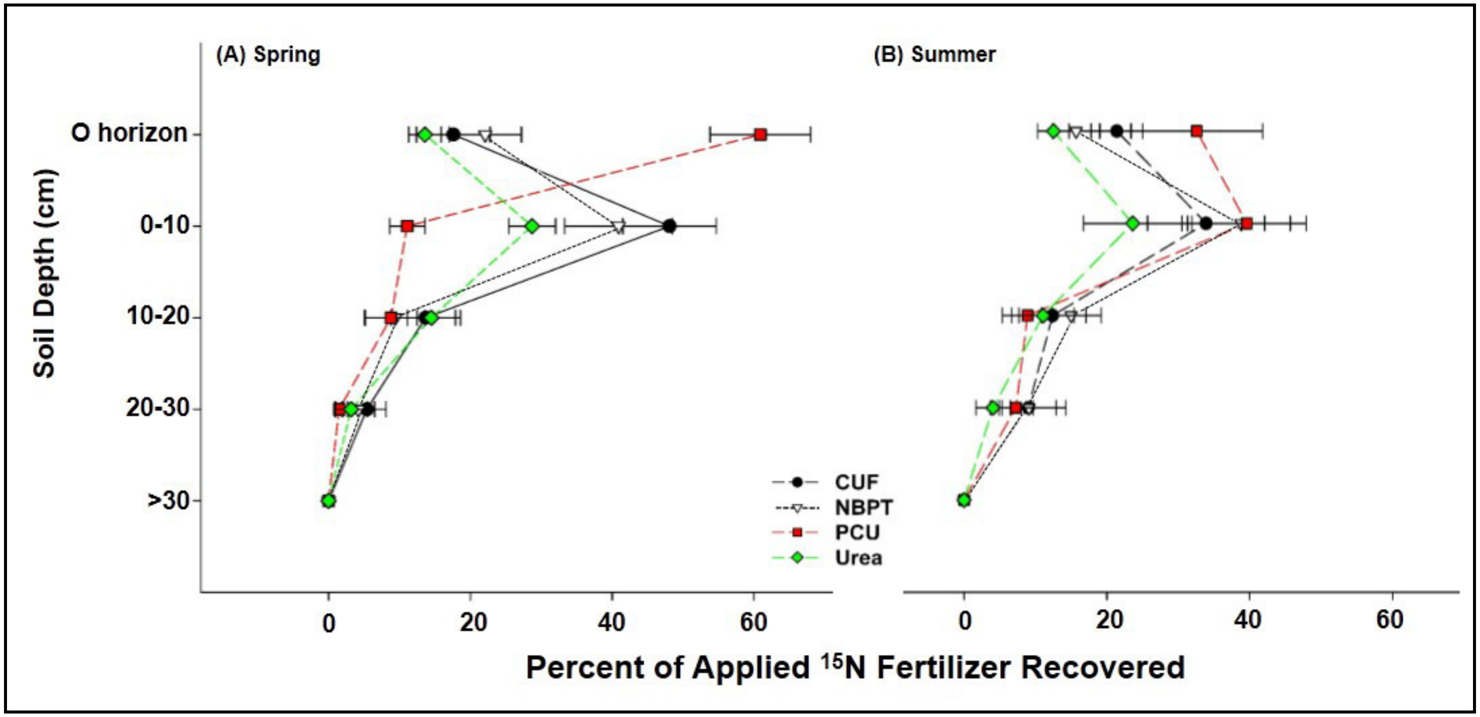
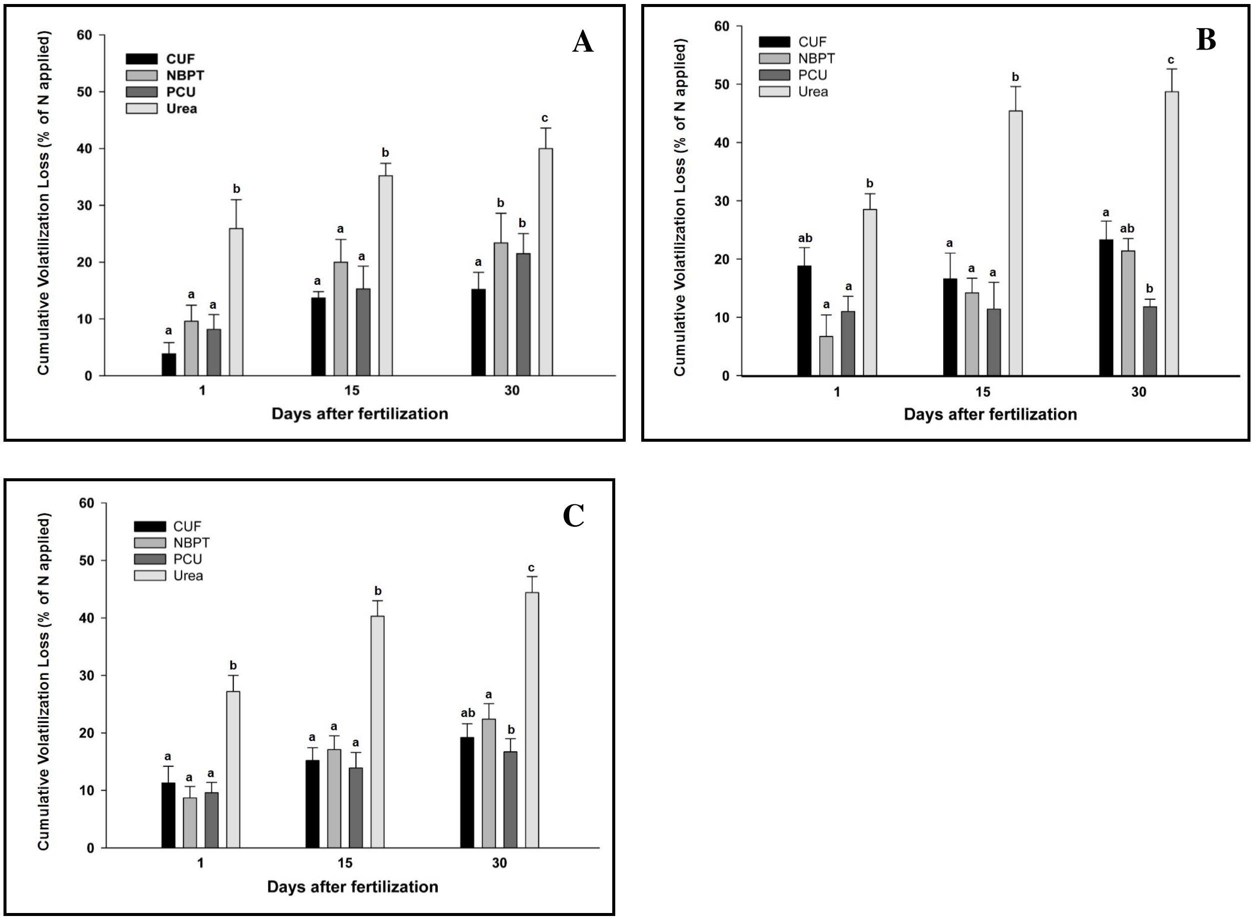


Figure 2.4. *Cumulative mean volatilization loss from microcosms, expressed as a percent loss of applied N fertilizer, for 6 mid-rotation loblolly pine plantations across the southern United States after a spring (A), summer (B), and spring + summer (C) application of* ^{15}*N enriched treatments (CUF, NBPT, PCU, urea). Different letters are significant differences at α = 0.05, and error bars are the standard error of the mean. The N application rate was 224 kg N ha*$^{-1}$.



Tables

Table 2.1. *Selected climatic and physical characteristics for sites in the southern United States selected to evaluate ammonia volatilization following fertilization with urea and enhanced efficiency fertilizers enriched with ^{15}N.*

Site	Latitude	Longitude	Altitude (m)	Mean Annual Precipitation (cm)	Mean Annual Temperature (°C)	Physiographic Region	Soil Taxonomic Class	Soil Series	Soil Drainage Class
VA	37.440087	78.662396	197	109	13	Southern Piedmont	fine, mixed, subactive, mesic Typic Hapludults	Littlejoe	well
SC	33.869400	79.289300	2	125	17	Atlantic Coast Flatwoods	fine, kaolinitic, thermic Umbric Paleaquults	Byars	very poorly
NC	35.317006	78.514167	0.5	121	16	Atlantic Coast Flatwoods	fine-silty, mixed, active, thermic Typic Albaquults	Leaf	poorly
AR	33.422310	91.732651	69	140	16	Western Gulf Coastal Plain	fine-silty, mixed, active, thermic, Typic Glossaqualfs	Calhoun	poorly
ALN	33.233371	87.232384	158	125	17	Appalachian Plateau	loamy-skeletal, mixed, subactive, thermic, shallow, Typic Dystrudepts	Montevallo	well
ALS	31.659464	86.272045	111	145	26	Southern Coastal Plain	fine, smectitic, thermic, Typic Hapludults	Arundel	well

Table 2.2. *Selected characteristics of loblolly pine stands in the southern United States selected to evaluate ammonia volatilization following fertilization with urea and enhanced efficiency nitrogen containing fertilizers.*

Site	**Age**	**Trees/Hectare**	**Height (m)**	**DBH (cm)**	**BA (m^2ha^{-1})**
VA	17	1100-1200	13.1	16.5	26.8
SC	16	500-600	16.0	25.7	29.7
NC	18	100-200	18.7	27.2	7.7
AR	13	300-400	15.3	20.8	22.4
ALN	19	300-400	18.8	24.2	25.2
ALS	19	500-600	18.5	21.1	20.4

Table 2.3. *Selected physical and chemical soil characteristics of loblolly pine stands in the southern United States selected to evaluate ammonia volatilization following fertilization with urea and enhanced efficiency nitrogen fertilizers enriched with* ^{15}N.

Site	**Depth**	**BD**	**CEC**	**pH**	**P**	**K**	**Ca**	**Mg**	**Zn**	**Mn**	**Cu**	**Fe**	**B**	**N**	**C**
				Water	**Mehlich I Extractable**									**Total**	
	cm	g cm^{-3}	cmol kg^{-1}		mg kg^{-1}									g kg^{-1}	
VA	Organic horizon													15.3	315.9
	0-10	1.24	6.40	4.2	3.0	39.3	68.3	14.7	0.5	7.1	0.4	62.7	0.1	1.7	24.9
	10-20	1.32	5.33	4.3	2.3	29.7	44.0	11.7	0.3	4.0	0.6	16.9	0.1	0.5	7.1
	20-30	1.45	5.80	4.4	2.0	39.0	46.0	12.7	0.5	2.4	0.3	29.8	0.1	0.4	8.4
SC	Organic horizon													7.1	252.6
	0-10	1.32	7.10	4.4	4.0	17.0	147.0	24.0	0.4	3.6	0.6	34.9	0.1	3.4	28.6
	10-20	1.35	7.80	4.5	6.0	18.0	217.0	30.0	0.7	2.2	0.5	34.7	0.1	1.2	27.6
	20-30	1.38	7.50	4.6	4.0	16.0	153.0	31.0	0.7	1.1	0.4	75.4	0.1	3.8	13.9
NC	Organic horizon													8.6	309.7
	0-10	1.28	9.5	3.8	8.0	22.0	113.0	22.0	0.6	1.5	0.3	100.3	0.1	1.0	43.5
	10-20	1.31	5.3	4.4	4.0	17.0	59.0	14.0	0.5	0.8	0.4	81.6	0.1	0.6	26.8
	20-30	1.48	4.2	4.4	2.0	16.0	36.0	10.0	0.3	0.3	0.2	74.2	0.1	0.5	15.5

Site	**Depth**	**BD**	**CEC**	**pH**	**P**	**K**	**Ca**	**Mg**	**Zn**	**Mn**	**Cu**	**Fe**	**B**	**N**	**C**
				Water				**Mehlich I Extractable**						**Total**	
	cm	g cm^{-3}	cmol kg^{-1}					mg kg^{-1}						g kg^{-1}	
AR	Organic horizon													11.0	279.1
	0-10	1.37	4.90	5.0	4.0	29.0	339.0	85.0	0.6	209.8	0.5	42.1	0.2	1.3	11.2
	10-20	1.41	4.60	5.0	3.0	25.0	265.5	73.0	0.5	192.8	0.5	35.2	0.2	0.7	6.7
	20-30	1.39	4.30	4.9	2.0	21.0	192.0	61.0	0.4	175.7	0.5	28.2	0.1	0.4	5.0
ALN	Organic horizon													9.9	224.7
	0-10	1.27	5.80	5.4	3.0	45.0	449.0	57.0	1.2	126.6	0.5	17.8	0.2	1.9	21.1
	10-20	1.31	4.6	5.4	2.0	44.0	421.0	71.0	0.8	81.7	0.6	13.6	0.2	0.6	6.9
	20-30	1.35	4.6	5.4	2.0	44.0	408.0	65.0	0.9	117.0	0.3	14.6	0.2	0.6	4.4
ALS	Organic horizon													8.7	279.3
	0-10	1.32	15.60	4.2	4.0	76.0	653.0	224.0	0.5	6.3	0.4	53.4	0.2	0.5	21.5
	10-20	1.25	15.35	4.3	3.0	74.0	580.0	207.0	0.5	3.5	0.5	40.7	0.2	0.6	14.4
	20-30	1.36	15.10	4.3	2.0	72.0	507.0	190.0	0.4	0.7	0.5	28.0	0.1	0.4	10.9

Table 2.4. *Mean ^{15}N (‰) values for depth increments of microcosms 30 days after a spring and summer application of ^{15}N enriched fertilizer treatments (CUF, NBPT, PCU, urea) at 6 mid-rotation loblolly pine plantations across the southern United States. The nitrogen application rate was 224 kg N ha^{-1}. Values in parentheses are the standard error of the mean.*

	Control	**CUF**	**NBPT**	**PCU**	**Urea**
A) Spring					
O horizon (Oi + Oe + Oa)	-1.98 (0.55)	56.80 (12.90)	62.90 (13.20)	125.30 (20.80)	36.47 (3.75)
0-10 cm	3.52 (0.43)	39.88 (7.53)	34.60 (12.00)	10.71 (1.66)	23.59 (2.91)
10-20 cm	4.99 (0.58)	16.53 (4.97)	12.78 (3.10)	10.13 (1.66)	17.10 (3.45)
20-30 cm	6.48 (1.01)	15.19 (2.59)	12.61 (2.24)	10.69 (2.00)	10.95 (1.56)
> 30 cm	7.05 (0.83)	6.35 (0.78)	6.16 (0.51)	6.54 (0.98)	6.34 (0.76)
B) Summer					
O horizon (Oi + Oe + Oa)	-2.33 (0.70)	80.14 (8.88)	90.90 (15.40)	111.00 (12.80)	58.36 (7.68)
0-10 cm	3.32 (0.92)	30.78 (4.22)	32.27 (2.71)	38.82 (6.76)	28.42 (7.87)
10-20 cm	3.78 (0.64)	21.34 (6.37)	22.71 (8.09)	17.33 (5.96)	20.85 (7.24)
20-30 cm	5.48 (0.95)	16.80 (7.31)	13.93 (3.08)	13.53 (2.37)	9.48 (2.41)
> 30 cm	6.59 (0.74)	6.03 (0.69)	6.15 (0.70)	5.72 (0.68)	5.96 (0.55)

Table 2.5. *Results (F-values and significance) of repeated measures ANOVA testing treatment (CUF, NBPT, PCU, urea), season (spring, summer), day (1, 15, 30), and the interactions of treatment*season, treatment*day, and treatment*season*day for NH_3 volatilization loss as a percentage of nitrogen fertilizer for sites at 6 mid-rotation loblolly pine plantations across the southern United States. The nitrogen application rate was 224 kg N ha^{-1}.*

Effect	**F value**	**Pr > F**
Treatment (CUF, NBPT, PCU, urea)	58.13	**<0.0001**
Season (spring, summer)	2.13	0.1519
Day (1, 15, 30)	20.73	**<0.0001**
Treatment * Season	5.11	**0.0044**
Treatment * Day	1.25	0.2962
Season * Day	0.74	0.4846
Treatment * Season * Day	1.31	0.2683

Table 2.6. *P > | t | values for treatment (CUF, NBPT, PCU, urea) contrasts for mean ammonia volatilization loss for days (1, 15, 30) after application of ^{15}N enriched nitrogen fertilizers during two seasons (spring, summer) and combined seasons (spring + summer) at 6 midrotation loblolly pine plantations in the southern United States. The nitrogen application rate was 224 kg N ha^{-1}.*

	Spring			**Summer**			**Overall (Spring+Summer)**		
	Number of days following fertilizer application								
Treatment Contrast	**1**	**15**	**30**	**1**	**15**	**30**	**1**	**15**	**30**
Urea vs. CUF	**<0.0001**	**<0.0001**	**<0.0001**	0.1119	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**
Urea vs. NBPT	**0.0006**	**0.0036**	**0.0033**	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**
Urea vs. PCU	**0.0002**	**0.0250**	**0.0002**	**0.0003**	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**
CUF vs. NBPT	0.2025	0.2062	**0.0287**	0.3358	0.9182	0.6285	0.4007	0.5760	0.1962
CUF vs. PCU	0.3380	0.7575	**0.0002**	0.0866	0.8454	**0.4061**	0.5809	0.7105	0.4314
NBPT vs. PCU	0.7461	0.3359	0.1811	0.4635	0.9979	0.7267	0.7711	0.3540	**0.0412**

Table 2.7. *Correlation matrix for selected weather variables 1 day following urea application for cumulative mean volatilization loss after application of ^{15}N enriched nitrogen fertilizers during two seasons (spring, summer) and combined seasons (spring + summer) at 6 mid-rotation loblolly pine plantations in the southern United States. Top values are the Pearson Correlation and number below in parentheses represents the associated p-value. The nitrogen application rate was 224 kg N ha^{-1}.*

	Cumulative Mean Volatilization Loss (% of N Applied)		
	Spring	**Summer**	**Spring+Summer**
Maximum Daily Temperature	0.312 (0.324)	-0.178 (0.736)	0.262 (0.216)
Minimum Daily Temperature	0.288 (0.365)	0.222 (0.673)	0.252 (0.234)
Mean Daily Temperature	0.303 (0.338)	0.036 (0.945)	0.261 (0.218)
Total Daily Precipitation	-0.512 (0.089)	N/A	-0.311 (0.139)
Maximum Daily Relative Humidity	-0.248 (0.756)	0.375 (0.464)	-0.245 (0.249)
Minimum Daily Relative Humidity	0.101 (0.756)	0.375 (0.464)	-0.062 (0.772)
Mean Daily Relative Humidity	-0.152 (0.636	0.554 (0.254)	-0.249 (0.240)
Percentage of Day where Critical Relative Humidity > Relative Humidity	-0.303 (0.338)	-0.036 (0.945)	-0.261 (0.218)

Chapter 3. Temporal Patterns of Foliar Nitrogen after Fertilization for Two Application Seasons in Mid-Rotation Loblolly Pine Plantations (*Pinus taeda* L) of the Southern United States

Abstract

The nitrogen (N) uptake of loblolly pines was determined following the surface application of urea and three enhanced efficiency N containing fertilizers (EEFs) at six thinned mid-rotation loblolly pine stands (*Pinus taeda* L.) across the southern United States. All fertilizer treatments were labeled with ^{15}N (~370 permil, 0.5 AP) and applied during two different seasons (spring, summer) in 2011 to individual 100 m^2 circular plots. Loblolly pine foliage was sampled every six weeks following N fertilization over a single growing season to assess foliar N uptake. Nitrogen fertilization increased the number of flushes and significantly increased the individual fascicle mean mass compared to unfertilized plots over the growing season. Nitrogen fertilization slightly increased foliar mean N concentrations (not significant), and significantly increased individual fascicle mean N content and foliar mean ^{15}N (‰) over the growing season after both a spring and summer fertilization. The proportion of individual fascicle mean fertilizer N also increased over the growing season, with individual treatment differences gradually dissipating towards the middle to end of the growing season. Both the cumulative N content and cumulative fertilizer N content gradually increased across the growing season after both a spring and summer fertilization, with more fertilizer N content in individual fascicles for the spring compared to a summer fertilization. This research indicates N fertilization improves growth directly through N addition, but may also make N existing naturally more readily available for plant uptake in these mid-rotation loblolly pine plantation systems.

3.1. Introduction

Soil nutrient deficiencies limiting the growth of forest plantations can be ameliorated through fertilization (Ballard 1984). The amelioration of nitrogen (N) deficiencies in forest plantations is particularly important because N is required in large quantities for foliar tissue development, photosynthesis, and numerous biochemical processes, all which are critical to growth (Miller 1981, Linder 1987). For example, a mid-rotation fertilization of loblolly pine (*Pinus taeda* L.) plantations in the southern United States with 224 kg ha^{-1} N and 28 kg ha^{-1} phosphorous (P) have a mean growth increase of 3 m^3 ha^{-1} over 8 years (Allen 1987, Fox et al. 2007a). Despite fertilization improving loblolly pine growth, field trials generally show low tree fertilizer N uptake (Mead and Pritchett 1975, Johnson and Todd 1988, Albaugh et al. 2004). In addition, some sites have no or a negative fertilizer response (Pritchett and Smith 1972, Martin et al. 1999, Amateis et al. 2000, Carlson et al. 2014) leading to a growth response ranging from 0 m^3 ha^{-1} yr^{-1} to 10 m^3 ha^{-1} yr^{-1} in loblolly pine fertilizer trials (Rojas 2005, Fox et al. 2007a). Conversely, laboratory studies for certain tree species show fertilizer N uptake ranging from 40% to 95% (Pang 1985, Salifu and Timmer 2003, Amponsah et al. 2004). Clearly an improved understanding of tree fertilizer N uptake and fertilizer N cycling in forest plantations is required to properly identify sites that are responsive to fertilization.

There are several explanations for differences in tree fertilizer N uptake among studies. Fertilizer N loss after application, primarily by ammonia (NH_3) volatilization (Raymond et al. 2016), denitrification (Shrestha et al. 2014) or leaching (Binkely et al. 1995, Aust and Blinn 2004) reduces total fertilizer N entering and remaining in the system, and may reduce fertilizer N availability for tree N uptake. Fertilizer N immobilization in ecosystem components with varying turnover rates can make fertilizer N unavailable to crop trees for extended periods during the rotation (Birk and Vitousek 1985, Albaugh et al. 2004, Meeks 2015). Sites unresponsive to N fertilization may simply have adequate plant available soil N due to high rates of soil organic matter mineralization (Fox et al. 2007a). Finally, soil nutrient deficiencies other than N may limit productivity (Vogel and Jokela 2011). These factors and their interactions may explain the observed variability in fertilizer trials in southern loblolly pine systems. Addressing these points through an improved understanding of the fertilizer N allocated to ecosystem components, pathways and rates of fertilizer N cycling, and fertilizer N uptake in forest plantations will improve fertilizer nitrogen use efficiency (FNUE) in these systems.

To improve fertilizer N uptake after fertilization, slow release (SRN), controlled release (CRN) and stabilized (SNF) N fertilizers were developed (Azeem et al. 2014, Fertilizer Institute 2015). The SRN, CRN and SNF are all enhanced efficiency fertilizers (EEFs) (AAPFCO 2015), defined as fertilizer products that reduce nutrient loss to the environment while increasing plant nutrient availability by slowing nutrient release or conversion of the nutrient to forms less susceptible to losses (The Fertilizer Institute 2015). The EEFs were developed to reduce the mechanisms of fertilizer N loss and provide a more flexible fertilizer N application and supply with seasonal plant demand (Hauck 1985, Goertz 1993, Azeem et al. 2014). The CRN is fertilizer N that is coated or encapsulated to provide specific rate, pattern and release durations (Chien et al. 2009). Fertilizer N in SRN is gradually released by microbial decomposition (Trenkel 2010). Fertilizer N in SNF is treated with inhibitors to avoid rapid N transformation to less environmentally stable forms (Shaviv 1996). Effective fertilizer N containing EEFs should have: 1) improved FNUE; 2) less N leaching; 3) low toxicity; 4) longer N supply; 5) reduced NH_3 volatilization or nitrification; and 6) lower application cost (Allen 1984, Hauck 1985). Although EEFs were developed to improve FNUE in agroecosystems, similar FNUE issues exist for pine plantations. Implementing EEF technology for fertilizer N application in southern pine plantations may improve FNUE and continue to increase the productivity of these systems. An increase in ecosystem fertilizer N retention may translate to an increase in N uptake over the growing season for loblolly pine trees.

Numerous methods have been developed to assess loblolly pine nutrient status and evaluate potential response to fertilization (Bowen and Nambier 1984, Carter 1992). The simplest method is visual observation (Stone 1968), but is limited because obvious symptoms (chlorosis) can be due to multi-nutrient deficiencies (Vogel or Jokela 2011) or pathogens (USDA 1989). Foliar analysis, including nutrient concentrations, critical nutrient levels (Tisdale et al. 1985, Havlin et al. 2014), nutrient ratios in Diagnosis of Recommendation Integrated System (DRIS) (Hockman and Allen 1990, Sypert 2005), and graphical analysis (Haase and Rose 1985) improve on visual assessment, but may be of limited value if sampling season and edaphic factors are not integrated (Carter 1992). Soil sampling and nutrient addition experiments also provide insight but may require large sample sizes to address soil heterogeneity (Binkely and Hart 1989). Integrated soils research has led to the development of soil groupings based on similar parent materials within physiographic regions that correlate with specific nutrient deficiencies, like the Cooperative Research in Forest Fertilization (CRIFF) soil groupings in the southern United States (Jokela and Long 2000), or groupings in the Pacific Northwest (Littke et al. 2014). Field or greenhouse bioassays using seedlings can also be valuable, but results may be confounded by artificial laboratory conditions and may be difficult to extrapolate to the field (Addoms 1937, Hobbs 1947, Morrison 1974, Mead and Pritchett 1975, Birk and Vitousek 1986). The most reliable, but expensive, assessment of nutrient deficiencies and fertilizer N uptake are fertilizer field trials (Miller 1981, Carter 1992, Albaugh et al. 2004, Fox et al. 2007a). Stable isotopes, as with ^{15}N, can be used to assess nutrient deficiencies and quantify fertilizer N uptake (Hauck 1968). Fertilizers enriched with stable isotopes (^{15}N) can trace the ultimate fate of applied N (Walker 1958, Hauck 1968, Powlson and Barraclough 1993) and be used to integrate ecosystem processes (Robinson 2001, Dawson et al. 2002, Templar et al. 2012). Numerous ^{15}N tracer studies were initially conducted in agroecosystems (Hauck and Bystrom 1971), but ^{15}N tracer studies have also improved the understanding of N dynamics in natural (Tietema et al. 1998, Currie and Nadelhoffer 1999, Dinkelmeyer et al. 2003, Nadelhoffer et al. 2004, Templar et al. 2012) and plantation (Walker 1958, Hauck 1968, Mead and Pritchett 1975, Melin et al. 1983, Chang et al. 1996, Bubb et al. 1999, Mead et al. 2008, Werner 2013) forests. Often several methods need to be combined at sites to improve the understanding of nutrient status and fertilizer responsiveness (Miller 1981, Birk and Vitousek 1985, Sypert 2005).

All fertilizer N in this study, both urea and EEFs, were enriched with ^{15}N (~370‰, 0.5 AP) to improve the understanding of N uptake of loblolly pines in the southern United States over the course of a single growing season. The statistical hypotheses for this research are:

Ho1: There are no differences in foliar growth among treatments.

Ho2: There are no differences in foliar N uptake among treatments.

Ho3: There are no differences in foliar fertilizer N uptake among treatments.

3.2. Methods and Materials

3.2.1. Experimental Design and Site Description

This experiment used a split plot complete block design to test temporal differences in fertilizer N uptake of loblolly pine. Five fertilizer treatments (main plots) were applied at two different seasons, spring versus summer (split plot) in mid-rotation loblolly pine plantations. The fertilizer treatments were the main plots with a single replication of each treatment combination (fertilizer source and season of application) at each individual site. The six sites served as blocks and provided replication. The split plot was the application date (spring, summer). The six sites were selected from an existing network of forest thinning and fertilization studies in mid-rotation loblolly pine plantations across the South (Figure 3.1). Selected site characteristics are detailed in Table 3.1 and Table 3.2.

Twelve 100 m^2 circular plots were installed at the six sites in early March 2011. At each plot center were two co-dominant loblolly pine trees with similar height (HT) and diameter (DBH). Six plots were used for the spring treatments and the other six plots were used for the summer treatment. Prior to treatments, the DBH and HT were measured for all trees within plots after installation but prior to treatment application.

3.2.2. Fertilizer Treatments

Five fertilizer treatments were applied at the two application times: 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 a 5) control treatment where no fertilizer N was added. Urea (46-0-0) was used because it is the most commonly applied fertilizer N source used in southern forests (Fox et al. 2007a). The enhanced efficiency fertilizers (EEFs) tested in this study were developed to reduce NH_3 volatilization and release N gradually to the environment. For the NBPT treatment, urea (46-0-0) granules were impregnated with N-(N-butyl) thiophosphoric triamide at 26.7% by weight to inhibit urease activity. For the CUF treatment (39-9-0), urea granules were impregnated with NBPT and then coated with an aqueous boron and copper sulfate binder solution to slow N release to the environment. A monoammonium phosphate coating was then added for P. The PCU (44-0-0) treatment had polymer coatings covering urea granules with pores in the coating to slowly release N (~80%) over 120 days. The application rate for all treatments was 224 kg N ha^{-1}. Because CUF contained P, P was added to the other fertilizer treatment at the equivalent rate of 28 kg P ha^{-1} using triple superphosphate (TSP). The urea used as the base for all treatments was enriched with the stable isotope ^{15}N (~370‰, 0.5 AP) (Nadelhoffer and Frye 1994). All fertilizer treatments were broadcast applied by hand to the forest floor in individual 100m^2 circular plots described above. Individual fertilizer N treatments were applied to the individual 100m^2 circular plots on a single day for each application season at each individual site.

3.2.3. Experimental Method- Field Sampling

Foliage was sampled from the upper 1/3rd of the canopy from both central trees immediately prior to individual fertilizer N treatment application and represented “week 0”. The spring application date was from March 26 to April 8 2011, and the summer application was from June 18 to June 30 2011. Sampling of foliage from the upper 1/3rd of the canopy from both central trees for all individual plots occurred every 6 weeks after the fertilizer treatments were applied until the dormant season. There were 6 sampling periods (week 0, 6, 12, 18, 24, 30) for spring fertilization and 4 sampling periods (week 0, 6, 12, 18) for summer fertilization at each site. Foliage was separated by each successive individual vegetative development over the growing season, termed flush, for each sampling period (Figure 3.2).

3.2.4. Experimental Method - Laboratory Processing

The foliage samples were oven dried in a forced air oven with a constant temperature of 60°C for 7 days. After oven drying, 25 intact and undamaged foliar fascicles were weighed. After weighing, the 25 fascicles from the sample were ground to a fine powder in a ball mill (Retsch® Mixer Mill MM 200, Haan, Germany) for 1 minute at 25 revolutions per second (rps). The ball mill was cleaned with an ethanol solution and allowed to dry to reduce contamination after the processing of each sample. Two milligrams (±0.20) of ground foliage were weighted on a Mettler-Toledo© MX5 microbalance (Mettler-Toledo, Inc., Columbus, OH, USA) and placed in separate tin capsules. Samples were analyzed to determine the

$^{15}N/^{14}N$ isotope ratio and total N of the sample on a coupled elemental analysis-isotope ratio mass spectrometer (IsoPrime 100 EA-IRMS, Isoprime© Ltd., Manchester, UK) at the Forest Soils and Plant Nutrition Laboratory at Virginia Polytechnic Institute and State University.

3.2.5. Calculations

Individual fascicle N content was calculated by multiplying individual fascicle mass (g) by N concentration (Equation 3.3). Individual fascicle fertilizer N content (Equation 3.1 to Equations 3.5) was calculated using individual fascicle mass, N concentration, and ^{15}N (*‰*) (Powlson and Barraclough 1993, Nadelhoffer and Fry 1994, Nadelhoffer et al. 1995). The uptake of N from sources existing in the system not originating from the fertilizer was calculated in Equation 3.6.

[3.1] %Fertilizer N in individual fascicles: $((\delta^{15}N_{final}-\delta^{15}N_{initial})/(\delta^{15}N_{labeled}-\delta^{15}N_{initial}))*(100)$

[3.2] $\%^{15}N$ in individual fascicles from labeled treatment: $((\delta^{15}N_{component}-\delta^{15}N_{pretreatment})$ / $(\delta^{15}N_{fertlizer}- \delta^{15}N_{pretreatment}))$ * (100)

[3.3] Total N (g) = (Individual fascicle dry weight * %N) / (100)

[3.4] Fertilizer N (g) in individual fascicle = (Total N * $\%^{15}N$ from fertilizer) / (100))

[3.5] %N originated from fertilizer in individual fascicle =

[(N originated from fertilizer (g) in individual fascicle) / (^{15}N fertilizer (g))] * (100)

[3.6] %N originated from native N sources in individual fascicle =

(Total N in individual fascicle) – (%N originated from fertilizer in individual fascicle)

3.2.6. Statistical Analysis

Individual fascicle mean mass, foliar N mean concentration, individual fascicle mean N content, mean foliar ^{15}N, and individual fascicle mean fertilizer N content was analyzed using a repeated measure analysis of variance (ANOVA) using PROC MIXED in SAS® 9.4 (SAS

Institute, Cary, NC). Several alternative spatial covariate structures were tested, and the optimal structure was the unstructured (UN) covariate structure based on Akaike's Information Criteria (AIC). Individual fascicle mean mass, mean foliar N concentration, individual fascicle mean N content, mean foliar ^{15}N, and individual fascicle mean fertilizer N content were the response variables for the model, sampling period (weeks 0, 6, 12, 18, 24, 30) and flush (0, 1, 2, 3) were repeated measures, fertilizer treatment (CUF, NBPT, PCU, urea, control) and season (spring, summer) were fixed effects, and site (VA, ALN, AR, ALS, SC, NC) was a random effect. (Littell et al. 2006). Levels of significance were set at $\alpha = 0.05$ and the $P > |t|$ values for the treatment means were tested using the SLICE function in PROC MIXED. The post-hoc analysis used was Tukey's HSD.

3.3. Results

3.3.1. Flush Development

As the growing season progressed, a larger number of flushes occurred in fertilized plots compared to control plots after both the spring and summer fertilization (Table 3.3).

Twelve weeks after the spring fertilization, all fertilized treatments had developed a 2nd flush (flush 2) at all 6 study sites while the control treatment had a single site with a flush 2. At the end of the growing season, 30 weeks after the spring fertilizer application, all fertilized plots had developed a 3rd flush (flush 3) at all 6 study sites while the control treatment had a single study site with a flush 3.

A similar trend occurred following the summer fertilizer application (Table 3.3.) At the end of the growing season, 18 weeks after the summer fertilizer application, fertilizer treatments at three of the study sites had produced a flush 3 but only one site had produced a flush 3 for the control treatment.

3.3.2. Individual Fascicle Mass

Eighteen weeks after a spring fertilization and 6 weeks after a summer fertilization, most individual fertilizer treatments had a greater mean individual fascicle mass (g) compared to the control for flush 1 and flush 2 (Table 3.4). Differences did not exist for the mean individual fascicle mass for flush 0 or flush 3 after either a spring or summer fertilization.

After a spring fertilization, the mean individual fascicle mass (g) for flush 0 and flush 1 in week 6 and week 12 for the control ranged from 0.14 g to 0.19 g, compared to the range of individual fertilizer treatments which ranged from 0.14 g to 0.21 g (ns). The mean individual fascicle mass for flush 1 in week 18 for all fertilizer treatments was 0.24 g compared to the 0.17 g for the control, and in flush 2 both CUF and urea (0.22 g) were greater than the control (0.17 g). By week 24 the mean individual fascicle mass for flush 1 was greater for both CUF (0.25 g) and PCU (0.23 g) compared to the control (0.18 g) while in flush 2 both CUF (0.21 g) and NBPT (0.21 g) were greater than the control (0.15 g). At the end of the growing season the mean individual fascicle mass for flush 1 for CUF (0.23 g), NBPT (0.25 g), PCU (0.24 g), and urea (0.24 g) was greater than the control (0.17 g), and in flush 2 CUF (0.25 g), NBPT (0.22 g) and urea (0.23 g) were greater than the control (0.17 g) while CUF was greater than PCU (0.19 g).

For the summer fertilization, mean individual fascicle mass (g) for flush 0 for all sampling periods ranged from 0.18 g to 0.20 g for the control and 0.17 g to 0.24 g for all individual fertilizer treatments (ns) (Table 3.4). The mean individual fascicle mass in week 6 for flush 1 and flush 2 was greater for NBPT (0.25 g, 0.23 g respectively), PCU (0.23 g, 0.20 g respectively), urea (0.24 g, 0.20 g respectively), and for CUF (0.22 g) only in flush 2, compared to the control (0.20 g, 0.16 g respectively). In week 12, the mean individual fascicle mass was greater for flush 1 and flush 2 for NBPT (0.23 g, 0.23 g respectively) and urea (0.24 g, 0.23 g respectively) compared to the control (0.18 g, 0.17 g respectively). By the end of the growing season, mean individual fascicle mass for flush 1 was greater for CUF (0.24 g), PCU (0.24 g) and urea (0.26 g) compared to the control (0.20 g), and both NBPT (0.24 g) and urea (0.25 g) were greater than the control (0.18 g) in flush 2.

3.3.3. Foliar N Concentration

The mean foliar N concentration (g kg^{-1}) increased slightly following both a spring and summer fertilization for all individual fertilizer treatments compared to the control, although most differences were not significant (ns) (Table 3.5; *In Appendix: Figure A.3.1., Table A.3.1, Table A.3.3, Table A.3.5., Table A.3.8.*).

Eighteen weeks after the spring fertilization, there was a significant increase in the mean foliar N concentration of flush 2 between NBPT (16.0 g kg^{-1}) and the control (11.4 g kg^{-1}). By week 30, the mean foliar N concentration of both NBPT (13.0 g kg^{-1}) and urea (12.6 g kg^{-1}) were greater than the control (8.6 g kg^{-1}) in flush 0.

Twelve weeks after summer fertilizer application, urea (14.2 g kg^{-1}) was greater than the control (10.4 g kg^{-1}) in flush 2. All other treatment differences for mean foliar N concentrations for individual flushes during each sampling period were not significant (ns).

3.3.4. Individual Fascicle N Content

The mean individual fascicle N content (mg N $fascicle^{-1}$) was greater for most individual treatments compared to the control 18 weeks after a spring fertilization and 6 weeks after the summer fertilization (Table 3.6; *In Appendix: Table A.3.6., Table A.3.9.*).

After the spring fertilization, the mean individual fascicle N content (mg) for all flushes in week 6 and week 12 ranged from 0.67 mg to 1.95 mg for the control compared to 1.43 mg to 2.38 mg for all individual fertilizer treatments (ns) (Table 3.6). There were no differences in mean individual fascicle N content in flush 0 among treatments for any sampling period except in week 30 where NBPT (2.72 mg) was greater than the control (1.74 mg). By week 18, differences occurred between individual fertilizer treatments and the control for the mean individual fascicle N content, where CUF (3.35 mg), NBPT (3.57 mg), PCU (3.43 mg) and urea (3.44 mg) were greater than the control (1.93 mg) in flush 1, and CUF (2.84 mg), NBPT (3.29 mg) and urea (3.08 mg) were greater than the control (1.82 mg) in flush 2. In week 24, all individual fertilizer treatments were greater in flush 1 (range: 2.86 mg to 3.45 mg) and in flush 2 (range: 2.43 mg to 3.00 mg) than the control (2.02 mg, 1.59 mg respectively). By week 30, all individual fertilizer treatments were greater than the control for both flush 1 and flush 2, but NBPT (3.62 mg) was greater than CUF (2.72 mg) in flush 1 and CUF (3.51 mg) was greater than PCU (2.75 mg) in flush 2.

For the summer fertilization, mean individual fascicle N content (mg) for flush 0 in week 6 and week 12 was greater for NBPT (2.58 mg, 2.64 respectively) compared to the control (1.99 mg, 1.98 mg respectively) (Table 3.6). Six weeks after the summer fertilization, all fertilizer treatments had a greater mean individual fascicle N content in both flush 1 (range: 2.94 mg to 3.45 mg) and flush 2 (range: 2.39 mg to 3.11 mg) than the control (2.25 mg, 1.78 mg respectively), and both CUF (2.98 mg) and NBPT (3.11 mg) were greater than PCU (2.39 mg) in flush 2. By week 12, all fertilizer treatments were greater in flush 1 (range: 2.68 mg to 3.26 mg) and flush 2 (range: 2.71 mg to 3.23 mg) than the control (1.77 mg, 1.18 mg respectively) with no differences among individual treatments. At the end of the growing season, CUF (3.19 mg) and urea (3.46 mg) were greater than the control (2.15 mg) in flush 1, all fertilizer treatments were greater in flush 2 (range: 2.46 mg to 3.46 mg) than the control (2.11 mg), and both PCU (2.78 mg) and urea (3.46 mg) greater than CUF (2.46 mg) in flush 2.

3.3.5. Foliar ^{15}N

The mean foliar ^{15}N (‰) was different for most fertilizer treatments for flush 0 and flush 1, and all fertilizer treatments for flush 2, compared to the control for all sampling periods after a spring fertilization (Table 3.7). After the summer fertilization, all individual fertilizer treatments were different compared to the control for all flushes during each sampling period beginning 12 weeks after a summer fertilization (Table 3.7; *In Appendix Figure A.3.2., Table A.3.2., Table A.3.4., Table A.3.5., Table A.3.8.*).

Across the entire growing season after a spring fertilization, the control mean foliar ^{15}N ranged from -3.43‰ to -1.56‰ for all flushes (Table 3.7). There were differences in flush 0 between the control and CUF (58.8‰) in week 12, in week 18 and week 24 between the control (-3.43‰, -3.38‰ respectively) and CUF (69.9‰, 56.7‰ respectively), NBPT (57.7‰. 56.3‰ respectively) and urea (60.4‰, 65.9‰ respectively), and the control (-2.50‰) and all individual fertilizer treatments (range: 53.5‰ to 80.1‰) by week 30. In week 6 and week 12, only PCU (17.8‰, 40.9‰ respectively) was not different from the control in flush 1. After 18 weeks, mean foliar ^{15}N (‰) of all individual fertilizer treatments for flushes 1, 2, and 3 ranged 73.8‰ to 134.2‰.

Six weeks after the summer fertilization there were differences in the mean foliar ^{15}N (‰) between the control (-2.90‰ to -2.10‰) and all fertilizer treatments (range: 52.1‰ to 72.8‰) except in flush 1 and flush 2 for PCU (22.9‰, 28.6‰ respectively). Twelve weeks after the summer fertilization, there were differences between the control (range: -3.04‰ to -2.55‰) and all individual fertilizer treatments (range: 40.5‰ to 102.6‰).

3.3.6. Individual Fascicle Fertilizer N Content

The mean individual fascicle fertilizer N content (mg N fascicle^{-1}) generally increased in all flushes across the entire growing season for all individual fertilizer treatments after both the spring and summer fertilization (Table 3.8; *In Appendix Table A.3.6., Table A.3.9.*). Because no fertilizer was applied to the control treatment, the value for all flushes for all sampling periods was 0.00 mg of fertilizer N.

Six weeks after the spring fertilization, the mean individual fascicle fertilizer N content of flush 0 for all treatments ranged from 0.00 mg to 0.19 mg (Table 3.8). In flush 1 of week 6, CUF (0.29 mg), NBPT (0.57 mg) and urea (0.33 mg) were greater than both the control and PCU (0.07 mg), and NBPT was greater than both CUF and urea. In week 12, CUF (0.25 mg) was greater than PCU (0.11 mg) in flush 0, while both NBPT (0.43 mg) and urea (0.39 mg) were greater than PCU (0.11 mg) in flush 1. In week 18 no differences occurred among individual fertilizer treatments for any flush, and mean individual fascicle fertilizer N content ranged from 0.25 mg to 0.90 mg. By week 24, the mean individual fascicle fertilizer N content for NBPT (0.87 mg) was greater than PCU (0.49 mg) in flush 2. At the end of the growing season NBPT (0.84 mg) was greater than PCU (0.54 mg) in flush 1, with no other differences among individual fertilizer treatments for mean individual fascicle fertilizer N content.

After the summer fertilization, the mean individual fascicle fertilizer N content for individual fertilizer treatments increased for all flushes for all sampling periods until the end of the growing season (Table 3.8). In week 6 for flush 2, CUF, NBPT and urea (range: 0.44 mg to 0.50 mg) were greater than PCU (0.16 mg). In week 12, the mean individual fascicle fertilizer N content increased for all treatments and ranged from 0.21 mg to 0.78 mg in flushes 0, 1, and 2. Both NBPT (0.78 mg) and urea (0.65 mg) also had greater mean individual fascicle fertilizer N content than both CUF (0.45 mg) and PCU (0.43 mg) in flush 2 of week 12. By week 30, the mean individual fascicle fertilizer N content was greater for urea (0.65 mg) than CUF (0.32 mg), NBPT (0.35 mg) and PCU (0.19 mg) in flush 0. The mean individual fascicle fertilizer N content for both urea (0.57 mg) and CUF (0.54 mg) were greater than PCU (0.33) in flush 1, and in flush 2 CUF (0.58 mg), NBPT (0.74 mg) and urea (0.70 mg) were all greater than PCU (0.40 mg).

3.3.7. Cumulative Individual Fascicle N Content

The cumulative individual fascicle N content (N mg fascicle^{-1}) increased for all treatments across the growing season, with differences between all individual fertilizer treatments and the control occurring by week 18 after the spring fertilization and week 6 after a summer fertilization (Figure 3.3; *In Appendix Table A.3.6., Table A.3.9.*).

After the spring fertilization, the cumulative individual fascicle foliar N content was similar in week 6 and week 12 between the control (3.34 mg, 4.11 mg respectively) and all individual fertilizer treatments (range: 4.17 mg to 4.88 mg, 4.11 mg to 5.07 mg respectively) (Figure 3.3). By week 18 the cumulative individual fascicle foliar N content for all individual fertilizer treatments (range: 8.04 mg to 9.33 mg) was greater than the control (5.77 mg). Differences for the cumulative individual fascicle foliar N content continued through the remainder of the growing season with individual fertilizer treatments (range: 10.50 mg to 12.48 mg) consistently greater than the control (range: 7.47 mg to 7.54 mg).

For the summer fertilization, with the cumulative individual fascicle foliar N content for all individual fertilizer treatments (range: 7.86 mg to 9.14 mg) was greater compared to the control (6.02 mg) 6 weeks after fertilization. By week 12, the cumulative individual fascicle foliar N content for individual fertilizer treatments increased to a range of 9.65 mg to 11.48 mg compared to 5.93 mg for the control which decreased from the previous sampling period. By the end of the growing season, the cumulative individual fascicle foliar N content of the control had increased to 8.52 mg but was still lower compared to all individual fertilizer treatments (range: 10.19 mg to 12.95 mg).

3.3.8. Total Cumulative Individual Fascicle Mean Fertilizer N Content

The total cumulative individual fascicle mean fertilizer N content (fertilizer N mg/fascicle) increased for all individual treatments across the entire growing season in all sampling periods after both the spring and summer fertilization (Figure 3.4; In *Appendix Table A.3.7.*).

For the sampling weeks 6, 12, and 18 after a spring fertilization, the total cumulative individual fascicle mean fertilizer N content for the PCU treatment was lower (0.13 mg, 0.40 mg, 1.47 mg respectively) than CUF (0.62 mg, 0.87 mg, 2.13 mg respectively), NBPT (0.79 mg, 0.87 mg, 2.0 mg respectively) and urea (0.58 mg, 0.71 mg, 1.87 mg respectively) (Figure 3.4). For week 24 and week 30, the total cumulative individual fascicle mean fertilizer N content was similar among all individual fertilizer treatments, ranging from 2.20 mg to 2.54 mg for week 24 and 2.41 mg to 3.02 mg in week 30.

For summer fertilization, the total cumulative individual fascicle mean fertilizer N content increased over the growing season (Figure 3.4). For all weeks sampled after fertilization (6, 12, 18), the PCU treatment (0.46 mg, 1.20 mg, 1.51. mg respectively) was generally lower than the other fertilizer treatments including CUF (1.18 mg, 1.35 mg, 2.02 mg respectively), NBPT (1.15 mg, 1.96 mg, 2.50 mg respectively) and urea (1.09 mg, 1.70 mg, 2.41 mg respectively).

3.4. Discussion

Our study compared the temporal trends of foliar N uptake of loblolly pines with five different treatments for two different application seasons across the southern United States. Our study tested three primary hypotheses: 1) no differences existed for foliar growth and development among treatments; 2) differences did not occur in foliar N uptake among treatments; and 3) that there were no differences in foliar fertilizer N uptake among fertilizer treatments.

Our first hypothesis that differences did not exist in foliar growth and development among treatments (control, CUF, NBPT, PCU, urea) was rejected. Differences in foliar growth, assessed by differences in the development and frequency of flushes and individual fascicle mass, occurred for all individual fertilizer treatments compared to the control after both a spring and summer fertilization. Loblolly pine shoot and foliar development, termed flushes or cohorts (Figure 3.2), are episodic growth events that increase in frequency under favorable conditions (Miller 1966, Shultz 1997). The first flush of the growing season, flush 1, generally has the largest shoot elongation, individual fascicle mass, needle length and foliar N of any flush during the growing season (Griffing and Elam 1971, Boyer and South 1989, Zhang et al. 1997). Improving soil nutrition through the application of N + P fertilizers generally increases flush development and individual fascicle mass (Adams and Allen 1985, Valentine and Allen 1990, Zhang et al. 1997, Albaugh et al. 1998, 2004, Tang et al. 1999, Carlson et al. 2014) for loblolly pines.

In our study, fertilizer treatments increased the frequency and development of both flushes and individual fascicle mass when compared to the control, although differences among treatments were difficult to distinguish. For the spring fertilization, all fertilizer plots at all research sites developed a 3rd flush, compared to one control plot at one site in SC. The SC site was one of the more productive sites in this study due to high amounts of decomposing soil organic matter and this site is likely not N limited. Additionally, flush development in this study was more rapid over the growing season compared to the control. Although an increase in flushes after the summer fertilization also occurred, differences in flush frequency were not as great as with the spring fertilization. For the summer fertilization, only the control site at SC had developed a 3rd flush. Additional flushes and increased cumulative foliar mass in the fertilized plots leads to expansion of leaf area (Miller 1981, Vose and Allen 1988, Albaugh et al. 1998, Fox et al. 2007a) increases light interception, and directly improves stand productivity (Russell et al. 1989, Cannell 1989).

Our second hypothesis that foliar N uptake among treatments did not differ was also rejected. Although foliar N concentrations were generally not significantly different between the control and individual fertilizer treatments for the spring or summer fertilization, there was an increasing trend for all fertilizer treatments compared to the control through the growing season and is similar to the findings of others (Switzer et al. 1966, Wells et al. 1975, Zhang and Allen 1996, Albaugh et al. 1998, Gough et al. 2004, Carlson et al. 2014). Significant differences in foliar N concentration can be muted due to dilution effects from increased biomass production in fertilized plots (Jose et al. 2003) but can be accounted for through calculating the individual fascicle N content. Once individual fascicle N content was calculated, there were significant differences between most fertilizer treatments and the control and was similar to the findings of others (Smith et al. 1970, 1971, Switzer et al. 1972, Murthy et al. 1996, Albaugh et al. 2004, Choi et al. 2005, Carlson et al. 2014). Additionally, more cumulative individual fascicle N uptake occurred with all fertilizer treatments compared to unfertilized treatments.

One additional point is the foliar N concentrations in flush 0 (foliage produced in the previous growing season) for the control was consistently low and gradually decreased through the growing season for both spring and summer sampling compared to fertilized treatments, indicating retranslocation of N prior to senescence. Photosynthesis is greatly reduced when foliar N concentrations go below 0.8% (Gough et al. 2004), a value approached for in flush 0 of the control treatments. Conversely, the flush 0 for all individual fertilizer treatments remained at or significantly above 1.00% for the entire growing season for both the spring and summer fertilization. This finding indicates a potential for flush 0 to extend photosynthesis for a period of time near the end of the growing season for fertilized sites, possibly increasing productivity.

The third hypothesis that the foliar fertilizer N uptake was the same among individual fertilizer N treatments was also rejected. Using N containing fertilizers enriched with the stable isotope ^{15}N can improve the understanding of the fate of applied N in forest systems and distinguish fertilizer N uptake in trees compared to the uptake of the N natural existing in the system (Walker 1958, Pritchett and Smith 1972, Nadelhoffer and Fry 1994, Templar et al. 2012). Our study found consistent increases in the foliar ^{15}N (‰) signature for all fertilizer treatments across the entire growing season after both a spring and summer fertilization which continued across the entire growing season for all fertilizer N treatments. There were also slight differences among fertilizer treatments for certain sampling weeks following both a spring and summer fertilization. Specifically, CUF, NBPT and urea all had greater foliar ^{15}N values compared to both the control and PCU earlier in the sampling period, although the differences between foliar ^{15}N values among all fertilizer treatments generally dissipated near the middle of the growing season. One explanation for this finding relates to the release mechanisms and patterns for of each fertilizer treatment.

The EEF treatments used in this study had inhibitors, coatings or a combination to reduce fertilizer N losses through ammonia (NH_3) volatilization and provide a more gradual, constant release of fertilizer N over the growing season (Hauck 1985, Goertz 1993, Azeem et al. 2014, Havlin et al. 2014). Large fertilizer N losses can occur via the ammonia (NH_3) volatilization pathway immediately following urea fertilization, with losses highly dependent on the interaction of environmental and edaphic factors (Cabrera et al. 2005, Zerpa and Fox 2011, Elliot and Fox 2014, Raymond et al. 2016). Coatings or chemical additives applied to fertilizer N (urea) may require degradation that are dependent on the interaction of weather and edaphic factors prior to the fertilizer N becoming plant available, creating a lag period between fertilizer N application and plant N availability. This release lag extends the period when fertilizer N is plant available further into the growing season compared to urea, providing a potentially more synchronous N supply to plant N demand.

Urea, the most commonly applied form of fertilizer N in southern forestry due to its lowest expense per unit N (Allen 1987, Fox et al. 2007a), rapidly degrades (urea dissolution) after application and provides N immediately to plants and soil microbes (Hauck and Bremner 1965). Both urea and urea treated with NBPT likely had the quickest fertilizer N release because there were no coatings to degrade. Evidence is provided in this study where certain flushes for both urea and NBPT have significantly greater individual fascicle mean fertilizer N compared to CUF, but more frequently with PCU. The CUF treatment had urea granules impregnated with NBPT to reduce urease activity with an exterior water soluble coating requiring degradation prior to fertilizer N release to the environment. The combined technologies in CUF assist in reducing NH_3 volatilization compared to urea (Raymond et al. 2016) and provide a more gradual release of fertilizer N to the environment. A lag in fertilizer N release for CUF is supported in this study by the foliar ^{15}N data where CUF, specifically earlier in the growing season for certain flushes, had lower foliar ^{15}N values compared to both NBPT and urea. Yet most of these differences disappeared as the growing season progressed. Research in agriculture systems has shown it can take 2 days to 21 days for complete hydrolysis of soluble fertilizers (NBPT, CUF and urea) and is highly dependent on environmental variables during this period such as relative humidity, precipitation, and temperature (Gould et al. 1973, Bayrakli 1990, Shaviv 1996, Azeem et al. 2014, Havlin et al. 2014).

Conversely, the PCU technology is a semi-permeable coating surrounding urea granules which requires water to enter the granule, urea dissolution to occur, and a threshold internal pressure in the granule to force the fertilizer N in solution out of the granule and into the environment. This technology likely provides the most extended, gradual release of fertilizer N to the environment with estimates of fertilizer N release ranging from 3 months to 6 months, or potentially longer, and dependent primarily on moisture and temperature (Shaviv 1996, Azeem 2014). Foliar ^{15}N data from this study again supports this statement, with PCU generally having the lowest foliar ^{15}N values compared to all other individual treatments early in the growing season.

Although fertilizer release patterns have been shown to be generally dependent on a complex interaction of environmental variables in agriculture, less is known about release patterns of EEFs and how this translates to fertilizer N uptake for trees in forested systems. One of the few studies examining EEF release patterns in loblolly pine plantation systems for these same products is by Werner (2013). Werner (2013) observed an increase in NH_4^+ using ion exchange resins (IER) 14 days after a fall application for CUF, NBPT and urea with no differences among these treatments, compared to an increase in NH_4^+ for PCU at 49 days after application. Werner (2013) also found a higher percentage of labeled ^{15}N fertilizer in certain components of the tree for CUF, NBPT and urea when compared to PCU, indicating a correlation between release patterns and fertilizer N uptake by loblolly pines. Results from our study indicate similar trends for the respective fertilizer treatments for both a spring and summer fertilizer application. Few differences occurred among fertilizer treatments early in the growing season, these differences gradually fade. Additional research will be needed to understand release patterns in forested systems, and how much of these products remain in the soil to extend potential plant N availability for successive growing seasons.

A final point of this research is the overall foliar N uptake of the loblolly pines. Although it is evident that fertilizer N additions increase overall foliar N uptake, it does not account for all of the additional foliar N uptake. If fertilizer N uptake had accounted for all the additional N in the foliage for the fertilizer treatments, the sum of the cumulative fertilizer

N for individual treatments in fascicles and the control (unfertilized) individual fascicle N would equal the cumulative individual fascicle N content for each fertilizer treatment- but this did not occur. For example, at the end of the growing season the individual fascicle mean fertilizer N content was approximately 3.00 mg (CUF = 2.78 mg, NBPT = 3.02 mg, PCU = 2.41 mg, urea = 2.98 mg). Yet when the cumulative individual fascicle N content for the control plot (7.47 mg) is added to individual fascicle mean fertilizer N content values for the individual fertilizer treatments, there is a discrepancy of approximately 2.50 mg (CUF = 12.26 mg, NBPT = 12.48 mg, PCU = 11.48 mg) for the EEFs and less than 0.50 mg for urea (10.77 mg). Additional research will be required to understand the mechanisms behind this process, but one hypothesis is that the enhanced efficiency fertilizers specifically retain more fertilizer N in the soil compared to urea (Raymond et al. 2016). This would translate to an increase in fertilizer N availability to the loblolly pines and also an increase in the availability of naturally existing N.

Fertilization research in southern loblolly pine plantations has improved the productivity of these systems (Albaugh et al. 1998, Vogel and Jokela 2011, Carlson et al. 2014), but understanding mechanisms that quantify the proportion of fertilizer N taken up by loblolly pines is generally hampered by the inability to distinguish the origins of natural versus fertilizer N. Fertilizer use efficiency is usually assessed through ecosystem productivity metrics such as foliar N concentrations, foliar nutrient ratios, diameter and height measurements, leaf area index, root to shoot ratios, or various other metrics. By using fertilizers enriched with the stable isotope ^{15}N, this research has been able to improve the understanding of plant uptake and integrate processes, mechanisms and pathways of the fertilizer N cycle in southern loblolly pine plantations.

This research has refined the understanding of foliar N uptake over the course of a growing season after a spring and summer N application in mid-rotation loblolly pines through the use of fertilizers enriched with the stable isotopes ^{15}N. Representative sites were chosen across an extensive geographic region where loblolly pine plantations are operationally fertilized and intensively managed to integrate the range of climatic and edaphic variables occurring in the southern United States. The synthesis of significant factors from this study help support and add to the knowledge obtained from the numerous other studies in forest ecosystems focused on smaller regions. The results from this study will continue to improve our ability to assess loblolly pine fertilizer N response through the refinement of fertilizer use efficiency for these high production systems.

3.5. Conclusions

The primary results from this research specific to loblolly pines were: 1) significantly more foliar growth occurs at a faster rate after N fertilization; 2) N fertilization increases N uptake in all stages of foliar development, including older foliage, which increases the N concentration of individual fascicles and increases photosynthetic potential of older foliage; and 3) significantly more foliar N uptake occurs after N fertilization of both fertilizer and naturally existing N. In a companion study, Raymond et al. (2016) found NH_3 volatilization was reduced with the same EEFs used in this study to assess foliar N uptake. Additional research will be required to determine if the additional fertilizer N remaining in the soil from the EEFs will translate into additional plant available N in successive seasons and continue to increase fertilizer N use efficiency. Results from this research can be applied through the southern United States where mid-rotation loblolly pine are intensively managed and should assist in improving forest managers goals of increasing N use efficiency to increase productivity.

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Figures

Figure 3.1. *Location map of loblolly pine stands in the southern United States selected to evaluate temporal foliar uptake of fertilizer nitrogen during the growing season using* ^{15}N *enriched fertilizers following the application of urea or enhanced efficiency nitrogen fertilizers after a spring and summer fertilizer application.*

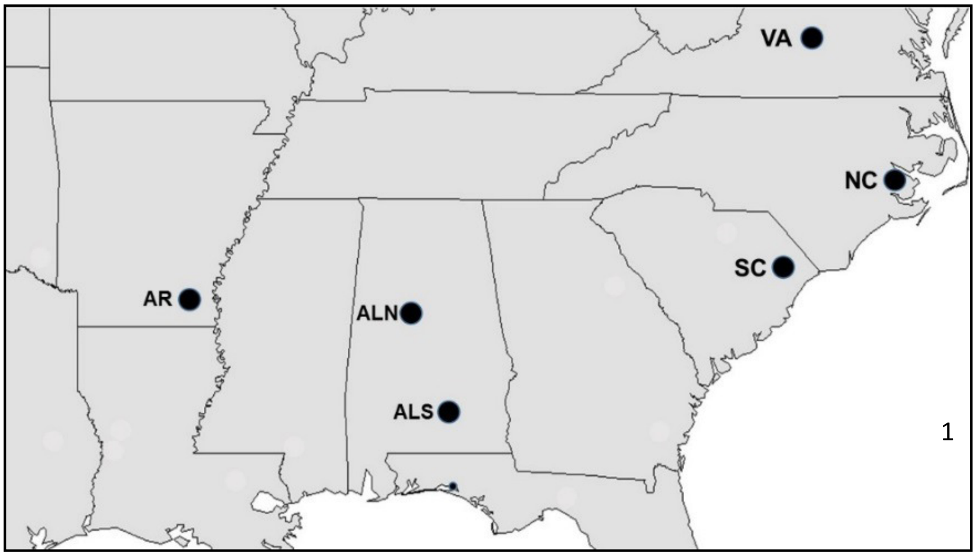


Figure 3.2. *Representation of individual flush growth on a loblolly pine sampled every six weeks over the growing season after a spring and summer fertilization in the southern United States selected to evaluate N uptake of urea or enhanced efficiency N containing fertilizers.*

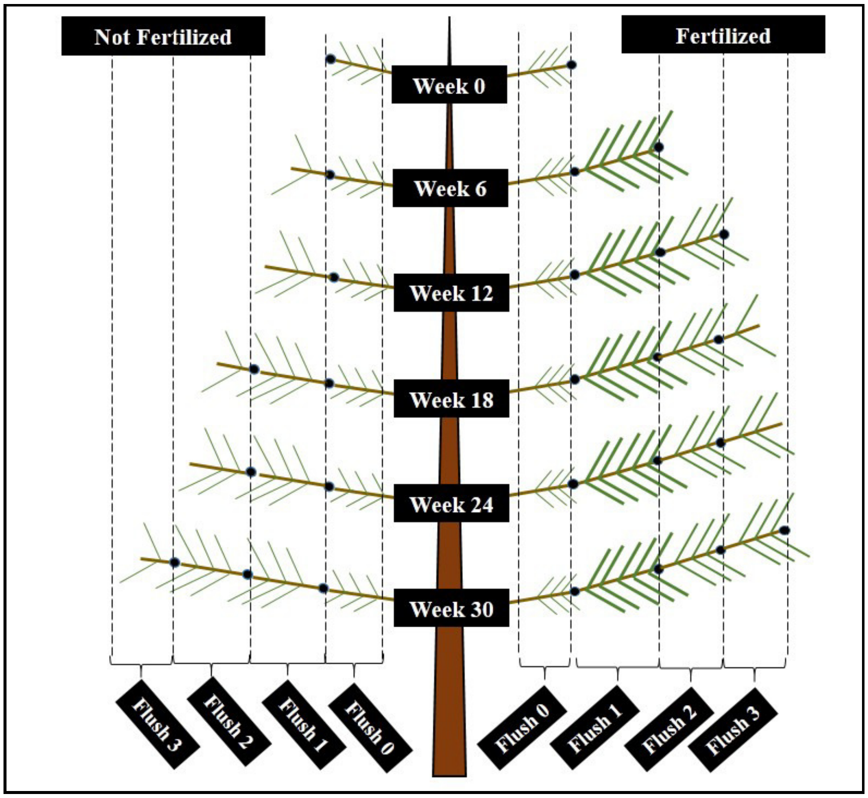


Figure 3.3. *Cumulative individual fascicle N content (mg N fascicle*$^{-1}$*) of individual flushes combined for sampling weeks (6, 12, 18, 24, 30) after spring and summer fertilization (2011) for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with* ^{15}*N. The N application rate was 224 kg N ha*$^{-1}$*. N = 6.*

Spring

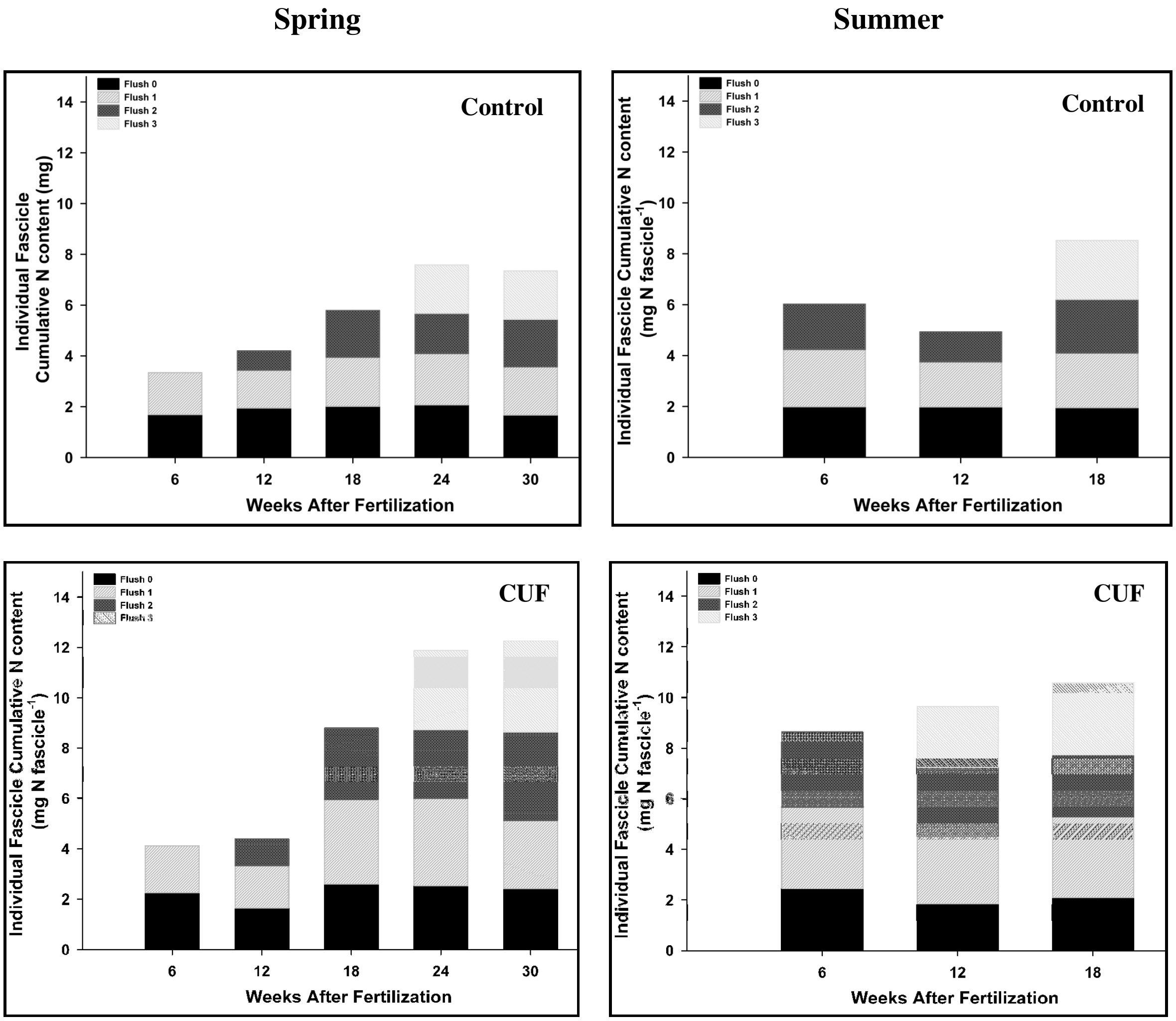


Figure 3.3 (cont.)

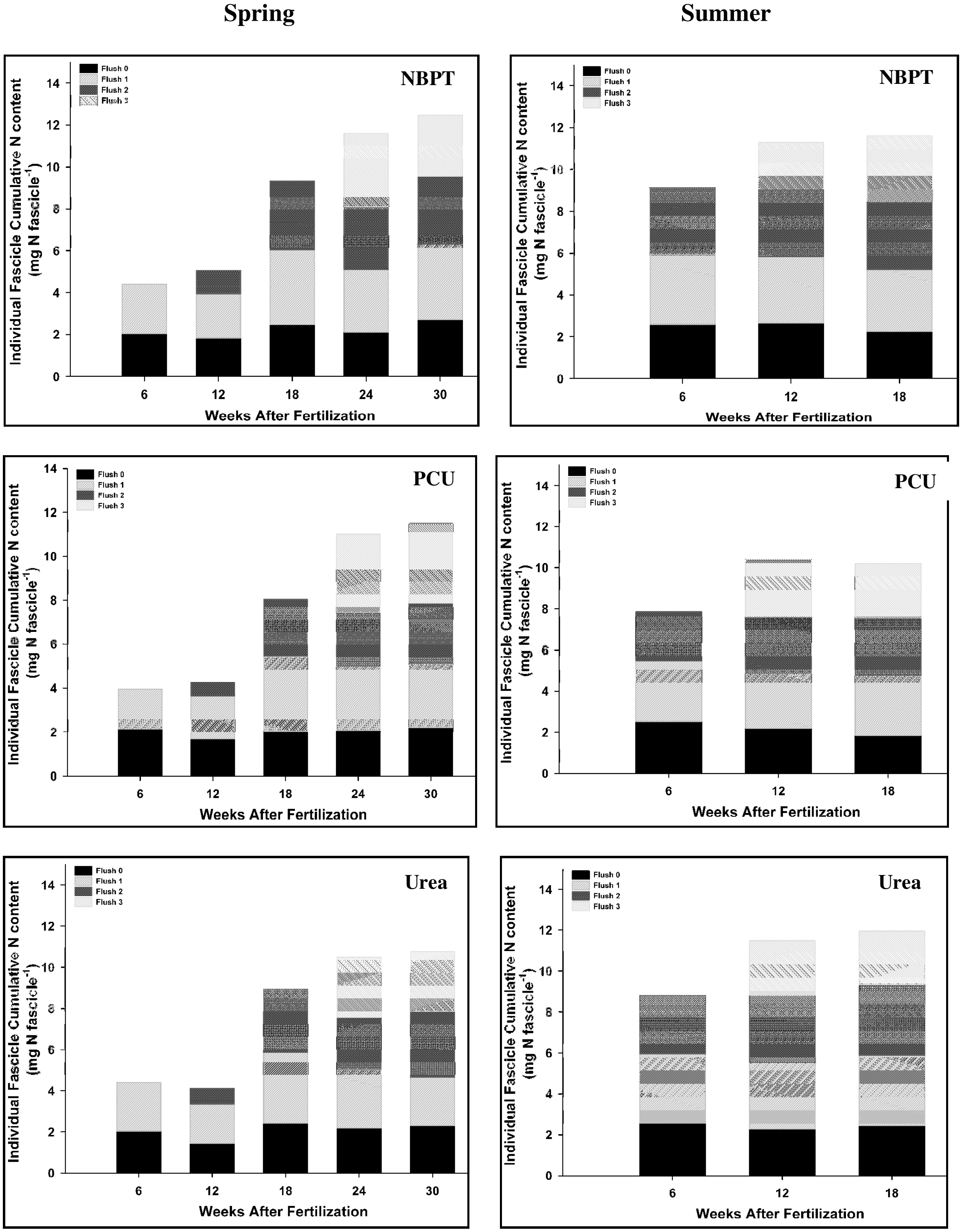


Figure 3.4. *Cumulative individual fascicle N content from fertilizer (mg N fascicle^{-1}) of individual flushes (0, 1, 2) combined for sampling weeks (6, 12, 18) after spring and summer fertilization (2011) for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. The N application rate was 224 kg N ha^{-1}. N = 6.*

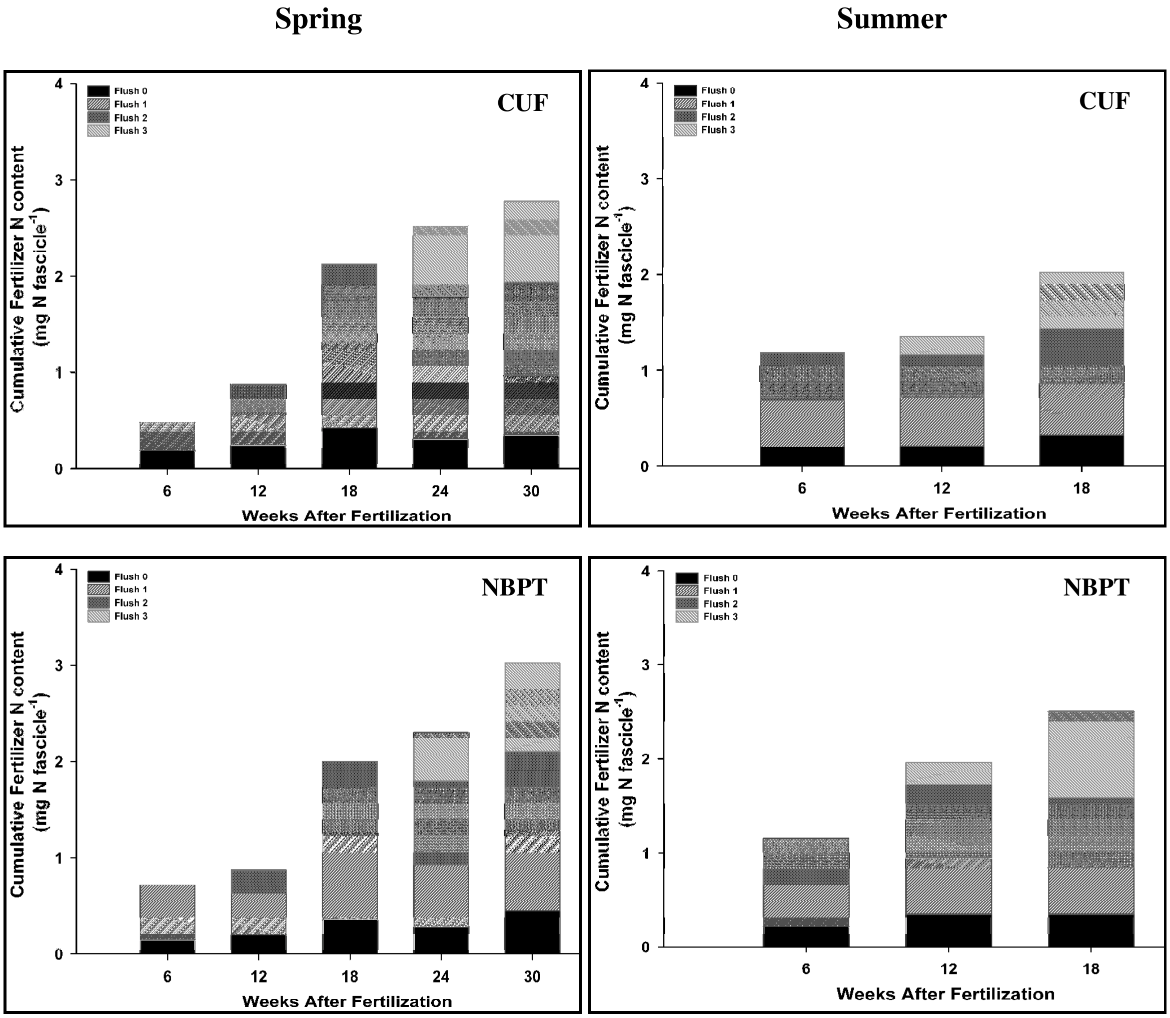
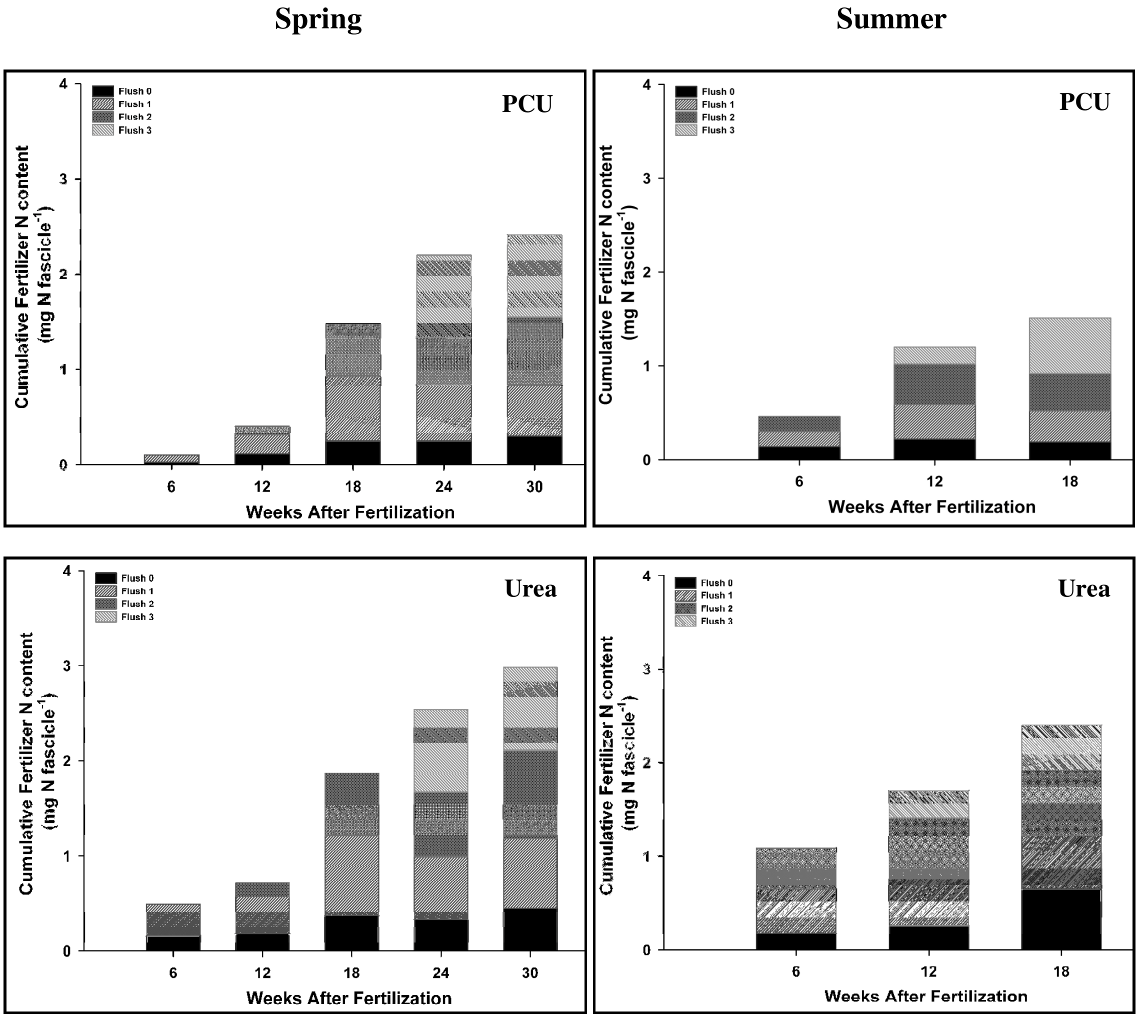


Figure 3.4. (continued)



1

Tables

Table 3.1. *Selected climate and physical characteristics of loblolly pine stands in the southern United States selected to evaluate temporal foliar uptake of fertilizer nitrogen following the application of urea or enhanced efficiency nitrogen fertilizers enriched with ^{15}N.*

Site	Latitude	Longitude	Altitude (m)	Mean Annual Precipitation (cm)	Mean Annual Temperature (°C)	Physiographic Region	Soil Taxonomic Class	Soil Series	Soil Drainage Class
VA	37.440087	78.662396	197	109	13	Southern Piedmont	fine, mixed, subactive, mesic Typic Hapludults	Littlejoe	Well
SC	33.869400	79.289300	2	125	17	Atlantic Coast Flatwoods	fine, kaolinitic, thermic Umbric Paleaquults	Byars	Poorly
NC	35.317006	78.514167	0.5	121	16	Atlantic Coast Flatwoods	fine-silty, mixed, active, thermic Typic Albaquults	Leaf	Poorly
AR	33.422310	91.732651	69	140	16	Western Gulf Coastal Plain	fine-silty, mixed, active, thermic, Typic Glossaqualfs	Calhoun	Poorly
ALN	33.233371	87.232384	158	125	17	Appalachian Plateau	loamy-skeletal, mixed, subactive, thermic, shallow, Typic Dystrudepts	Montevallo	Well
ALS	31.659464	86.272045	111	145	26	Southern Coastal Plain	fine, smectitic, thermic, Typic Hapludults	Arundel	Well

Table 3.2. *Selected characteristics of loblolly pine stands in the southern United States selected to evaluate selected to evaluate temporal foliar uptake of fertilizer nitrogen following the application of urea or enhanced efficiency nitrogen fertilizers enriched with ^{15}N.*

Site	**Age**	**Trees/Hectare**	**Height (m)**	**DBH (cm)**	**BA (m^2 ha^{-1})**
VA	17	1100-1200	13.1	16.5	26.8
SC	16	500-600	16.0	25.7	29.7
NC	18	100-200	18.7	27.2	7.7
AR	13	300-400	15.3	20.8	22.4
ALN	19	300-400	18.8	24.2	25.2
ALS	19	500-600	18.5	21.1	20.4

Table 3.3. *The number of study sites where flushes were observed for each treatment (control, CUF, NBPT, PCU, urea) in sampling periods after a spring and summer fertilization for loblolly pine stands in the southern United States selected to evaluate N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. (*) represents date of fertilization. (-) represents potential flushes that were not measured in the sampling period.*

Spring Fertilization

Calendar Date		**3/27-4/8***		**5/8-5/13**				**6/20-6/30**				**7/31-8/4**				**9/11-9/15**				**10/25-10/27**			
Weeks after Fertilization		***0***		***6***				***12***				***18***				***24***				***30***			
Number Flushes Present		**0**	**1**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Treatment	**Control**	6	6	6	6	0	0	6	6	1	0	6	6	6	0	6	6	6	1	6	6	6	1
	CUF	6	6	6	6	2	0	6	6	6	0	6	6	6	2	6	6	6	4	6	6	6	6
	NBPT	6	6	6	6	2	0	6	6	6	0	6	6	6	2	6	6	6	4	6	6	6	6
	PCU	6	6	6	6	2	0	6	6	6	0	6	6	6	2	6	6	6	4	6	6	6	6
	Urea	6	6	6	6	2	0	6	6	6	0	6	6	6	2	6	6	6	4	6	6	6	6

Summer Fertilization

Calendar Date		**3/27-4/8***		**5/8-5/13**				**6/20-6/30***				**7/31-8/4**				**9/11-9/15**				**10/25-10/27**			
Weeks after Fertilization		**-**		**-**				***0***				***6***				***12***				***18***			
Number Flushes Present		**0**	**1**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Treatment	**Control**	-	-	-	-	-	-	6	6	1	0	6	6	6	0	6	6	6	0	6	6	6	1
	CUF	-	-	-	-	-	-	6	6	2	0	6	6	6	0	6	6	6	2	6	6	6	3
	NBPT	-	-	-	-	-	-	6	6	1	0	6	6	6	0	6	6	6	1	6	6	6	3
	PCU	-	-	-	-	-	-	6	6	2	0	6	6	6	0	6	6	6	1	6	6	6	3
	Urea	-	-	-	-	-	-	6	6	1	0	6	6	6	0	6	6	6	3	6	6	6	3

Table 3.4. *The mean individual fascicle mass (g) of flushes in sampling periods after a spring and summer fertilization for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Different letters represent significant differences at $\alpha = 0.05$ among treatments in a sampling period. Numbers in parentheses are the standard error of the mean, and absent values and letters indicate post-hoc analysis not conducted due to a limited sample size. The N application rate was 224 kg N ha^{-1}. N = 6.*

Spring Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	***6***			***12***			***18***			***24***				***30***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	0.16^{a} (0.02)	0.15^{a} (0.04)	-	0.14^{a} (0.02)	0.19^{a} (0.02)	0.07	0.19^{a} (0.02)	0.17^{a} (0.01)	0.17^{a} (0.01)	0.21^{a} (0.0)	0.18^{a} (0.02)	0.15^{a} (0.02)	0.19	0.19^{a} (0.02)	0.17^{a} (0.01)	0.16^{a} (0.01)	0.19
CUF	0.18^{a} (0.02)	0.18^{a} (0.03)	0.04	0.14^{a} (0.01)	0.21^{a} (0.01)	0.08	0.21^{a} (0.01)	**0.24^{b}** **(0.01)**	**0.22^{b}** **(0.01)**	0.21^{a} (0.02)	**0.25^{b}** **(0.02)**	**0.21^{b}** **(0.02)**	0.24	0.21^{a} (0.02)	**0.23^{b}** **(0.01)**	**0.25^{c}** **(0.01)**	0.24
NBPT	0.17^{a} (0.02)	0.18^{a} (0.03)	0.02	0.15^{a} (0.02)	0.19^{a} (0.02)	0.08	0.19^{a} (0.02)	**0.24^{b}** **(0.01)**	0.21^{ab} (0.03)	0.18^{a} (0.02)	0.22^{ab} (0.01)	**0.21^{b}** **(0.02)**	0.26	0.20^{a} (0.01)	**0.25^{b}** **(0.01)**	**0.22^{bc}** **(0.01)**	0.26
PCU	0.19^{a} (0.02)	0.16^{a} (0.03)	0.03	0.16^{a} (0.02)	0.17^{a} (0.03)	0.06	0.17^{a} (0.03)	**0.24^{b}** **(0.02)**	0.20^{ab} (0.02)	0.18^{a} (0.01)	**0.23^{b}** **(0.02)**	0.18^{ab} (0.03)	0.25	0.20^{a} (0.02)	**0.24^{b}** **(0.02)**	0.19^{ab} (0.02)	0.25
Urea	0.16^{a} (0.02)	0.19^{a} (0.02)	0.05	0.15^{a} (0.02)	0.20^{a} (0.02)	0.06	0.20^{a} (0.02)	**0.24^{b}** **(0.01)**	**0.22^{b}** **(0.02)**	0.18^{a} (0.02)	0.21^{ab} (0.01)	0.19^{ab} (0.01)	0.23	0.18^{a} (0.02)	**0.24^{b}** **(0.02)**	**0.23^{bc}** **(0.02)**	0.23

Summer Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	**-**			**-**			***6***			***12***				***18***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	-	-	-	-	-	-	0.20^{a} (0.01)	0.20^{a} (0.01)	0.16^{a} (0.01)	0.18^{a} (0.01)	0.18^{a} (0.01)	0.17^{a} (0.01)	-	0.19^{a} (0.02)	0.20^{a} (0.02)	0.18^{a} (0.01)	0.22
CUF	-	-	-	-	-	-	0.21^{a} (0.01)	0.22^{ab} (0.02)	**0.22^{b}** **(0.02)**	0.21^{a} (0.02)	0.21^{ab} (0.02)	0.20^{ab} (0.03)	0.16 (0.01)	0.19^{a} (0.02)	**0.24^{b}** **(0.03)**	0.19^{ab} (0.02)	0.20 (0.02)
NBPT	-	**-**	**-**	-	-	-	0.21^{a} (0.01)	**0.25^{c}** **(0.01)**	**0.23^{b}** **(0.01)**	0.23^{a} (0.01)	**0.23^{b}** **(0.01)**	**0.23^{b}** **(0.01)**	0.15	0.19^{a} (0.02)	0.24^{ab} (0.02)	**0.24^{b}** **(0.01)**	0.20 (0.02)
PCU	-	**-**	**-**	-	-	-	0.21^{a} (0.01)	**0.23^{bc}** **(0.01)**	**0.20^{b}** **(0.01)**	0.21^{a} (0.03)	0.21^{ab} (0.03)	0.20^{ab} (0.02)	0.17	0.17^{a} (0.03)	**0.24^{b}** **(0.01)**	0.21^{ab} (0.02)	0.20 (0.01)
Urea	-	**-**	**-**	-	-	-	0.21^{a} (0.01)	**0.24^{bc}** **(0.01)**	**0.20^{b}** **(0.01)**	0.24^{a} (0.01)	**0.24^{b}** **(0.01)**	**0.23^{b}** **(0.02)**	0.17 (0.01)	0.21^{a} (0.01)	**0.26^{b}** **(0.01)**	**0.25^{b}** **(0.01)**	0.23 (0.04)

Table 3.5. *The mean foliar N concentration (g kg^{-1}) of individual flushes for sampling periods after a spring and summer fertilization for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Different letters represent significant differences at $\alpha = 0.05$ among treatments in a sampling period. Numbers in parentheses are the standard error of the mean, and absent values and letters indicate post-hoc analysis not conducted due to a limited sample size. The N application rate was 224 kg N ha^{-1}. N = 6.*

Spring Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	***6***			***12***			***18***			***24***				***30***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	10.8^{a} (0.5)	11.2^{a} (0.8)	-	10.8^{a} (0.4)	11.0^{a} (0.4)	10.6	10.3^{a} (0.5)	11.6^{a} (0.8)	11.4^{a} (0.7)	9.6^{a} (0.7)	11.4^{a} (1.0)	10.5^{a} (1.0)	10.2	8.6^{a} (0.6)	10.9^{a} (0.8)	11.7^{a} (0.6)	10.2
CUF	12.8^{a} (0.5)	10.4^{a} (0.7)	8.6	11.9^{a} (0.7)	12.1^{a} (0.4)	12.9	12.7^{a} (0.9)	14.0^{a} (0.8)	12.8^{ab} (0.7)	12.0^{a} (0.9)	13.6^{a} (1.3)	13.1^{a} (1.2)	12.9	11.5^{ab} (0.6)	12.3^{a} (1.3)	14.0^{a} (0.5)	13.7
NBPT	12.1^{a} (0.8)	12.5^{a} (0.8)	6.8	12.5^{a} (1.0)	13.0^{a} (1.3)	13.9	13.0^{a} (1.2)	15.3^{a} (1.3)	**16.0^{b}** **(1.7)**	11.4^{a} (0.6)	13.4^{a} (1.6)	13.8^{a} (1.2)	13.5	**13.0^{b}** **(1.6)**	14.5^{a} (1.6)	14.1^{a} (0.7)	13.8
PCU	11.1^{a} (0.2)	11.7^{a} (0.7)	7.1	11.7^{a} (0.5)	12.4^{a} (0.7)	10.1	12.3^{a} (0.5)	14.4^{a} (0.8)	12.8^{ab} (0.6)	11.3^{a} (0.7)	12.8^{a} (1.1)	13.4^{a} (0.7)	14.1	11.5^{ab} (0.5)	12.4^{a} (1.0)	13.9^{a} (0.4)	14.1
Urea	12.5^{a} (0.4)	12.8^{a} (1.2)	7.7	12.2^{a} (0.9)	12.8^{a} (1.0)	12.0	12.2^{a} (0.7)	14.4^{a} (1.4)	13.8^{ab} (1.6)	12.4^{a} (0.5)	13.7^{a} (1.2)	13.3^{a} (0.9)	12.8	**12.6^{b}** **(0.7)**	12.3^{a} (1.0)	13.9^{a} (0.8)	12.8

Summer Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	**-**			**-**			***6***			***12***				***18***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	-	-	-	-	-	-	9.8^{a} (0.5)	11.5^{a} (0.9)	11.1^{a} (0.9)	10.0^{a} (0.8)	10.2^{a} (0.6)	10.4^{a} (0.7)	-	10.3^{a} (1.0)	10.9^{a} (1.0)	11.8^{a} (0.4)	10.5
CUF	-	-	-	-	-	-	11.6^{a} (0.7)	14.5^{a} (1.4)	13.6^{a} (1.4)	10.6^{a} (0.7)	13.6^{a} (1.6)	13.1^{ab} (0.6)	14.4	10.8^{a} (0.9)	12.9^{a} (1.4)	13.3^{a} (0.8)	13.6
NBPT	-	-	-	-	-	-	12.7^{a} (1.0)	14.1^{a} (0.9)	14.2^{a} (1.1)	12.8^{a} (1.3)	13.2^{a} (1.1)	14.1^{ab} (0.9)	14.6	11.9^{a} (0.8)	12.5^{a} (1.2)	13.8^{a} (0.9)	15.4
PCU	-	-	-	-	-	-	11.9^{a} (0.7)	12.9^{a} (1.0)	11.6^{a} (1.2)	1.13^{a} (0.6)	13.2^{a} (1.0)	13.3^{ab} (1.2)	16.3	10.0^{a} (1.2)	11.5^{a} (0.8)	12.8^{a} (1.0)	12.7
Urea	-	-	-	-	-	-	12.6^{a} (0.6)	14.1^{a} (0.6)	14.1^{a} (1.0)	11.6^{a} (1.1)	14.0^{a} (1.2)	**14.2^{b}** **(1.0)**	14.8	11.3^{a} (0.7)	13.4^{a} (0.9)	13.9^{a} (0.9)	15.4

Table 3.6. *The mean individual fascicle N content (mg N fascicle^{-1}) of flushes per sampling periods after a spring and summer fertilization for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Different letters represent significant differences at α = 0.05 among treatments in a sampling period. Numbers in parentheses are the standard error of the mean, and absent values and letters indicate post-hoc analysis not conducted due to a limited sample size. The N application rate was 224 kg N ha^{-1}. N = 6.*

Spring Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	***6***			***12***			***18***			***24***				***30***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	1.69^{a} (0.28)	1.65^{a} (0.49)	-	1.95^{a} (0.24)	1.49^{a} (0.23)	0.67	2.02^{a} (0.21)	1.93^{a} (0.21)	1.82^{a} (0.16)	2.03^{a} (0.23)	2.02^{a} (0.32)	1.59^{a} (0.34)	1.90	1.74^{a} (0.03)	1.91^{a} (0.02)	1.91^{a} (0.01)	1.91
CUF	2.24^{a} (0.28)	1.87^{a} (0.28)	0.56	1.64^{a} (0.34)	1.69^{a} (0.18)	1.06 (0.04)	2.60^{a} (0.12)	**3.35^{b}** **(0.36)**	**2.84^{b}** **(0.13)**	2.53^{a} (0.36)	**3.45^{b}** **(0.54)**	**2.75^{b}** **(0.40)**	3.15 (0.79)	2.41^{ab} (0.02)	**2.72^{b}** **(0.02)**	**3.51^{c}** **(0.03)**	3.62
NBPT	2.04^{a} (0.26)	2.38^{a} (0.50)	0.58	1.82^{a} (0.39)	2.13^{a} (0.34)	1.12 (0.06)	2.47^{a} (0.25)	**3.57^{b}** **(0.18)**	**3.29^{b}** **(0.50)**	2.11^{a} (0.22)	**3.01^{b}** **(0.45)**	**3.00^{b}** **(0.43)**	3.49 (0.72)	**2.72^{b}** **(0.04)**	**3.62^{c}** **(0.05)**	**3.22^{bc}** **(0.03)**	2.92
PCU	2.13^{a} (0.24)	1.83^{a} (0.41)	0.21	1.69^{a} (0.29)	1.95^{a} (0.20)	0.62 (.20)	2.04^{a} (0.29)	**3.43^{b}** **(0.29)**	2.57^{ab} (0.32)	2.08^{a} (0.26)	**2.93^{b}** **(0.37)**	**2.43^{b}** **(0.38)**	3.56 (1.03)	2.21^{ab} (0.03)	**2.91^{bc}** **(0.02)**	**2.75^{b}** **(0.02)**	3.61
Urea	2.04^{a} (0.04)	2.36^{a} (0.36)	0.48	1.43^{a} (0.28)	1.92^{a} (0.27)	0.76 (0.15)	2.42^{a} (0.03)	**3.44^{b}** **(0.41)**	**3.08^{b}** **(0.53)**	2.18^{a} (0.23)	**2.86^{b}** **(0.40)**	**2.54^{b}** **(0.39)**	2.92 (0.47)	2.31^{ab} (0.03)	**2.34^{bc}** **(0.03)**	**3.21^{bc}** **(0.04)**	2.91

Summer Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization							***6***			***12***				***18***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	-	-	-	-	-	-	1.99^{a} (0.20)	2.25^{a} (0.02)	1.78^{a} (0.22)	1.98^{a} (0.24)	1.77^{a} (0.18)	1.18^{a} (0.02)	-	1.95^{a} (0.30)	2.15^{a} (0.28)	2.11^{a} (0.15)	2.31
CUF	-	-	-	-	-	-	2.45^{ab} (0.24)	**3.21^{b}** **(0.49)**	**2.98^{c}** **(0.41)**	1.85^{a} (0..23)	**2.68^{b}** **(0.36)**	**2.71^{b}** **(0.04)**	2.41 (0.37)	2.10^{a} (0.39)	**3.19^{b}** **(0.51)**	**2.46^{b}** **(0.26)**	2.82 (0.47)
NBPT	-	-	-	-	-	-	**2.58^{b}** **(0.16)**	**3.45^{b}** **(0.27)**	**3.11^{c}** **(0.30)**	**2.64^{b}** **(0.43)**	**3.18^{b}** **(0.21)**	**3.23^{b}** **(0.02)**	2.24	2.26^{a} (0.35)	2.94^{ab} (0.37)	**3.29^{c}** **(0.28)**	3.12 (0.32)
PCU	-	-	-	-	-	-	2.53^{ab} (0.20)	**2.94^{b}** **(0.82)**	**2.39^{b}** **(0.36)**	2.19^{ab} (0.20)	**2.69^{b}** **(0.39)**	**2.72^{b}** **(0.04)**	2.81	1.86^{a} (0.33)	2.91^{ab} (0.16)	**2.78^{b}** **(0.33)**	2.64 (0.61)
Urea	-	-	-	-	-	-	2.57^{ab} (0.10)	**3.38^{b}** **(0.35)**	**2.86^{bc}** **(0.21)**	2.27^{ab} (0.17)	**3.26^{b}** **(0.33)**	**3.31^{b}** **(0.03)**	2.64 (0.18)	2.43^{a} (0.28)	**3.46^{b}** **(0.24)**	**3.46^{c}** **(0.32)**	3.60 (0.62)

Table 3.7. *The mean foliar ^{15}N (‰) of individual flushes for sampling periods after a spring and summer fertilization for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Different letters represent significant differences at α = 0.05 among treatments in a sampling period. Numbers in parentheses are the standard error of the mean, and absent values and letters indicate post-hoc analysis not conducted due to a limited sample size. The N application rate was 224 kg N ha^{-1}. N = 6.*

Spring Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	***6***			***12***			***18***			***24***				***30***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	-3.22^{a} (1.0)	-2.61^{a} (1.2)	-	-2.96^{a} (0.9)	-3.07^{a} (1.1)	-2.80	-3.43^{a} (0.6)	-3.28^{a} (1.0)	-1.56^{a} (0.7)	-3.38^{a} (1.0)	-3.94^{a} (1.3)	-2.84^{a} (0.9)	0.41	-2.50^{a} (0.73)	-3.15^{a} (0.89)	-2.92^{a} (0.58)	0.42
CUF	36.3^{a} (4.1)	**70.0^{bc}** **(11.0)**	45.6	**58.8^{b}** **(7.2)**	**92.6^{c}** **(13.9)**	106.7	**69.9^{b}** **(8.3)**	**114.4^{b}** **(18.1)**	**123.1^{b}** **(10.2)**	**56.7^{b}** **(8.5)**	**92.1^{b}** **(10.6)**	**113.4^{b}** **(10.7)**	111.2	**65.4^{b}** **(7.4)**	**95.4^{b}** **(12.2)**	**120.1^{b}** **(11.2)**	114.4
NBPT	32.0^{a} (3.0)	**89.4^{c}** **(19.5)**	38.5	43.9^{ab} (7.5)	**90.2^{bc}** **(16.6)**	87.6	**57.7^{b}** **(7.8)**	**100.8^{b}** **(17.5)**	**109.1^{b}** **(15.0)**	**56.3^{b}** **(6.0)**	**84.0^{b}** **(8.0)**	**116.3^{b}** **(12.2)**	92.8	**64.6^{b}** **(10.5)**	**94.0^{b}** **(14.3)**	**105.5^{b}** **(14.5)**	134.2
PCU	5.37^{a} (2.9)	17.8^{ab} (7.03)	9.72	24.5^{ab} (7.5)	40.9^{ab} (8.7)	60.4	44.0^{ab} (7.4)	**76.8^{b}** **(12.5)**	**85.4^{b}** **(12.3)**	48.4^{b} (6.3)	**77.2^{b}** **(13.9)**	**80.3^{b}** **(5.7)**	106.1	**53.5^{b}** **(9.3)**	**73.8^{b}** **(7.4)**	**107.2^{b}** **(11.5)**	106.1
Urea	31.1^{a} (2.5)	**58.6^{bc}** **(11.2)**	41.2	47.4^{ab} (7.5)	**83.2^{bc}** **(13.7)**	106.4	**60.4^{b}** **(6.9)**	**96.3^{b}** **(17.3)**	**103.5^{b}** **(14.2)**	**65.9^{b}** **(18.6)**	**90.7^{b}** **(12.3)**	**112.1^{b}** **(7.4)**	119.3	**80.1^{b}** **(11.8)**	**94.4^{b}** **(12.5)**	**117.9^{b}** **(8.1)**	119.3

Summer Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	***-***			***-***			***6***			***12***				***18***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	-	-	-	-	-	-	-3.11^{a} (0.70)	-2.90^{a} (0.66)	-2.10^{a} (0.93)	-3.04^{a} (1.01)	-2.84^{a} (0.81)	-2.55^{a} (0.89)	-	-2.87^{a} (0.66)	-3.14^{a} (0.81)	-2.84^{a} (0.81)	0.51
CUF	-	-	-	-	-	-	35.2^{a} (8.3)	**61.7^{b}** **(13.3)**	**72.8^{c}** **(19.1)**	**49.5^{b}** **(6.3)**	**73.1^{b}** **(8.9)**	**76.0^{b}** **(10.3)**	92.1	**58.7^{b}** **(9.4)**	**69.4^{b}** **(9.9)**	**102.6^{b}** **(16.4)**	109.2
NBPT	-	-	-	-	-	-	33.6^{a} (6.2)	**52.1^{b}** **(8.6)**	**64.8^{bc}** **(10.4)**	**53.3^{b}** **(5.4)**	**75.0^{b}** **(9.2)**	**99.3^{b}** **(11.3)**	158.9	**60.9^{b}** **(8.0)**	**65.5^{b}** **(9.7)**	**90.5^{b}** **(11.5)**	118.6
PCU	-	-	-	-	-	-	21.0^{a} (7.0)	22.9^{ab} (6.2)	28.6^{ab} (5.9)	**40.5^{b}** **(3.8)**	**52.4^{b}** **(5.9)**	**67.2^{b}** **(10.9)**	58.7	**40.8^{b}** **(5.8)**	**45.6^{b}** **(5.2)**	**58.0^{b}** **(7.8)**	76.99
Urea	-	-	-	-	-	-	30.3^{a} (7.7)	**59.2^{b}** **(5.9)**	**66.5^{bc}** **(9.6)**	**47.1^{b}** **(6.4)**	**63.7^{b}** **(9.9)**	**82.6^{b}** **(7.6)**	95.0	**47.7^{b}** **(10.0)**	**68.4^{b}** **(7.1)**	**86.3^{b}** **(9.01)**	94.64

Table 3.8. *The mean individual fascicle fertilizer N (mg N fascicle^{-1}) content for flushes in sampling periods of loblolly pine stands in the southern United States selected to evaluate seasonal fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after a spring and summer fertilization. Different letters represent significant differences at $\alpha = 0.05$ among treatments in a sampling period. Numbers in parentheses are the standard error of the mean, and absent values and letters indicate post-hoc analysis not conducted due to a limited sample size. The N application rate was 224 kg N ha^{-1}. N = 6.*

Spring Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization	***6***			***12***			***18***			***24***				***30***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	0.00^{a}	0.00^{a}	0.00	0.00^{a}	0.00^{a}	0.00	0.00^{a}	0.00^{a}	0.00^{a}	0.00^{a}	0.00^{a}	0.00^{a}	0.00	0.00^{a}	0.00^{a}	0.00^{a}	0.00
CUF	0.19^{a} (0.03)	**0.29^{b}** **(0.05)**	0.14	**0.25^{c}** **(0.08)**	**0.34^{bc}** **(0.04)**	0.28	**0.43^{b}** **(0.06)**	**0.88^{b}** **(0.13)**	**0.82^{b}** **(0.07)**	**0.31^{b}** **(0.04)**	**0.76^{b}** **(0.16)**	**0.72^{bc}** **(0.10)**	0.73	**0.35^{b}** **(0.02)**	**0.62^{bc}** **(0.11)**	**0.97^{b}** **(0.09)**	0.84
NBPT	0.15^{a} (0.02)	**0.57^{c}** **(0.21)**	0.16	**0.20^{bc}** **(0.07)**	**0.43^{c}** **(0.08)**	0.24	**0.36^{b}** **(0.06)**	**0.90^{b}** **(0.18)**	**0.74^{b}** **(0.22)**	**0.29^{b}** **(0.04)**	**0.64^{b}** **(0.13)**	**0.87^{c}** **(0.20)**	0.50	**0.45^{b}** **(0.14)**	**0.84^{c}** **(0.16)**	**0.82^{b}** **(0.15)**	0.91
PCU	0.03^{a} (0.01)	0.07^{a} (0.02)	0.03	0.11^{ab} (0.04)	**0.20^{b}** **(0.05)**	0.09	0.25^{ab} (0.06)	**0.68^{b}** **(0.14)**	**0.54^{b}** **(0.09)**	0.25^{ab} (0.03)	**0.60^{b}** **(0.14)**	**0.49^{b}** **(0.08)**	0.86	**0.30^{b}** **(0.06)**	**0.54^{b}** **(0.06)**	**0.71^{b}** **(0.09)**	0.86
Urea	0.16^{a} (0.08)	**0.33^{b}** **(0.08)**	0.09	**0.18^{bc}** **(0.05)**	**0.39^{c}** **(0.09)**	0.14	**0.37^{b}** **(0.10)**	**0.84^{b}** **(0.21)**	**0.66^{b}** **(0.21)**	**0.33^{ab}** **(0.09)**	**0.66^{b}** **(0.17)**	**0.68^{bc}** **(0.09)**	0.87	**0.45^{b}** **(0.11)**	**0.73^{bc}** **(0.17)**	**0.93^{b}** **(0.16)**	0.87

Summer Fertilization

Date	**5/8-5/13**			**6/20-6/30**			**7/31-8/4**			**9/11-9/15**				**10/25-10/27**			
Weeks after fertilization							***6***			***12***				***18***			
Flush	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Control	-	-	-	**-**	**-**	**-**	0.00^{a}	0.00^{a}	0.00^{a}	0.00^{a}	0.00^{a}	0.00^{a}	0.00	0.00^{a}	0.00^{a}	0.00^{a}	0.00
CUF	-	-	-	**-**	**-**	**-**	**0.20^{b}** **(0.05)**	**0.48^{b}** **(0.13)**	**0.50^{b}** **(0.13)**	**0.21^{b}** **(0.03)**	**0.50^{b}** **(0.09)**	**0.45^{b}** **(0.05)**	0.19	**0.32^{b}** **(0.08)**	**0.54^{c}** **(0.11)**	**0.58^{c}** **(0.10)**	0.58
NBPT	-	-	-	**-**	**-**	**-**	**0.22^{b}** **(0.04)**	**0.44^{b}** **(0.09)**	**0.49^{b}** **(0.09)**	**0.35^{b}** **(0.07)**	**0.60^{b}** **(0.12)**	**0.78^{c}** **(0.12)**	0.23	**0.35^{b}** **(0.09)**	**0.50^{bc}** **(0.12)**	**0.74^{c}** **(0.14)**	0.91
PCU	-	-	-	**-**	**-**	**-**	0.14^{ab} (0.05)	0.16^{a} (0.04)	0.16^{a} (0.03)	**0.22^{b}** **(0.03)**	**0.37^{b}** **(0.07)**	**0.43^{b}** **(0.07)**	0.18	0.19^{ab} (0.07)	**0.33^{b}** **(0.05)**	**0.40^{b}** **(0.07)**	0.59
Urea	-	-	-	**-**	**-**	**-**	**0.18^{b}** **(0.05)**	**0.47^{b}** **(0.05)**	**0.44^{b}** **(0.06)**	**0.26^{b}** **(0.05)**	**0.50^{b}** **(0.08)**	**0.65^{c}** **(0.09)**	0.29	**0.65^{c}** **(0.09)**	**0.57^{c}** **(0.07)**	**0.70^{c}** **(0.08)**	0.48

Chapter 4. Understanding the fate of applied fertilizer nitrogen in mid-rotation loblolly pine plantations (*Pinus taeda* L) of the southern United States using stable isotopes

Abstract

The recovery of applied fertilizer nitrogen (N) following surface application of urea and three enhanced efficiency N containing fertilizers (EEFs) were compared in two studies (Study 1, Study 2) at eighteen thinned mid-rotation loblolly pine stands (*Pinus taeda* L.) across the southern United States. All fertilizer treatments were labeled with ^{15}N (~370 permil, 0.5 AP) and applied during two different seasons (spring, summer) in Study 1 in 2011, and a single season (spring) in Study 2 in 2012 to 100 m^2 circular plots. After N fertilization, all ecosystem components were sampled at the end of a single growing season to determine the ecosystem partitioning of fertilizer N. Total fertilizer N recovery for each treatment was determined using a mass balance calculation for individual ecosystem components. Significantly more fertilizer N was recovered for all EEFs compared to urea for both Study 1 and Study 2, although there were no differences among EEFs. There were no differences in fertilizer N recovery between the season of application for Study 1. For Study 1, total fertilizer N recovery ranged from 78.8% to 82.1% for EEFs compared to 64.6% for urea, and for Study 2 total fertilizer N recovery ranged from 77.1% to 79.5% for EEFs compared to 55.5% for urea. Most of the fertilizer N recovered for all treatments was in the loblolly pines or soil. Recovery in loblolly pine for urea was 34.8% for Study 1 and 33.8% for Study 2. For the EEFs, fertilizer N recovery in loblolly pines for Study 1 ranged from 38.5% to 49.9%, and in Study 2 ranged from 33.2% to 42.1%. There was an increasing amount of fertilizer N recovered with EEFs in the soil for both studies (Study 1 =

30.5% to 38.7%, Study 2 = 35.5% to 42.5%) compared to urea (Study 1 = 28.4%, Study 2 = 20.5%). This research highlights increased fertilizer N recovery and ecosystem partitioning of fertilizer N for EEFs compared to urea in southern loblolly pine plantations, potentially increasing fertilizer N use efficiency in these pine plantations.

4.1. Introduction

Loblolly pine (*Pinus taeda* L.) is the most widely planted tree species in the United States (USDA 2012), extending from southeast Oklahoma and east Texas to north Florida and southern New Jersey (Shultz 1997). Loblolly pine plantations are often intensively managed through the planting of improved genetic stock, site preparation, competition control, and fertilization (Fox et al. 2007a). The combined interactions of these treatments often doubles loblolly pine growth, with many stands producing more than 10 m^3 ha^{-1} yr^{-1} (Fox et al. 2007a). One primary constraint limiting growth in many loblolly pine plantations in the South is the deficiency of plant nutrients, specifically nitrogen (N) and phosphorous (P) (Allen 1987, Fox et al. 2007b). Soil N deficiencies are common in the South because larger quantities of N are required by trees compared to other nutrients, and most forest soils are unable to adequately supply plant available N to meet tree demand (Miller 1981, Chapin et al. 1986, Vitousek and Howarth 1991). Low N availability restricts leaf area production which decreases photosynthetic capacity, net primary productivity and hence the growth of the stand (Linder 1987, LeBauer and Treseder 2008).

Tree N uptake requirements increase with stand age (Switzer and Nelson 1972, Wells and Jorgenson 1979, Fox et al. 2007a) and leads to nutrient limitations in loblolly pine plantations when the potential nutrient use of the stand is not provided by the soil nutrient supply (Allen et al. 1990). The ecosystem disturbance associated with stand establishment activities increases soil N availability due to N mineralization from the soil organic matter and plant residues produced by harvesting activities (Vitousek and Matson 1985, Fox et al. 1986). As a stand ages, plant available N declines because of increased N immobilization within the ecosystem (Birk and Vitousek 1986). To ameliorate reduced plant N availability, fertilization with N is required to maintain forest productivity (Miller 1981). On average, southern loblolly pine plantation growth increases 3 $m^3 ha^{-1} yr^{-1}$ up to 8 years following a mid-rotation application of 224 kg ha^{-1} N plus 28 kg ha^{-1} of P (Fox et al. 2007a). These results have led to the annual fertilization of over 500,000 ha of pine plantations in the southern United States (Albaugh et al. 2007). However, certain sites do not respond, or respond negatively, to fertilization (Pritchett and Smith 1975, Martin et al. 1999, Amateis et al. 2000, Carlson et al. 2014). These inconsistent fertilizer trial results have led to a wide range of productivity in pine plantations (Rojas 2005, Fox et al. 2007a).

Despite fertilization improving the growth of most loblolly pine plantations, tree N uptake is generally less than 25% of applied fertilizer N (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), although some studies have reported larger amounts of N uptake (Raison et al. 1990). Explanations for low fertilizer N uptake in field studies include: 1) fertilizer N loss from the system following fertilizer application; 2) variable fertilizer N immobilization in ecosystem components with fluctuating turnover rates; and 3) asynchronous soil supply of fertilizer N with seasonal plant N demand, all which reduce fertilizer N use efficiency (FNUE). As with all agroecosystems, an improved understanding of fertilizer N uptake and ecosystem retention in southern pine plantations is required to enhance FNUE to continue to increase growth and productivity of these systems in the future (Zhang et al. 2015).

To improve FNUE in agroecosystems, slow release (SRN), controlled release (CRN) and stabilized (SNF) N fertilizers were developed (Azeem et al. 2014, Fertilizer Institute 2015). CRN is fertilizer N that is coated or encapsulated to alter the rate, pattern and duration of N release (Chien et al. 2009). SRN are N fertilizer formulations that gradually release fertilizer N slowly as a result of microbial decomposition (Trenkel 1997). SNF is fertilizer N that is treated with chemical compounds that inhibit rapid N transformation to less stable forms (Shaviv 1996, 2005). The SRN, CRN and SNF products can all be broadly categorized as enhanced efficiency fertilizers (EEFs) (AAPFCO 2015). 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 the conversion of the nutrient to forms less susceptible to losses. The N containing EEFs are designed to: 1) increase N uptake by the crop; 2) decrease N leaching; 3) lower toxicity; 4) provide extended N supply; 5) reduce volatilization and/or nitrification losses of N; and 6) lower application cost (Allen 1984, Hauck 1985). Therefore, EEFs can reduce fertilizer N loss and provide greater flexibility for fertilization under a variety of weather conditions and enhance fertilizer N uptake by the crop (Hauck 1985, Goertz 1993, Azeem et al. 2014). Although EEFs were developed to address FNUE issues in agroecosystems, similar issues related to FNUE exist for southern pine plantations. Implementing EEF technology into forest fertilization programs in southern pine plantations may improve FNUE and increase the productivity of these systems.

Pelletized urea (46-0-0) is the primary N fertilizer used in southern pine plantations 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). The initial reaction, urea hydrolysis, is catalyzed by the extracellular enzyme urease which produces ammonium bicarbonate (Conrad 1942, Pettit et al. 1976). Ammonium bicarbonate dissociates into ammonium (NH_4^+) and bicarbonate (HCO_3^-). The bicarbonate consumes hydrogen (H^+), raising the microsite pH surrounding the urea granule. The elevated microsite pH surrounding the urea granules cause ammonium ions (NH_4^+) to dissociate to ammonia (NH_3) that can be volatilized to the atmosphere. High losses of fertilizer N after urea fertilization can occur through 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). Substantial fertilizer N loss after urea fertilization in southern loblolly pine plantations translates to less fertilizer N remaining in the ecosystem, and may lead to less fertilizer N availability for tree N uptake. To reduce NH_3 volatilization, urea application in southern pine plantations usually occurs in the 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). Other N loss pathways may still be significant at this time including denitrification (Shrestha et al. 2014) or leaching (Binkely et al. 1995, Aust and Blinn 2004). Fertilizer N lost immediately following fertilization translates to less fertilizer N remaining in the ecosystem and a lower FNUE (Galloway et al. 2003).

One SNF mechanism to reduce urea N loss is to add urease inhibitors to urea prior to fertilization. Urease inhibitors are substances which temporarily inhibit the hydrolytic action on urea by the urease enzyme, resulting in less urea N lost through NH_3 volatilization (AAPFCO 2015). These products generally reduce urease activity for two to three weeks after fertilizer application (Gowariker and Krishnamurty 2009, Havlin et al. 2014), although under certain conditions effectiveness can be longer (Engel et al. 2011). Urease is abundant in soils and produced by numerous soil organisms and plants (Bremner and Douglas 1971, Bremner and Chai 1986, Antisari et al. 1996, Sanz-Cobena et al. 2008). When urease inhibitors are released to the environment, they bind to the urease enzyme which reduces the activity of the enzyme and provides time for urea to dissolve and move into the soil. Once in the soil, loss via NH_3 volatilization from urea is reduced because the soil buffers the pH. Many compounds inhibit urease activity, but few are chemically stable when added to urea, effective when applied at low concentrations, and are non-toxic when applied in the environment (Hauck 1985, Azeem et al. 2014). The most widely used urease inhibitor is N-(n-Butyl) triphosphoric triamide (NBPT) (Havlin et al. 2014). Urea impregnated with NBPT can reduce losses of fertilizer N from NH_3 volatilization significantly compared to untreated urea (Engel et al. 2011, Raymond et al. 2016) and may translate to improved productivity for desired species (Engel et al. 2011).

The CRN products generally have a urea granule base which is initially coated with a compound (i.e. sulfur (S), boron, (B), copper (Cu)) and additionally sealed with a wax-like substance to fill imperfections (Chien et al. 2009). Fertilizer N release from CRN depends on the thickness and coating quality (Allen 1984). As the coating degrades in the environment, cracks develop in the coating allowing moisture to enter the product, and urea dissolution is initiated. Urea N release to the environment is slowed by the coating compared to uncoated urea. The substrate coating of CRN may reduce urease activity, and CRNs may also be impregnated with urease or nitrification inhibitors to slow loss mechanisms as urea N is released to the environment. Additionally, CRN products may include other nutrients in the coatings to address additional nutrient deficiencies.

An alternative CRN approach encapsulates fertilizer N (generally urea) in a semi-permeable polymer coating. The coatings have small pores which allow water to enter the capsules. As water enters the CRN product, urea dissolution occurs, the capsule expands and the resulting internal pressure of the capsule forces fertilizer N solution into the environment through diffusion (Shaviv 2005). Because of the low mass of the polymer coatings, these products do not significantly reduce the N percentage in the product as may occur with the aforementioned CRN products (Chien et al. 2009). Release patters for polymer coated CRN products are related to the type of coating, soil moisture, temperature, relative humidity, precipitation, pH, and microbial activity (Christianson 1988, Shoji et. al. 2001, Shaviv 2005).

Although tree growth is often N limited, forest soils contain large quantities of N, most of which is unavailable for plant uptake (Fisher and Binkley 2000). Because of the large amount of N present in forest soils, ranging from 2 to 7 Mg ha^{-1}, it is difficult to distinguish changes in soil N following a single or multiple applications of fertilizer N, which typically adds only 150 kg ha^{-1} to 250 kg ha^{-1} of N (Fisher and Binkley 2000, Flint 2007, Kiser and Fox 2014). However, fertilizers enriched with the stable isotope ^{15}N can be used to trace the fate of fertilizer N in the ecosystem to improve our understanding of mechanisms and cycling of applied N (Knowles and Blackburn 1993, Nadelhoffer and Fry 1994, Robinson 2001, Dawson et al. 2002).

Numerous ^{15}N tracer studies have been conducted in agroecosystems to improve our understanding of fertilizer N cycling in crop systems (Hauck and Bystrom 1971). Although research using ^{15}N to trace the fate of fertilizer N has been conducted in forest, most recent work with ^{15}N in forest ecosystems has focused on the natural cycling of N (Currie and Nadelhoffer 1999, Dinkelmeyer et al. 2003, Nadelhoffer et al. 2004) and/or the potential negative effects of increased N deposition from industrialization (Tietema et al. 1998, Templar et al. 2012) in natural systems. Research using ^{15}N enriched fertilizers to study FNUE in intensively managed forest plantations have been surprisingly limited (Hauck 1968, Mead and Pritchett 1975, Melin et al. 1983, Chang et al. 1996, Bubb et al. 1999, Werner 2014).

Using ^{15}N enriched fertilizers to study fertilizer N uptake and ecosystem fate in southern pine plantations will improve our fundamental understanding of the fate of fertilizer N in these systems. Combined with enhanced efficiency fertilizer technology, this improved understanding will increase FNUE efficiency in southern pine plantations into the future. The statistical hypotheses for this research are:

H_O1: There are no differences in the total fertilizer N recovery between urea and the enhanced efficiency N containing fertilizers.

H_O2: There are no differences in total fertilizer N recovery between urea and the enhanced efficiency N containing fertilizers between a spring and summer fertilizer application.

Ho3: There are no differences in the total fertilizer N recovery between urea and the enhanced efficiency N containing fertilizers for each primary ecosystem component.

4.2. Methods

4.2.1. Experimental Design and Site Description

Two separate experiments (Study 1, Study 2) were designed to test differences of total fertilizer N recovery among fertilizer treatments using urea and three EEFs in southern loblolly pine plantations. Study 1 (2011) used a split plot complete block design to test differences between five fertilizer treatments (main plots) at two different application seasons (split plot). Five treatments (CUF, NBPT, PCU, urea, control) were applied at six sites during 2011 in two separate seasons (spring: March-April; summer: June) in mid-rotation loblolly pine plantations across the southern United States (Figure 4.1). The five fertilizer treatments were the main plots with single replication at each individual site. Each individual site served as a block. The split plot was the application season (spring vs. summer). Study 2 (2012) expanded the geographic scope of Study 1 and evaluated the same five treatments at 12 additional sites in one application season (spring) (Figure 4.1). Study 2 was a complete block design where fertilizer treatments served as the treatment plots with single replication at each individual site, and each individual site served as a block. Selected physical site characteristics of the sites used in Study 1 and Study 2 are described in Tables 4.1 and 4.2, and selected soil chemical and physical data is detailed in Table 4.3.

4.2.2. Fertilizer Treatments

The five treatments were used in both Study 1 and Study 2. The five treatments 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 where no fertilizer N was added. Urea (46-0-0) was the conventional N fertilizer used because of its common use in southern forests. The enhanced efficiency fertilizers (EEFs) tested in this study were developed to reduce NH_3 volatilization and release fertilizer N slowly to the environment. In the NBPT treatment, urea (46-0-0) granules were impregnated with N-(n-butyl) thiophosphoric triamide at a rate of 26.7% by weight to inhibit urease activity. In the CUF treatment (39-9-0), urea granules were impregnated with NBPT and coated with an aqueous binder containing a boron and copper sulfate solution to slow the release of N to the environment. A coating of monoammonium phosphate was then added to provide P. The PCU (44-0-0) treatment had a polymer coating covering urea granules with pore openings designed to slowly release N (~80%) over a 120 day period. The application rate for all treatments was equivalent to 224 kg N ha^{-1}. Because the CUF treatment contained P in the monoammonium phosphate coating, P was added to the other fertilizer treatment at the equivalent rate of 28 kg P ha^{-1} using triple superphosphate (TSP). The urea for all treatments was enriched with the stable isotope ^{15}N (~370‰, 0.5 AP) (Nadelhoffer and Frye 1994). All individual fertilizer treatments for both studies were hand broadcast applied to individual $100 m^2$ circular plots described below.

4.2.3. Experimental Method- Field Sampling – Pre-treatment

Five 100 m^2 circular plots were installed at each site for each application season for both studies in areas with similar stand, soils and landscape characteristics and a minimum distance of 30.5 m from individual plots (Figure 4.2). The plot center was located equidistant between two co-dominant loblolly pine trees with similar height (HT) and diameter (DBH). After plot installation and prior to treatment application, the HT and DBH was measured for all trees greater than 2.54 cm DBH including coniferous and deciduous species. Percent cover

(PC) was ocular estimated for each of the following understory strata: 1) sapling; 2) shrub; 3) vine; and 4) herbaceous. Sapling and shrub strata were identified by species. Foliage, fine (< 2 cm) and coarse (> 2 cm) branches were sampled from the two central crop trees from the upper 1/3rd of the canopy. Foliage (if present) and branches were sampled from individual species in the sapling and shrub strata. Vines and herbaceous plants were sampled by composite for respective strata. The forest floor, or O horizon (Oi + Oe + Oa), was sampled using a 0.07 m^2 circular frame at four randomly selected areas and composited. The mineral soil was sampled with a push-tube auger at 8 random locations for two depths (0-15 cm, 15-30 cm) and composited by depth. Root samples of the mineral soil was composited from four random locations with a bulk density sample corer (AMS Inc.) up to a 30 cm depth. Two circular litterfall traps were randomly placed in each plot to sample litterfall after N fertlization. Bulk density samples of the 0-15 cm and 15-30 cm depths of mineral soil was determined for each individual plot using a bulk density sample corer (AMS Inc.).

4.2.4. Experimental Method- Field Sampling – Post-treatment

For both studies, the individual fertilizer N treatments were randomly applied to the individual 100m^2 circular plots on a single day for each application season at each individual site. The spring application date for both studies was between March 26 to April 8, and summer application in Study 1 was from June 18 to June 30. All plots were resampled between November 1st and March 31st at the end of the growing season in both studies using the same procedures as the pre-treatment sampling. In addition, litterfall was collected from traps and composited in each plot. One central crop tree per plot was harvested and separated into the following components: 1) foliage; 2) fine branches; 3) coarse branches; 4) dead branches; and 5) stem. Each biomass component was weighed in the field to obtain green weights. Subsamples of each biomass component were returned to the lab and dried to determine moisture content which was used to convert biomass to a dry weight basis. Stem samples (cookies) were cut at DBH, the base of the live crown (HLC), and at mid-crown (HMC). The sapling stratum, if present, was divided in 2.54 cm individual species diameter classes, and one tree from each diameter class was harvested and weighed in the field for green weights. Each plot was divided in 32 equal wedge sections, and one section was randomly selected to sample the entire shrub, vine and herbaceous strata. Shrubs were divided by species whereas vine and herbaceous strata were composited in their respective strata. Samples from each component were put in separate labeled paper lawn and grocery bags and returned to the laboratory for processing.

4.2.5. Experimental Method - Laboratory Processing

All biomass samples were dried in a forced air oven at 60°C until samples achieved a constant weight. Litterfall was separated into the subcategories of pine needles, deciduous leaves, fine branches, coarse branches, bark and unidentifiable litterfall. The stem cookies collected at DBH were subsampled for bark, wood in the current year of growth when treatment was applied (CGR), and all wood composited from all the previous growth prior to fertilizer treatment (PGR). Roots were washed from mineral soil with an elutriation system and separated into fine (<2 mm) and coarse (>2 mm) and oven dried. The O horizon samples were sieved through a 6 mm sieve and mineral soil was sieved through a 2 mm sieve. Soil chemical data presented in Table 4.3 was determined through the Mehlich I procedure at the Virginia Tech Soil Testing Laboratory.

All individual plant material, litterfall and O horizon samples were initially coarse ground with a Wiley Mill to pass a 2 mm sieve. Individual samples were then ground to a fine powder in a ball mill (Retsch® Mixer Mill MM 200, Haan, Germany) with all individual organic substrate samples ball milled for 1 minute at 25 revolutions per second (rps). 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). Individual samples were analyzed to determine the $^{15}N/^{14}N$ isotope ratio, total N and total C on a coupled elemental analysis-isotope ratio mass spectrometer (IsoPrime 100 EA-IRMS, Isoprime© Ltd., Manchester, UK) at the Forest Soils and Plant Nutrition Laboratory at Virginia Polytechnic Institute and State University (Virginia Tech) between January 2012 and May 2015. All instrumentation used during the entire process, from initial coarse grinding to weighing on the microbalance, was cleaned with an ethanol solution and allowed to dry after each individual sample was processed to reduce cross sample contamination.

4.2.6. Calculation of Fertilizer N Recovery

The amount of total N in each component derived from the added fertilizer labeled with ^{15}N was calculated using a mass balance tracer technique that compared individual ecosystem component ^{15}N prior to and 1 year after N fertilization (Powlson and Barraclough 1993, Nadelhoffer and Fry 1994, Nadelhoffer et al. 1995).

Equation [4.1] calculated the individual system component N percent that originated from the fertilizer N:

[4.1] %N from fertilizer in individual ecosystem component =

$$((\delta^{15}N_{final} - \delta^{15}N_{initial}) / (\delta^{15}N_{labeled} - \delta^{15}N_{initial})) * (100)$$

Equations [4.2] to [4.5] determined the percent recovery of fertilizer N for individual ecosystem components:

[4.2] % ^{15}N from labeled treatment in individual ecosystem component =

$$((\delta^{15}N_{sample} - \delta^{15}N_{pretreatment}) / (\delta^{15}N_{fertlizer} - \delta^{15}N_{pretreatment})) * (100)$$

[4.3] Total N (g) = (Component dry weight * %N) / (100)

[4.4] Fertilizer N in individual ecosystem components (mg)

((Equation 4.3) * (Equation 4.4.) / (100) *1000)

[4.5] % N originated from fertilizer in individual ecosystem component =

[(Equation 4.4) / (amount fertilizer applied)] *100

After individual ecosystem component fertilizer N recovery was determined, individual component fertilizer N recovery was summed per plot to calculate total fertilizer N recovery for the plot. The fertilizer N recovery value for the individual loblolly pine sampled in each plot was multiplied by the number of loblolly pine trees in each individual plot to obtain the total fertilizer N recovery for loblolly pine trees on an individual plot basis. The difference between the amount of fertilizer N applied to the plot and the amount of fertilizer N recovered after 1 growing season was considered lost from the system.

4.2.7. Statistical Analysis

Total fertilizer N recovery, expressed as a percentage of fertilizer N applied, was analyzed using a general linear model (GLM) analysis of variance (ANOVA) with Minitab® 17 (Minitab Inc., State College, Pennsylvania, USA). Percent data not normally distributed was arcsin transformed prior to analysis. For Study 1, 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. Individual ecosystem components were analyzed using a GLM ANOVA except the analysis of individual mineral soil depth increments (0-15 cm, 15-30cm) which were analyzed as a repeated measures ANOVA. For Study 2, percent fertilizer recovery (%) was the response variable for the model, fertilizer treatment (CUF, NBPT, PCU, urea, control) was the fixed effect, and site was a random effect in the analysis. Individual ecosystem components were analyzed using a GLM ANOVA except the analysis of individual mineral soil depth increments (0-15 cm, 15-30cm) which were analyzed as a repeated measures ANOVA. To determine if site factors (physiographic region, soil drainage, soil texture, etc.) were correlated with total fertilizer recovery, a GLM ANOVA was also used for analysis. The percent fertilizer recovery (%) was the response variable for the model, site factors (physiographic region, soil drainage, soil texture, etc.) were fixed effects, and site was a random effect in the analysis. 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.

4.3. Results

4.3.1. Study 1 – 2011 Application – Spring vs. Summer

Nitrogen concentrations (g kg^{-1}) in the foliage did not differ between application season but were greater overall in the fertilized treatments (Table 4.4; *In Appendix Figure A.4.1., Figure A.4.2., Table A.4.1. to Table A.4.7., Table A.4.8*) except for the CUF treatment. Nitrogen concentrations in the foliage increased from 12.3 g kg^{-1} in the control to 14.1 g kg^{-1} in CUF (NS – not significant) and 14.4 g kg^{-1} to 15.0 g kg^{-1} in the other 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 (4.9 g kg^{-1}) with no differences among the EEFs. There were no differences in N concentration between the control and the fertilizer treatments for any other tree or soil component (Table 4.4) except in litterfall for the urea treatment (5.1 g kg^{-1}), and coarse roots in the urea (6.8 g kg^{-1}) and PCU (7.0 g kg^{-1}) treatments compared to the unfertilized control (litterfall = 7.9 g kg^{-1}, coarse roots = 8.8 kg g^{-1}) plots.

The ^{15}N (‰) signature in all tree and soil components sampled was greater for the fertilizer treatments compared to the control (Table 4.4). There were no differences in the ^{15}N values among the fertilizer treatments or between sampling dates for any components of the loblolly pine trees. The ^{15}N values were greatest in the foliage (83.9‰ to 97.1‰), fine branches (74.3‰ to 86.9‰) and wood formed in the current growing season following fertilization (59.3‰ to 73.8‰). The ^{15}N 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 ^{15}N (30.7‰ to 36.6‰), whereas the ^{15}N was lowest in bark (15.0‰ to 18.9‰), fine roots (14.0‰ to 24.1‰) and coarse roots (8.5‰ to 15.8‰). In the soil, the ^{15}N was greatest in the O horizon (65.0‰ to 89.6‰), comparable to the ^{15}N values in the foliage. Conversely, the ^{15}N signature in the surface 0-15 cm mineral soil in fertilized treatments ranged from 10.2‰ to 18.0‰ compared to the control (3.70‰). In the 15-30 cm mineral soil the ^{15}N values ranged from 8.5‰ to 11.9‰, with only the CUF treatment being significantly greater than the control (5.44‰).

There were no differences in fertilizer N recovery in the individual components of loblolly pine among the different fertilizer treatments (Table 4.4). Fertilizer N recovery in the 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% in 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 11.1%, but less in the coarse roots which averaged 1.7%. Fertilizer N recovered in the fresh litterfall was low, averaging 0.4%. In the soil, fertilizer N recovery in the O horizon averaged 2.8% among the fertilizer treatments. In contrast, fertilizer N recovery in the surface 0-15 cm of the mineral soil was high, ranging from 13.4% in the urea treatment to 31.2% in the PCU treatment. Fertilizer N recover in the mineral soil from 15-30 cm was lower, ranging from 5.1% to 8.3% among the fertilizer treatments.

There were no differences between the total ecosystem fertilizer N recovery between the two seasons of application (Table 4.5). Total fertilizer N recovery differed among the N fertilizer treatments for the combined spring and summer data with greater recovery for all EEFs (CUF = 76.4%, NBPT = 80.6%, PCU = 76.2%) compared to urea (54.6%) (Figure 4.3) but no differences among the EEFs. There were slight difference (NS) in the total fertilizer N recovered in the tree, determined as the sum of N recovery in all aboveground and belowground portions of the tree, between the treatments (CUF = 37.1%, NBPT = 44.7%, PCU = 33.2%, urea = 33.8%) (Table 4.5, Table 4.8). Total fertilizer N recovery in the soil was lower in the urea treatment (20.5%) compared to PCU (42.5%) and CUF (38.9%) but there were no differences with NBPT (35.5%).

4.3.2. Study 2 – 2012 Application – Spring

Nitrogen concentrations (g kg^{-1}) in the foliage increased (NS) from 12.5 g kg^{-1} in the control to between 13.2 g kg^{-1} and 14.2 g kg^{-1} for fertilizer treatments (Table 4.6; *In Appendix Table A.4.7.*). There was a difference in the N concentrations of fine branches between the control (5.1 g kg^{-1}) and CUF (7.3 g kg^{-1}) the N concentration of bark between the control (2.1 g kg^{-1}) and CUF (2.9 g kg^{-1}), and for the N concentration in the wood produced for the year of fertilization between the control (1.8 g kg^{-1}) and CUF (2.9 g kg^{-1}). There were no other differences in N concentrations between the control and the fertilizer treatments, or among fertilizer treatments, for any other tree, soil or other ecosystem component (Table 4.6).

The ^{15}N (‰) signature in all of the tree and soil components sampled was greater in the fertilizer treatments compared to the control, although there were no differences in the ^{15}N signatures among the individual fertilizer treatments (Table 4.6). For the loblolly pine trees, the ^{15}N was greatest in the foliage (101.6‰ to 126.1‰), fine branches (98.7‰ to 111.8‰), and wood produced in the current year after fertilization (71.9‰ to 88.1‰). The ^{15}N in the current year litterfall was lower than foliage (48.6‰ to 55.8‰). Wood formed in years prior to fertilization had low ^{15}N (34.8‰ to 40.3‰), whereas the ^{15}N values were lowest in bark (20.5‰ to 22.5‰), fine roots (31.2‰ to 36.1‰) and coarse roots (13.9‰ to 19.0‰). In the understory, ^{15}N values were high in both the sapling (59.6‰ to 83.3‰) and shrub (55.4‰ to 112.0‰) strata compared to the control (-0.24‰, -3.00‰ respectively). For the soil, there were no differences among fertilizer treatments. The ^{15}N was greatest in the O horizon for all fertilizer treatments (55.2‰ to 91.1‰). The ^{15}N signature for fertilizer treatments in the surface 0-15 cm mineral soil ranged between 15.9‰ to 22.3‰ compared to the control (3.27‰), whereas in the 15-30 cm mineral soil the ^{15}N values ranged between 11.7‰ to 16.3‰ compared to 5.38‰ for the control (Table 4.6).

Differences did exist in fertilizer N recovery in the individual components of loblolly pine among the different fertilizer treatments (Table 4.6). Fertilizer N recovery for loblolly pine trees for all treatments was greatest in the foliage, ranging from 8.1% to 14.8% of the N applied. More fertilizer N was recovered for foliage for NBPT (14.8%) compared to PCU (8.1%) and fine branches (NBPT = 4.1%, PCU = 2.6%), with no other differences among treatments for any other tree component. Fertilizer N recovery in the other components of the tree was less, averaging 2.9% for coarse branches, 0.6% for bark, 2.2% for wood produced the year of fertilization, and 4.0% for wood produced prior to fertilization (Table 4.6). There were differences between fertilizer N recovery in fine roots between CUF (19.2%) and urea (10.8%) but not for coarse roots (1.7% to 3.8%). The fertilizer N recovered in the litterfall ranged from 1.4% to 1.8% with no differences among treatments. Fertilizer N recovery in the soil ranged from 1.2% to 3.8% for the O horizon among treatments. The fertilizer N recovery in the surface 0-15 cm of the mineral soil was high, ranging from 15.9% in the urea treatment to 24.2% in the PCU treatment. There were no differences in fertilizer N recovery for the 15-30 cm mineral soil which ranged from 5.4% to 11.3% among the fertilizer treatments.

Total fertilizer N recovery differed among the fertilizer N treatments (Figure 4.4). There was greater fertilizer N recovery in the EEFs (CUF 82.2%, NBPT 83.4%, PCU 80.7%) compared to urea (66.2%) but no differences among the EEFs. There was a difference in the total fertilizer N recovered in the tree, determined as the sum of N recovery in all aboveground and belowground portions of the tree. For the tree, fertilizer N recovery was greater in both CUF (47.0%) and NBPT (49.8%) compared to both PCU (38.5%) and urea (34.8%), with no differences between CUF and NBPT or PCU and urea. For soil, PCU (38.7%) was greater than urea (28.4%) (Table 4.7 and Table 4.8). For soil, determined as the sum of N recovery in all both organic and mineral soil components, slight differences (NS) occurred among treatments (CUF = 32.8%, NBPT = 30.6%, PCU = 38.7%, urea = 28.4%).

4.4. Discussion

Our study focused on assessing the differences in N uptake and ecosystem retention following N fertilization in loblolly pine plantations of the southern United States. These differences were assessed by determination of fertilizer N recovery among four different N containing fertilizers and between different application seasons. By installing a study across the natural range of loblolly pine we hoped to improve the understanding of fertilizer N uptake efficiency in these systems through a regional synthesis, compared to a more detailed analysis at a single site. This study tested the primary hypotheses: 1) whether differences existed in the total fertilizer N recovery among conventional and enhanced efficiency fertilizers; 2) if application season caused a difference in fertilizer N recovery among individual fertilizer treatments; and 3) whether differences existed in the ecosystem partitioning of fertilizer N among conventional and enhanced efficiency fertilizers. Our overall objective was to improve the fertilizer N use efficiency of southern loblolly pine plantations.

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 studies. Although significantly more fertilizer N was recovered for enhanced efficiency fertilizers, differences generally did not exist among individual enhanced efficiency fertilizers. The lower fertilizer N recovery for urea may be due to larger NH_3 volatilization losses in the urea treatment compared to the EEFs.

In a companion study, Raymond et al. (2016) used sites from Study 1 and found NH_3 volatilization losses following fertilization for urea ranged from 26% to 49% compared to NH_3 volatilization losses following fertilization with the same EEFs used in this research ranged from 4% to 26%. Other studies have also reported large NH_3 volatilization losses following fertilization with urea (Kissel et al. 2004, Zerpa and Fox 2011). Elliot and Fox (2014) found NH_3 volatilization losses were approximately 50% less following fertilization in loblolly 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 for both Study 1 and Study 2, the percent recovery of the fertilizer N ranged 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.

Weather variables are often highly correlated with large NH_3 volatilization losses from urea, and include relative humidity exceeding a calculated critical relative humidity (Elliot and Fox 2014, Raymond et al. 2016), high temperatures (Koelliker and Kissel 1988), high surficial wind speed (Kissel et al. 2004), and low precipitation (Kissel et al. 2004). In the Raymond et al. (2016) companion study, days when the calculated critical relative humidity exceeded the relative humidity was correlated with high NH_3 volatilization losses for urea but not for the EEFs. Site edaphic factors, such as the presence of an organic horizon (forest floor) are also correlated with NH_3 volatilization losses for urea application in forested ecosystems (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011). All sites for both Study 1 and Study 2 had an O horizon of variable thickness and had a potential influence on NH_3 volatilization.

Although NH_3 volatilization loss appears 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 the sample for ^{15}N. If this loss pathway was important large ^{15}N signatures would be present and the percent fertilizer N recovery would be significantly different among treatments and the control, which did not occur. In the 15-30 cm mineral soil, fertilizer N recovery ranged from 5.1% to 8.3% for Study 1 and 5.4% to 11.3% in Study 2. These results indicate that although some fertilizer N was moving deeper in soil profiles, the percent loss for leaching was a minor loss pathway one year after fertilization. Another potential loss pathway, denitrification, was not measured. Recent work examining denitrification on similar sites found applied N losses through this pathway were low during spring and summer months (Shrestha et al. 2014), and is typically considered a minor loss pathway in aerobic forest soils (Bowden et al. 1991). 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), and this result indicates denitrification is likely a minor loss mechanism. Although losses of fertilizer N through leaching and denitrification may have occurred, we concluded that these loss pathways were minor in this study and NH_3 volatilization was the primary loss pathway for fertilizer N from these systems.

The second hypothesis that no differences occurred in fertilizer N recovery among treatments between a spring and summer application was rejected. There was a higher percentage of fertilizer N recovery in the ecosystem for urea in spring compared to summer, whereas there were no differences between seasons for the EEFs. As previously stated, a primary loss mechanism for applied urea N in loblolly pine plantations is NH_3 volatilization (Raymond et al. 2016), with fertilizer N loss generally correlated with weather variables (Koelliker and Kissel 1988, Kissel et al. 2004, Elliot and Fox 2014, Raymond et al. 2016) and their interaction with edaphic factors (Cabrera et al. 2005, Kissel et al. 2009, Zerpa and Fox 2011). High losses of fertilizer N from urea can occur regardless of application season (Raymond et al. 2016). Although losses are generally higher in the summer under humid, hotter conditions they can also be high in the winter or spring and highly dependent on weather conditions at the time of fertilizer N application (Koelliker and Kissel 1988, Kissel et al. 2004, Elliot and Fox 2014). Losses of fertilizer N were lower for EEFs compared to urea (Raymond et al. 2016) for both seasons in Study 1 which translated to similar N recovery rates for each EEF treatment between seasons. A likely explanation for the differences in results between these two studies was Raymond et al. (2016) excluded roots to specifically focus on loss mechanisms of fertilizer N. This study focused on ecosystem cycling of fertilizer N and did not exclude roots for plant uptake. It is likely that plant uptake of fertilizer N was rapid due to the presence of roots in the O horizon and upper mineral soil horizons (Parker and Van Lear 1995, Adegbidi et al. 2004) and is a partial explanation of the small increase in fertilizer N recovery for both seasons in Study 1 for urea.

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, O horizon, etc.) were analyzed separately, differences did occur among fertilizer treatments for individual ecosystem.

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 (Nadelhoffer et al. 1995, 2004, Tietema et al. 1998, Burke et al. 2002, Blanes et al. 2002). Yet the results for many of these studies indicate higher amounts of applied N remaining in the soil and less N uptake for trees. Studies specific to southern loblolly pine plantations indicate generally low fertilizer N uptake (<25%) by loblolly 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). Yet most studies in southern loblolly pine used urea as the fertilizer N source. In this study, loblolly pine tree fertilizer N uptake of urea was 35% compared to 33% to 50% for all EEFs. A clear trend exists for increased recovery of fertilizer N in loblolly pine trees for EEFs compared to urea. Additionally, an increasing amount of fertilizer N was recovered with EEFs in the soil for both studies (Study 1 = 30.5% to 38.7%, Study 2 = 35.5% to 42.5%) compared to urea (Study 1 = 28.4%, Study 2 = 20.5%). These differences in the partitioning of fertilizer N among ecosystem components between EEFs and urea is likely important in the long term cycling of fertilizer N of loblolly pine plantations. Additional research will be required to determine if this increase in fertilizer N retention for EEFs compared to urea translates to an increase in FNUE for southern loblolly pine plantations.

Nitrogen containing fertilizers enriched with ^{15}N can be used to integrate processes, mechanisms and pathways of applied N in terrestrial ecosystems (Knowles and Blackburn 1993, Robinson 2001, Sulzman 2007) by improving the resolution of the amount of fertilizer

N partitioned in ecosystem components. Fertilization studies in southern loblolly pine plantations (Mead and Pritchett 1975, Albaugh et al. 1998, 2004, Jokela and Martin 2000, Vogel and Jokela 2011, Carlson et al. 2014) have improved our understanding of loblolly pine fertilizer response and provided a foundation in understanding the mechanisms and pathways of applied N cycling in these systems. Yet these studies have generally been unable to distinguish N originating from fertilizers compared to naturally existing N (Johnson 1992, Fisher and Binkley 2000, Flint 2007, Kiser and Fox 2012). Differences in soil N between control and fertilized plots in long term annual N fertilization experiments in loblolly pine plantations is generally not detectable (Kiser and Fox 2012, Villanueva 2015), but using fertilizers enriched with stable isotopes can improve the detection and resolution of ecosystem partitioning and dynamics of N origination from fertilizers.

A primary benefit of this research has been the refinement of understanding ecosystem fate of fertilizer N by using stable isotopes in mid-rotation southern loblolly pine stands across an extensive geographic area. The results from this study encompass the entire region where loblolly pine plantations are operationally fertilized and intensively managed, and incorporates a large range of edaphic, climatic and stand variables. The results from this study integrate factors across this entire region, and combined with other fertilizer N studies in forest ecosystems focused on a smaller region or single site, improve the fundamental understanding of the ecosystem partitioning of fertilizer N in southern loblolly pine plantations.

4.5. Conclusions

The primary results for this research were: 1) significantly more fertilizer N was recovered from the ecosystem for all enhanced efficiency fertilizers compared to urea; 2) differences occurred in the ecosystem partitioning of fertilizer N among treatments; 3) no differences occurred in the ecosystem partitioning of fertilizer N among treatments between seasonal fertilizer application; and 4) ecosystem fertilizer N recovery was independent of edaphic, climatic or stand variables. The reduction of fertilizer N loss using enhanced efficiency fertilizers compared to urea, regardless of the season of application, translates to an increase in fertilizer N that remains in the system that is available for plant uptake. This finding resulted in an increase in fertilizer nitrogen use efficiency for the enhanced efficiency fertilizers compared to urea. Additional retention of fertilizer N in the soil for enhanced efficiency fertilizers may also increase fertilizer nitrogen use efficiency into the stand rotation if this fertilizer N becomes available to the plants, but additional research will be required for determination.

These findings will help improve and refine management of southern loblolly pine plantations. If economically viable, the use of enhanced efficiency fertilizers will enable forest managers to apply fertilizer N during the spring or summer, providing a potentially more synchronous timing for fertilizer application to seasonal plant demand. Overall, this research provides a biological foundation for the effectiveness of using enhanced efficiency fertilizers over the entire range where loblolly pine plantations are operationally viable in the southern United States to improve fertilizer nitrogen use efficiency.

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Figures

Figure 4.1. *Location map of loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N following the application of urea or enhanced efficiency N fertilizers enriched with* ^{15}*N.*

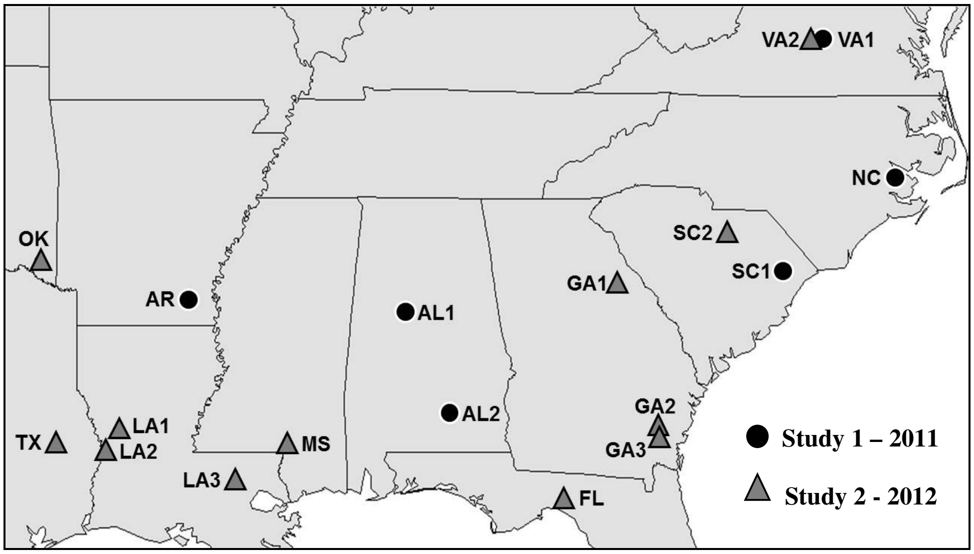


Figure 4.2. *Plot sampling schematic for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N following application of urea or enhanced efficiency N fertilizers enriched with* ^{15}*N. Boxes below each category (crop tree, understory, soils, litterfall) represent ecosystem components sampled.*

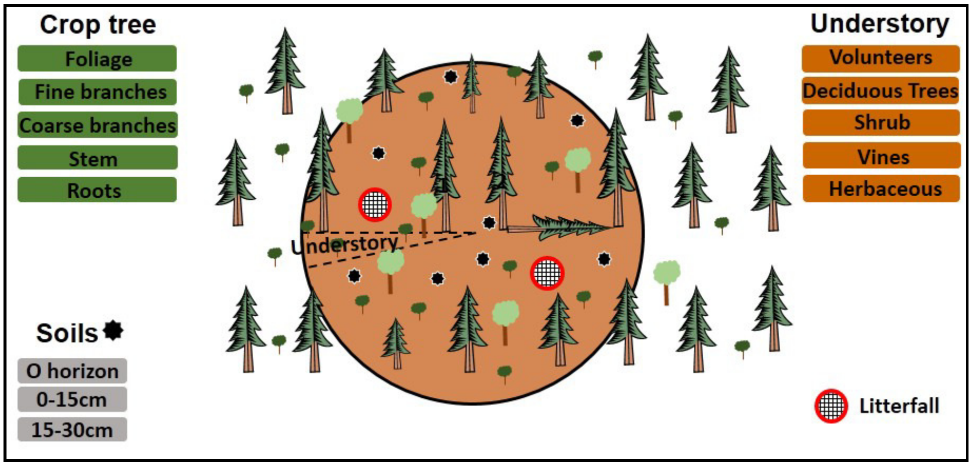


Figure 4.3. *The total fertilizer N recovery (% of fertilizer N applied) of the major ecosystem components (tree - loblolly pine aboveground + belowground biomass, litterfall, and soil (O horizon + mineral soil- 0 to 30cm)) for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N of urea or enhanced efficiency N fertilizers enriched with* ^{15}*N. Data represents combined fertilizer application dates for Study 1 for spring (March-April 2011) and summer (June 2011). Study 1 did not have a significant understory category as in Study 2. Different letters represent significant differences at α = 0.05. Different letter fonts represent comparisons among treatments between same ecosystem components (soil, litterfall, tree). The N application rate was 224 kg N ha*$^{-1}$*. N =10.*

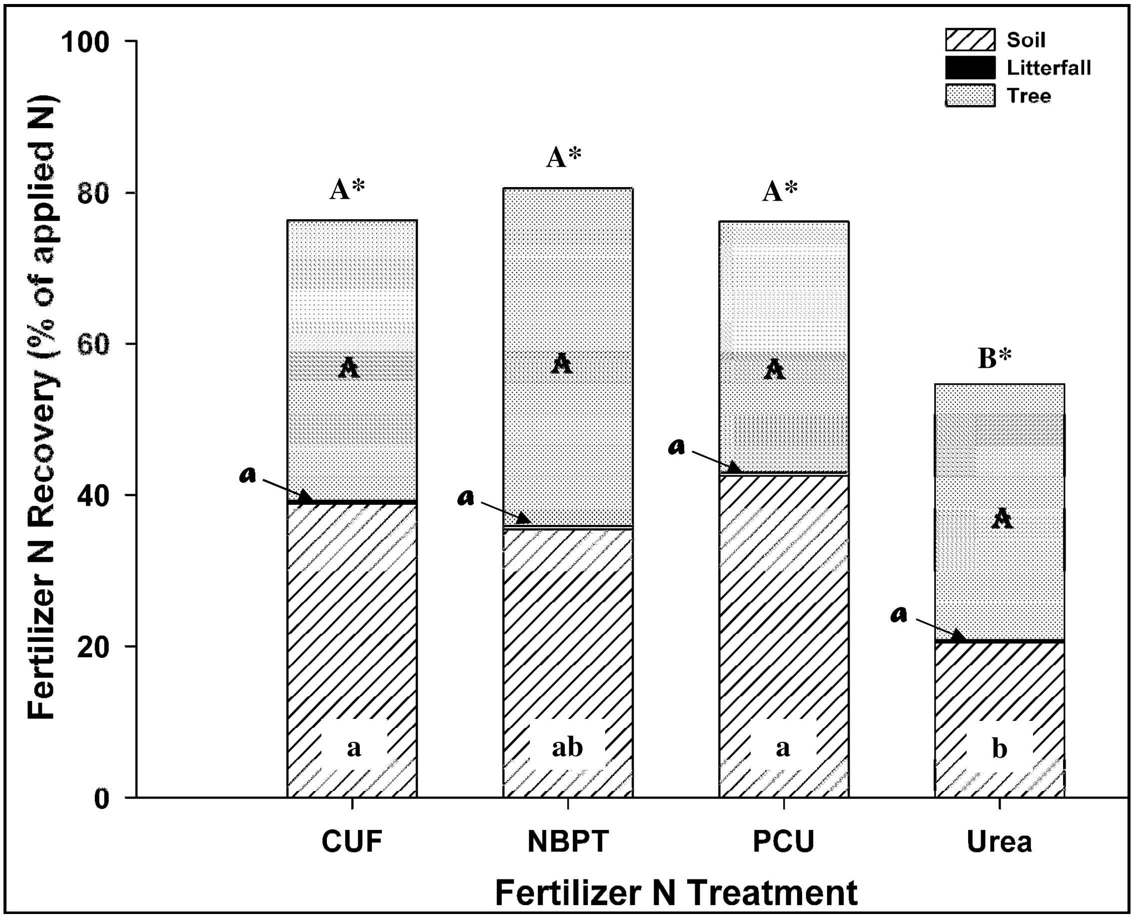
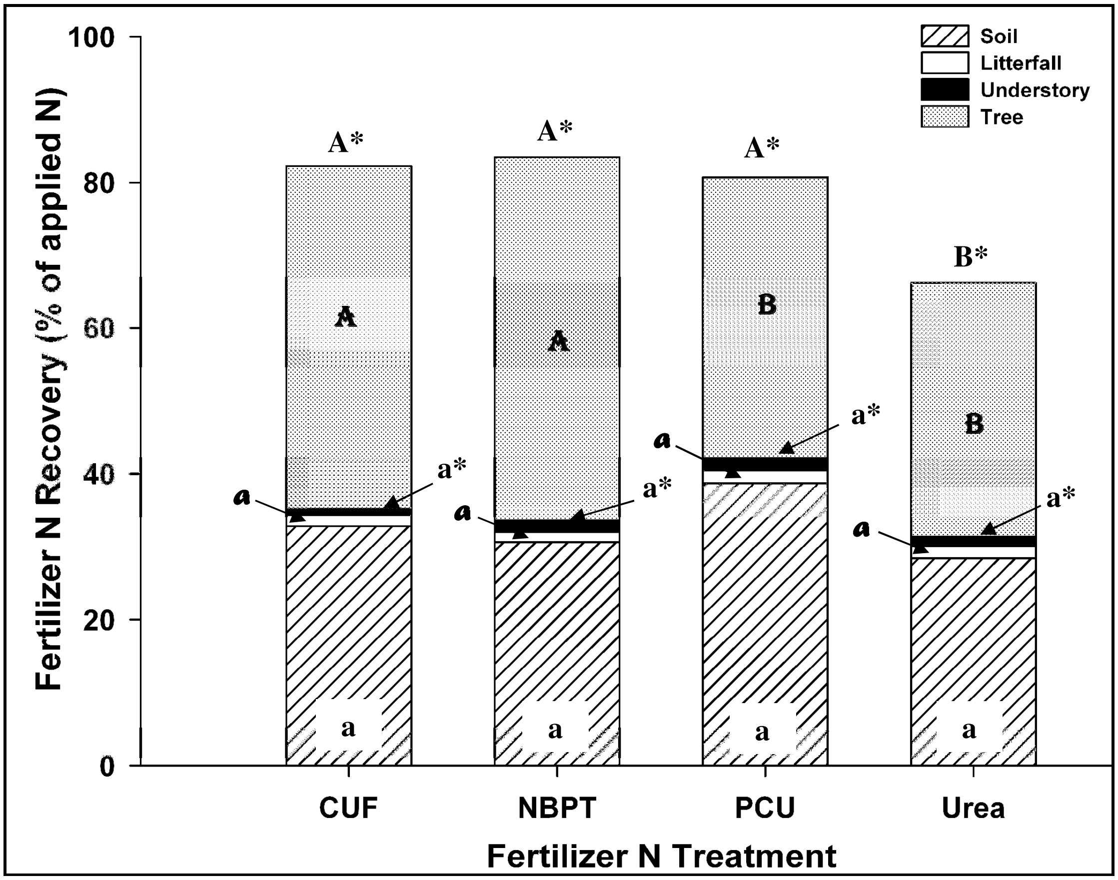


Figure 4.4. *The total fertilizer N recovery (% of fertilizer N applied) of the major ecosystem components (Tree - loblolly pine aboveground + belowground biomass, understory, litterfall, and soil (O horizon + mineral soil- 0 to 30cm)) for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N of urea or enhanced efficiency N fertilizers enriched with ^{15}N. Data represents fertilizer application dates for Study 2 for spring (March-April 2012). Different letters represent significant differences at α = 0.05. Different letter fonts represent comparisons among treatments between same ecosystem components (soil, litterfall, understory, tree). The N application rate was 224 kg N ha^{-1}. N = 12.*



Tables

Table 4.1. *Selected climate and physical characteristics of loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N following application of urea or enhanced efficiency N fertilizers enriched with* ^{15}N*. MAP = Mean annual precipitation. MAT = Mean annual temperature.*

Site	Latitude	Longitude	Altitude (m)	MAP (cm)	MAT (°C)	Physiographic Region	Soil Taxonomic Class	Soil Series	Soil Drainage Class
Study 1									
VA1	37.440087	78.662396	60	109	13	Piedmont	fine, mixed, subactive, mesic Typic Hapludults	Littlejoe	Well
SC1	33.869400	79.289300	2	125	17	Atlantic Coastal Plain	fine, kaolinitic, thermic Umbric Paleaquults	Byars	Very Poorly
AL1	33.233371	87.232384	48	125	17	Appalachian Plateau	loamy-skeletal, mixed, subactive, thermic, Typic Dystrudepts	Montevallo	Well
AL2	31.659464	86.272045	34	145	26	Southern Coastal Plain	fine, smectitic, thermic, Typic Hapludults	Arundel	Well
AR	33.422310	91.732651	21	140	17	Western Gulf Coastal Plain	fine-silty, mixed, active, thermic, Typic Glossaqualfs	Calhoun	Poorly
Study 2									
VA2	37.445331	78.662917	60	109	13	Piedmont	fine, mixed, subactive, mesic Typic Hapludults	Littlejoe	Well
SC2	34.450000	80.505383	29	107	16	Sandhills	thermic coated Typic Quartzipsamments	Lakeland	Excessively
GA1	33.625317	82.801183	35	118	16	Piedmont	fine kaolinitic thermic Rhodic Kandiudults	Davidson	Well
GA2	31.339978	81.857283	1	114	18	Atlantic Coastal Plain	sandy siliceous thermic Aeric Alaquods	Leon	Poorly
GA3	31.299333	81.847217	1	114	18	Atlantic Coastal Plain	loamy, siliceous, subactive, thermic Arenic Paleaquults	Pelham	Somewhat Poorly
FL	30.205267	83.866817	0.6	142	20	Eastern Gulf Coastal Plain	loamy siliceous superactive thermic Aquic Arenic Hapludalfs	Melvina	Somewhat Poorly
MS	31.066717	89.602467	26	152	19	Western Gulf Coastal Plain	coarse loamy siliceous subactive thermic Typic Paleudults	Lucy	Well
LA1	31.337017	93.182783	28	147	19	Western Gulf Coastal Plain	fine smectitic thermic Albaquic Hapludalfs	Corrigan	Moderately Well
LA2	31.013333	93.422600	28	147	19	Western Gulf Coastal Plain	fine loamy siliceous subactive thermic Plinthic Paleudults	Malbis	Well

Site	Latitude	Longitude	Altitude (m)	MAP (cm)	MAT (°C)	Physiographic Region	Soil Taxonomic Class	Soil Series	Soil Drainage Class
Study 2									
LA3	30.560533	90.727650	0.9	160	19	Western Gulf Coastal Plain	fine silty mixed active thermic Typic Glossaqualfs	Gilbert	Poorly
OK	34.029333	94.825017	42	136	16	Western Gulf Coastal Plain	fine silty mixed active thermic Aquic Paleudalfs	Muskogee	moderately well
TX	31.13255	94.462533	32	127	20	Western Gulf Coastal Plain	fine loamy siliceous semiacitve thermic Oxyaquic Glossudalfs	Kurth	moderately well

Table 4.2. *Characteristics of loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N following application of urea or enhanced efficiency N fertilizers enriched with* ^{15}*N.*

Site	Trees plot^{-1}	Trees ha^{-1}	Mean Height (m)	Mean DBH (cm)	Basal Area (m^2 ha^{-1})
Study 1					
VA1	13	1260	13.1	16.4	27.4
SC1	5	540	15.7	25.9	29.4
AL1	4	400	19.2	24.4	19.0
AL2	6	600	18.1	20.5	20.5
AR	6	620	15.4	20.7	21.3
Study 2					
VA2	8	880	9.1	15.1	16.1
SC2	15	1560	12.5	16.1	33.9
GA1	19	1800	7.2	8.6	10.6
GA2	15	1460	14.7	16.5	33.1
GA3	13	1340	13.6	15.1	25.0
FL	16	1580	10.2	13.4	23.2
MS	21	2160	12.5	13.8	35.4
LA1	7	720	14.9	19.3	21.5
LA2	6	780	14.8	17.5	18.7
LA3	23	2380	12.0	13.3	36.7
OK	15	1580	3.0	4.0	2.1
TX	13	1360	13.9	11.2	20.0

Table 4.3. *Selected physical and chemical soil characteristics of loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N following application of urea or enhanced efficiency N fertilizers enriched with* ^{15}N.

Site	Depth	Bulk Density	CEC	pH	P	K	Ca	Mg	Zn	Mn	Cu	Fe	B
					Mehlich I Extracted								
	cm	**g cm^{-3}**	**cmol kg^{-1}**	**Water**	**mg kg^{-1}**								
Study 1													
VA1	0-15	1.50	6.46	4.22	4.20	35.80	84.30	15.90	0.80	7.58	0.35	63.86	0.10
	15-30	1.46	5.75	4.47	2.20	38.60	59.00	13.10	0.55	4.96	0.34	47.31	0.10
SC1	0-15	1.31	8.84	4.33	9.63	23.79	290.05	39.32	1.10	2.96	0.33	54.32	0.16
	15-30	1.30	6.67	4.49	4.45	12.50	181.25	27.50	0.75	1.26	0.35	63.24	0.10
AL1	0-15	1.37	13.00	4.32	4.10	62.60	684.70	188.70	0.53	10.28	0.38	65.77	0.14
	15-30	1.28	15.50	4.32	2.30	75.70	757.90	217.90	0.40	1.82	0.55	48.36	0.17
AL2	0-15	1.31	5.58	5.13	5.00	49.00	334.70	44.10	1.07	110.50	0.33	23.33	0.22
	15-30	1.29	4.97	5.07	2.80	46.10	254.00	40.20	0.61	82.68	0.33	19.65	0.17
SC2	0-15	1.57	2.75	4.27	4.00	10.75	41.00	8.00	0.35	2.70	0.28	23.50	0.10
	15-30	1.52	0.98	4.62	2.50	4.00	26.75	6.00	0.20	2.58	0.30	17.15	0.10
Study 2													
VA2	0-15	1.47	6.99	4.45	3.90	39.40	135.50	21.20	1.01	13.84	0.36	96.24	0.10
	15-30	1.43	6.10	4.53	2.80	45.80	64.00	15.70	0.81	7.27	0.35	38.30	0.10
GA1	0-15	1.41	6.14	5.48	3.56	53.11	552.56	123.78	0.78	185.53	1.09	24.08	0.20
	15-30	1.36	5.69	5.19	2.80	43.30	408.30	150.60	0.36	123.85	0.96	18.27	0.15
GA2	0-15	1.23	5.90	3.74	4.78	11.56	70.11	19.56	0.34	0.46	0.28	11.30	0.10
	15-30	1.32	4.59	4.02	6.11	6.78	47.67	11.22	0.22	3.81	0.27	12.59	0.10
GA3	0-15	1.23	6.34	3.73	3.00	10.13	70.75	19.88	0.36	0.49	0.21	15.45	0.10
	15-30	1.32	4.99	3.98	4.44	7.00	49.89	11.89	0.29	0.41	0.17	13.67	0.10
FL	0-15	1.39	5.40	5.04	24.75	19.75	348.75	57.50	0.45	7.40	0.28	20.30	0.10
	15-30	1.47	3.04	5.30	16.40	8.20	245.40	28.60	0.24	2.76	0.26	13.52	0.10

Site	Depth	Bulk Density	CEC	pH	P	K	Ca	Mg	Zn	Mn	Cu	Fe	B
								Mehlich I Extracted					
	cm	g cm^{-3}	cmol kg^{-1}	Water				mg kg^{-1}					
Study 2													
MS	0-15	1.44	5.28	4.99	6.25	19.75	235.50	26.00	0.38	25.15	0.35	19.00	0.10
	15-30	1.45	2.96	4.89	2.20	15.20	59.00	14.60	0.24	26.10	0.40	14.26	0.10
LA1	0-15	1.37	4.78	4.46	2.40	22.80	138.80	49.00	1.00	9.94	0.78	66.48	0.10
	15-30	1.36	6.86	4.53	1.40	25.20	148.80	96.20	0.48	3.78	0.82	37.98	0.10
LA2	0-15	1.48	3.32	4.74	2.00	16.00	130.80	20.60	0.30	27.16	0.26	37.32	0.10
	15-30	1.37	2.03	4.79	1.25	12.50	93.75	23.75	0.18	14.65	0.28	25.85	0.10
LA3	0-15	1.48	5.48	4.43	3.44	16.78	215.11	57.33	0.78	73.88	0.29	110.42	0.11
	15-30	1.48	6.69	4.46	1.78	14.67	174.22	83.11	0.36	23.99	0.29	71.69	0.10
AR1	0-15	1.21	4.85	5.00	3.05	27.53	331.42	73.84	0.62	178.62	0.50	43.21	0.13
	15-30	1.38	5.06	4.86	2.05	22.95	230.42	65.53	0.55	138.03	0.53	33.30	0.10
OK	0-15	1.50	8.67	4.87	4.43	41.71	652.71	125.57	0.84	54.56	0.20	33.96	0.13
	15-30	1.45	10.40	4.74	3.33	39.00	652.33	147.00	0.83	33.67	0.20	28.37	0.13
TX	0-15	1.50	5.55	4.67	2.75	26.75	217.25	63.25	0.40	10.73	0.36	39.36	0.11
	15-30	1.57	6.51	4.62	2.00	26.50	209.00	101.25	0.26	7.11	0.41	30.64	0.13

Table 4.4. *The mean ^{15}N (‰), N (g kg^{-1}) and fertilizer N recovery (% of fertilizer N applied) for individual ecosystem components for Study 1 (spring + summer 2011) for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Different letters represent significant differences at α = 0.05. Numbers in parentheses represent the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 10.*

		Spring + Summer		
Ecosystem Component	**Treatment**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery (% fertilizer N applied)**
Foliage	Control	*-3.23^{a} (0.51)*	*12.3^{a} (0.4)*	0.0^{b}
	CUF	**96.5^{b} (9.8)**	14.1^{ab} (0.5)	11.7^{a} (2.1)
	NBPT	**97.1^{b} (9.1)**	**15.0^{b} (0.7)**	14.8^{a} (2.9)
	PCU	**83.9^{b} (9.3)**	**14.4^{b} (0.4)**	9.1^{a} (0.9)
	Urea	**92.7^{b} (8.4)**	**14.7^{b} (0.5)**	12.2^{a} (2.4)
Fine Branches	Control	*-3.01^{a} (0.78)*	*4.9^{a} (0.2)*	0.0^{b}
	CUF	**86.9^{b} (7.4)**	**7.4^{b} (0.4)**	1.7^{a} (0.3)
	NBPT	**83.3^{b} (6.7)**	**7.3^{b} (0.5)**	1.9^{a} (0.4)
	PCU	**74.3^{b} (7.2)**	**6.9^{b} (0.3)**	1.1^{a} (0.2)
	Urea	**75.1^{b} (8.9)**	**6.6^{b} (0.4)**	1.2^{a} (0.3)
Coarse Branches	Control	*-2.58^{a} (0.78)*	3.4^{a} (0.6)	0.0^{b}
	CUF	**58.4^{b} (5.7)**	3.3^{a} (0.4)	4.6^{a} (0.8)
	NBPT	**59.9^{b} (6.3)**	3.3^{a} (0.3)	5.0^{a} (1.0)
	PCU	**52.8^{b} (5.7)**	3.3^{a} (0.3)	3.5^{a} (0.6)
	Urea	**55.7^{b} (3.1)**	3.2^{a} (0.2)	4.1^{a} (0.6)
Bark	Control	*-2.77^{a} (1.62)*	2.1^{a} (0.3)	0.0^{b}
	CUF	**15.3^{b} (3.4)**	2.1^{a} (0.2)	0.9^{a} (0.4)
	NBPT	**18.9^{b} (4.7)**	2.5^{a} (0.3)	1.1^{a} (0.3)
	PCU	**16.0^{b} (2.8)**	2.9^{a} (0.5)	1.1^{a} (0.3)
	Urea	**15.0^{b} (2.3)**	2.3^{a} (0.1)	0.7^{a} (0.1)
Growth Ring (CGR) year of sampling	Control	*-3.78^{a} (0.60)*	1.3^{a} (0.2)	0.0^{b}
	CUF	**73.8^{b} (7.3)**	1.5^{a} (0.1)	1.7^{a} (0.4)
	NBPT	**72.9^{b} (7.5)**	1.9^{a} (0.1)	1.9^{a} (0.2)
	PCU	**59.3^{b} (8.1)**	1.8^{a} (0.1)	1.6^{a} (0.3)
	Urea	**70.45^{b} (7.1)**	2.0^{a} (0.2)	2.1^{a} (0.4)
Growth Rings (PGR) composited prior to year of sampling	Control	*-3.38^{a} (1.13)*	1.0^{a} (0.1)	0.0^{b}
	CUF	**35.7^{b} (4.0)**	1.1^{a} (0.2)	3.4^{a} (0.7)
	NBPT	**36.6^{b} (4.8)**	1.3^{a} (0.2)	5.0^{a} (1.05)
	PCU	**30.7^{b} (4.3)**	1.2^{a} (0.2)	4.4^{a} (1.0)
	Urea	**31.6^{b} (4.5)**	0.9^{a} (0.2)	2.7^{a} (0.6)
Fine Roots (< 2mm)	Control	*1.55^{a} (1.89)*	12.1^{a} (0.8)	0.0^{b}
	CUF	**24.1^{b} (3.5)**	12.3^{a} (0.6)	11.1^{a} (1.8)
	NBPT	**19.8^{b} (3.4)**	12.8^{a} (0.7)	12.6^{a} (2.4)
	PCU	**19.3^{b} (3.2)**	12.4^{a} (0.6)	10.9^{a} (1.3)
	Urea	14.0^{ab} (1.5)	12.2^{a} (0.6)	10.0^{a} (1.8)

		Spring + Summer		
Ecosystem Component	**Treatment**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery (% fertilizer N applied)**
Coarse Roots (> 2mm)	Control	*0.86^{a} (0.73)*	*8.8^{a} (0.6)*	0.0^{b}
	CUF	***15.8^{c} (2.3)***	7.9^{ab} (0.5)	2.0^{a} (0.5)
	NBPT	14.0^{bc} (1.4)	7.7^{ab} (0.5)	2.4^{a} (0.8)
	PCU	12.2^{bc} (1.8)	**7.0^{b} (0.2)**	1.5^{a} (0.5)
	Urea	**8.5^{b} (1.0)**	**6.8^{b} (0.3)**	0.8^{a} (0.2)
Total Tree (Aboveground + Belowground Biomass)	Control			0.0^{b}
	CUF			37.1^{a}
	NBPT			44.7^{a}
	PCU			33.2^{a}
	Urea			33.8^{a}
Litterfall (Current year)	Control	*-2.38^{a} (0.75)*	*7.9^{a} (0.5)*	0.0^{b}
	CUF	**29.7^{b} (7.2)**	6.1^{ab} (0.6)	0.4^{a} (0.1)
	NBPT	**31.0^{b} (7.5)**	5.9^{ab} (0.8)	0.4^{a} (0.2)
	PCU	**33.8^{b} (8.2)**	6.0^{ab} (0.6)	0.5^{a} (0.2)
	Urea	**28.9^{b} (6.9)**	**5.1^{b} (0.7)**	0.3^{a} (0.1)
Organic Horizon (Oi + Oe + Oa)	Control	*-2.35^{a} (0.35)*	7.3^{a} (0.6)	0.0^{b}
	CUF	**77.5^{b} (10.1)**	9.4^{a} (0.7)	2.8^{a} (0.8)
	NBPT	**71.5^{b} (9.3)**	8.6^{a} (0.5)	2.8^{a} (0.8)
	PCU	**89.6^{b} (7.8)**	9.4^{a} (0.8)	3.4^{a} (1.0)
	Urea	**65.0^{b} (9.1)**	8.5^{a} (0.7)	2. 0^{a} (0.4)
0-15 cm Mineral Soil	Control	*3.70^{a} (0.59)*	1.0^{a} (0.1)	0.0^{c}
	CUF	**16.6^{b} (1.9)**	1.2^{a} (0.2)	**27.8^{a} (3.8)**
	NBPT	**16.1^{b} (1.7)**	1.1^{a} (0.2)	**26.5^{a} (4.2)**
	PCU	**18.0^{b} (2.8)**	1.3^{a} (0.3)	**31.2^{a} (4.2)**
	Urea	**10.2^{b} (1.5)**	1.2^{a} (0.3)	**13.4^{b} (3.7)**
15-30 cm Mineral Soil	Control	*5.44^{a} (0.37)*	0.7^{a} (0.1)	0.0^{b}
	CUF	**11.9^{b} (2.3)**	0.7^{a} (0.1)	8.3^{a} (3.3)
	NBPT	10.3^{ab} (1.0)	0.7^{a} (0.2)	6.2^{a} (1.3)
	PCU	11.2^{ab} (1.5)	0.9^{a} (0.3)	7.9^{a} (1.7)
	Urea	8.5^{ab} (0.6)	0.9^{a} (0.3)	5.1^{a} (2.1)
Total Soil (Organic + Mineral)	Control			0.0^{c}
	CUF			38.9^{a}
	NBPT			35.5^{ab}
	PCU			42.5^{a}
	Urea			20.5^{b}

Table 4.5. *Analysis of variance for treatment differences for fertilizer N recovery (% of fertilizer N applied) for total and the primary ecosystem components for Study 1 (spring + summer 2011) at mid-rotation loblolly pine plantations in the southern United States. N application rate was 224 kg N ha^{-1}. N = 10.*

Source	Degrees of Freedom	Adj. Sum of Squares	Adj. Mean Squares	F-Value	P-Value
Total Ecosystem Fertilizer N Recovery					
Treatment	4	36904.2	9226.04	21.31	**0.000**
Season	1	82.5	82.52	0.19	0.665
Treatment*Season	4	416.0	104.00	0.24	0.914
Error	40	17317.7	432.94		
Total	49	54720.4			
Loblolly pine tree (foliage + fine branch + coarse branch + bark + CGR + PGR + fine roots + coarse roots)					
Treatment	4	7433.0	1858.2	17.71	0.112
Season	1	760.2	760.2	1.25	0.150
Treatment*Season	4	400.4	100.1	0.95	0.443
Error	40	4196.7	104.9		
Total	49	12790.4			
Litterfall					
Treatment	4	1.4512	0.36281	1.94	0.122
Season	1	0.3003	0.30035	1.61	0.212
Treatment*Season	4	0.1236	0.03090	0.17	0.955
Error	40	7.4662	0.18665		
Total	49	9.3414			
Soil (O horizon + 0-15 cm mineral soil + 15-30 cm mineral soil)					
Treatment	4	6196.03	2065.34	6.91	**0.001**
Season	1	28.56	28.56	0.10	0.759
Treatment* Season	4	393.14	131.05	0.44	0.727
Error	40	9558.22	298.69		
Total	49				
Foliage					
Treatment	4	1308	327.05	8.45	**0.000**
Error	45	1742	38.72		
Total	49				
Fine Branches					
Treatment	4	21.20	5.3009	7.59	**0.000**
Error	45	31.45	0.6988		
Total	49	52.65			
Coarse Branches					
Treatment	4	159.5	39.871	9.29	**0.000**
Error	45	193.0	4.290		
Total	49				
Bark					
Treatment	4	8.199	2.0497	3.50	**0.014**
Error	45	26.327	0.5850		
Total	49	34.526			

Source	Degrees of Freedom	Adj. Sum of Squares	Adj. Mean Squares	F-Value	P-Value
Current Growth Ring					
Treatment	4	28.79	7.1981	8.06	**0.000**
Error	45	40.18	0.8930		
Total	49	68.98			
Previous Growth Rings					
Treatment	4	152.2	38.054	6.57	**0.000**
Error	45	260.8	5.796		
Total	49	413.1			
Fine Roots (< 2 mm)					
Treatment	4	655.8	163.94	4.23	**0.005**
Error	45	1742.3	38.72		
Total	49				
Coarse Roots (> 2 mm)					
Treatment	4	36.07	9.017	3.85	**0.009**
Error	45	105.26	2.339		
Total	49	141.33			
Litterfall					
Treatment	4	1.451	0.3628	2.07	0.101
Error	45	7.890	0.1753		
Total	49	9.341			
Organic Horizon					
Treatment	4	69.57	17.393	3.79	**0.010**
Error	45	206.31	4.585		
Total	49	275.88			
0-15 cm Mineral Soil					
Treatment	4	6732	1683.1	13.47	**0.000**
Error	45	5621	124.9		
Total	49	12353			
15-30 cm Mineral Soil					
Treatment	4	442.3	110.59	2.76	**0.039**
Error	45	1802.5	40.06		
Total	49	2244.8			

Table 4.6. *The mean ^{15}N (‰), N (g kg^{-1}) and fertilizer N recovery (% of applied fertilizer N) for individual ecosystem components for Study 2 (March-April 2012) for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Different letters represent significant differences at α = 0.05. Numbers in parentheses represent the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 12 for all ecosystem components except sapling/pole and shrub strata where N = 7.*

Ecosystem Component	**Treatment**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery (% of applied N)**
Foliage	Control	*-2.37^{a} (0.51)*	12.5^{a} (0.4)	0.0^{c}
	CUF	**118.3^{b} (11.2)**	14.1^{a} (0.8)	10.7^{ab} (1.1)
	NBPT	**126.1^{b} (8.5)**	13.9^{a} (0.6)	**14.8^{b} (1.8)**
	PCU	**101.6^{b} (9.8)**	13.2^{a} (0.5)	*8.1^{a} (1.1)*
	Urea	**124.5^{b} (11.2)**	14.2^{a} (0.5)	11.0^{ab} (1.7)
Fine Branches	Control	*-2.02^{a} (0.56)*	*5.1^{a} (0.3)*	0.0^{c}
	CUF	**110.2^{b} (10.2)**	**7.3^{b} (0.6)**	3.2^{ab} (0.4)
	NBPT	**111.8^{b} (7.4)**	6.7^{ab} (0.3)	**4.1^{b} (0.5)**
	PCU	**98.7^{b} (10.5)**	6.1^{ab} (0.5)	*2.6^{a} (0.5)*
	Urea	**108.4^{b} (9.67)**	6.2^{ab} (0.4)	2.9^{ab} (0.5)
Coarse Branches	Control	*-1.82^{a} (0.41)*	2.8^{a} (0.2)	0.0^{b}
	CUF	**72.6^{b} (9.3)**	3.7^{a} (0.3)	2.9^{a} (0.4)
	NBPT	**78.9^{b} (8.2)**	3.6^{a} (0.4)	3.0^{a} (0.6)
	PCU	**73.7^{b} (8.6)**	3.3^{a} (0.3)	2.6^{a} (0.4)
	Urea	**69.3^{b} (6.1)**	3.9^{a} (0.4)	3.2^{a} (1.0)
Bark	Control	*-2.09^{a} (0.44)*	*2.1^{a} (0.2)*	0.0^{b}
	CUF	**22.1^{b} (2.1)**	2.5^{ab} (0.2)	0.6^{a} (0.2)
	NBPT	**22.5^{b} (2.1)**	**2.9^{b} (0.1)**	0.8^{a} (0.2)
	PCU	**20.5^{b} (2.4)**	2.4^{ab} (0.1)	0.5^{a} (0.1)
	Urea	**20.8^{b} (2.1)**	2.5^{ab} (0.2)	0.4^{a} (0.1)
Growth Rings (CGR)- year of sampling	Control	*-2.03^{a} (0.44)*	*1.8^{a} (0.1)*	0.0^{b}
	CUF	**80.0^{b} (8.0)**	**2.9^{b} (0.5)**	2.2^{a} (0.4)
	NBPT	**88.1^{b} (5.8)**	2.7^{ab} (0.1)	3.1^{a} (0.6)
	PCU	**71.9^{b} (7.6)**	2.3^{ab} (0.1)	1.7^{a} (0.3)
	Urea	**78.9^{b} (7.5)**	2.3^{ab} (0.1)	1.9^{a} (0.3)
Growth Rings (PGR) - composited prior to year of sampling	Control	*-1.85^{a} (0.39)*	1.4^{a} (0.1)	0.0^{b}
	CUF	**40.2^{b} (3.9)**	1.6^{a} (0.1)	4.4^{a} (0.8)
	NBPT	**40.3^{b} (1.3)**	1.6^{a} (0.1)	5.1^{a} (0.7)
	PCU	**37.1^{b} (3.9)**	1.6^{a} (0.1)	3.7^{a} (0.4)
	Urea	**34.8^{b} (5.0)**	1.4^{a} (0.1)	2.9^{a} (0.5)
Fine roots (< 2 mm)	Control	*-0.28^{a} (0.86)*	9.4^{a} (0.9)	0.0^{b}
	CUF	**36.1^{b} (3.1)**	10.5^{a} (0.6)	*19.2^{a} (1.8)*
	NBPT	**33.2^{b} (3.8)**	10.4^{a} (0.4)	16.2^{ab} (1.50)
	PCU	**36.0^{b} (3.5)**	10.3^{a} (0.5)	16.4^{ab} (1.8)
	Urea	**31.2^{b} (5.1)**	9.7^{a} (0.5)	**10.8^{b} (1.4)**

Ecosystem Component	Treatment	^{15}N (‰)	N (g kg^{-1})	Fertilizer N Recovery (% of applied N)
Coarse roots (> 2 mm)	Control	*-0.09^{a} (0.37)*	6.0^{a} (0.6)	0.0^{b}
	CUF	**18.3^{b} (1.6)**	6.2^{a} (0.5)	3.8^{a} (1.4)
	NBPT	**19.0^{b} (3.2)**	6.3^{a} (0.5)	2.7^{a} (0.6)
	PCU	**16.5^{b} (2.2)**	6.6^{a} (0.5)	2.9^{a} (0.9)
	Urea	**13.9^{b} (3.1)**	6.0^{a} (0.5)	1.7^{a} (0.5)
Total Tree (Aboveground + Belowground Biomass)	Control			0.0^{c}
	CUF			47.0^{a}
	NBPT			49.8^{a}
	PCU			38.5^{b}
	Urea			34.8^{b}
Sapling/Pole Stratum (n = 7)	Control	*-0.49^{a} (0.54)*	6.2^{a} (1.0)	0.0^{b}
	CUF	**61.9^{b} (14.9)**	8.6^{a} (1.2)	0.3^{a} (0.1)
	NBPT	**83.3^{b} (21.3)**	9.7^{a} (2.8)	0.9^{a} (0.5)
	PCU	**64.1^{b} (15.4)**	8.2^{a} (1.6)	0.3^{a} (0.1)
	Urea	59.6^{ab} (22.3)	8.1^{a} (1.4)	0.9^{a} (0.7)
Shrub Stratum (n = 7)	Control	*-0.24^{a} (0.56)*	10.3^{a} (1.4)	0.0^{b}
	CUF	**90.1^{b} (16.1)**	11.0^{a} (1.4)	0.5^{a} (0.2)
	NBPT	**112.0^{b} (24.4)**	9.4^{a} (2.7)	0.7^{a} (0.2)
	PCU	55.4^{ab} (14.9)	9.0^{a} (1.2)	1.4^{a} (0.5)
	Urea	**93.4^{b} (18.3)**	8.4^{a} (0.7)	0.4^{a} (0.2)
Litterfall	Control	*-3.00^{a} (0.66)*	7.4^{a} (0.8)	0.0^{b}
	CUF	**48.6^{b} (5.5)**	8.0^{a} (0.5)	1.6^{a} (0.3)
	NBPT	**55.8^{b} (5.6)**	8.3^{a} (0.9)	1.4^{a} (0.3)
	PCU	**55.6^{b} (6.9)**	8.1^{a} (0.6)	1.8^{a} (0.5)
	Urea	**55.1^{b} (6.9)**	8.2^{a} (0.6)	1.7^{a} (0.3)
Organic horizon (Oi + Oe + Oa)	Control	*-1.93^{a} (0.44)*	6.6^{a} (0.6)	0.0^{b}
	CUF	**62.9^{b} (6.8)**	8.1^{a} (0.6)	3.0^{a} (1.4)
	NBPT	**59.3^{b} (5.8)**	8.0^{a} (0.6)	2.8^{a} (1.3)
	PCU	**91.1^{c} (9.91)**	8.7^{a} (0.6)	3.8^{a} (0.9)
	Urea	**55.2^{b} (6.34)**	8.0^{a} (0.7)	1.2^{a} (0.4)
0-15 cm Mineral soil	Control	*3.27^{a} (0.70)*	0.8^{a} (0.1)	0.0^{b}
	CUF	**21.8^{b} (3.6)**	0.6^{a} (0.1)	23.1^{a} (3.6)
	NBPT	**22.3^{b} (3.5)**	0.6^{a} (0.1)	22.4^{a} (2.7)
	PCU	**21.0^{b} (4.1)**	0.7^{a} (0.1)	24.2^{a} (5.1)
	Urea	**15.9^{b} (2.1)**	0.6^{a} (0.1)	15.9^{a} (2.6)
15-30 cm Mineral Soil	Control	*5.38^{a} (0.43)*	0.4^{a} (0.1)	0.0^{b}
	CUF	**13.2^{b} (1.5)**	0.4^{a} (0.03)	6.7^{a} (1.1)
	NBPT	11.7^{ab} (1.0)	0.4^{a} (0.1)	5.4^{a} (0.9)
	PCU	**16.3^{b} (3.0)**	0.4^{a} (0.0)	10.7^{a} (3.4)
	Urea	**15.2^{b} (2.4)**	0.4^{a} (0.1)	11.3^{a} (3.12)
Total Soil (Organic + Mineral)	Control			0.0^{b}
	CUF			32.8^{a}
	NBPT			30.6^{a}
	PCU			38.7^{a}
	Urea			28.4^{a}

Table 4.7. *Analysis of variance for treatment differences for fertilizer N recovery (% of fertilizer N applied) for total and the primary ecosystem components for Study 2 (spring 2012) at mid-rotation loblolly pine plantations in the southern United States. N application rate was 224 kg N ha^{-1}. N = 12 except for Understory where N = 7.*

Source	**Degrees of Freedom**	**Adj. Sum of Squares**	**Adj. Mean Squares**	**F-Value**	**P-Value**
Total Ecosystem Fertilizer N Recovery					
Treatment	4	61761	15440.4	87.77	**0.000**
Error	55	9676	175.9		
Total	59	71437			
Loblolly pine tree (foliage + fine branch + coarse branch + bark + CGR + PGR + fine roots + coarse roots					
Treatment	4	19159	4789.75	77.09	**0.000**
Error	55	3417	62.13		
Total	59	22576			
Litterfall					
Treatment	4	26.13	6.533	4.75	**0.002**
Error	55	75.60	1.375		
Total	59	101.73			
Understory (Saplings+Shrubs+Vines+Herbaceous) (n = 7)					
Treatment	4	10.60	2.6489	4.03	**0.010**
Error	29	19.05	0.6568		
Total	33	29.64			
Soil (Organic Horizon + 0-15 cm mineral soil + 15-30 cm mineral soil)					
Treatment	4	10823	2705.8	19.11	**0.000**
Error	55	7788	141.6		
Total	59	18611			
Foliage					
Treatment	4	1472	367.93	18.36	**0.000**
Error	55	1102	20.04		
Total	59				
Fine Branches					
Treatment	4	112.6	28.154	12.95	**0.000**
Error	55	119.6	2.175		
Total	59	232.2			
Coarse Branches					
Treatment	4	85.04	21.259	5.35	**0.001**
Error	55	218.62	3.975		
Total	59				
Bark					
Treatment	4	4.700	1.1749	5.79	**0.001**
Error	55	11.164	0.2030		
Total	59	15.864			
Current Growth Ring					
Treatment	4	62.43	15.607	9.67	**0.000**
Error	55	88.73	1.613		
Total	59	151.16			
Previous Growth Ring					
Treatment	4	187.5	46.872	12.43	**0.000**
Error	55	207.4	3.770		
Total	59	394.9			

Source	**Degrees of Freedom**	**Adj. Sum of Squares**	**Adj. Mean Squares**	**F-Value**	**P-Value**
Fine Roots (< 2mm)					
Treatment	4	2751	687.81	26.99	**0.000**
Error	55	1401	25.48		
Total	59	4153			
Coarse Roots (> 2mm)					
Treatment	4	99.74	24.934	3.16	**0.021**
Error	55	434.48	7.900		
Total	59	534.22			
Litterfall					
Treatment	4	26.13	6.533	4.75	**0.002**
Error	55	75.60	1.375		
Total	59	101.73			
Organic Horizon (Oi + Oe + Oa)					
Treatment	4	115.2	28.79	2.54	**0.050**
Error	55	622.8	11.32		
Total	59	738.0			
0-15 cm Mineral Soil					
Treatment	4	4895	1223.7	9.74	**0.000**
Error	55	6910	125.6		
Total	59	11805			
15-30 cm Mineral Soil					
Treatment	4	1005	251.25	4.49	**0.003**
Error	55	3077	55.95		
Total	59	4082			

Table 4.8. *P > | t | values for fertilizer N treatment (CUF, NBPT, PCU, urea) contrasts for total ecosystem fertilizer N recovery (% of fertilizer N applied) for Study 1 (spring + summer 2011) and Study 2 (spring 2012) at mid-rotation loblolly pine plantations in the southern United States. The N application rate was 224 kg N ha^{-1}. N =10.*

	Study 1- 2011 (n = 10)	***Study 2 – 2011 (n = 12)***
Differences of Fertilizer N Treatment	***Adj. P-Value***	***Adj. P-Value***
Total Ecosystem Fertilizer N Recovery		
Control – CUF	**0.000**	**0.000**
Control – NBPT	**0.000**	**0.000**
Control - PCU	**0.000**	**0.000**
Control - Urea	**0.000**	**0.000**
CUF - NBPT	0.903	0.975
CUF - PCU	1.000	0.795
CUF - Urea	**0.008**	**0.004**
NBPT - PCU	0.864	0.984
NBPT - Urea	**0.001**	**0.020**
PCU - Urea	**0.010**	**0.054**
Loblolly pine tree (foliage + fine branch + coarse branch + bark + CGR + PGR + fine roots + coarse roots		
Control – CUF	**0.000**	**0.000**
Control – NBPT	**0.000**	**0.000**
Control - PCU	**0.000**	**0.000**
Control - Urea	**0.000**	**0.000**
CUF - NBPT	0.687	0.887
CUF - PCU	0.945	**0.053**
CUF - Urea	0.868	**0.003**
NBPT - PCU	0.261	**0.007**
NBPT - Urea	0.174	**0.000**
PCU - Urea	0.999	0.775
Litterfall		
Control – CUF	0.364	0.364
Control – NBPT	0.218	0.218
Control - PCU	0.102	0.102
Control - Urea	0.683	0.683
CUF - NBPT	0.998	0.998
CUF - PCU	0.956	0.956
CUF - Urea	0.984	0.984
NBPT - PCU	0.995	0.995
NBPT - Urea	0.917	0.917
PCU - Urea	0.737	0.737
Soil (Organic Horizon + 0-15 cm mineral soil + 15-30 cm mineral soil)		
Control – CUF	**0.000**	**0.000**
Control – NBPT	**0.000**	**0.000**
Control - PCU	**0.000**	**0.000**
Control - Urea	**0.019**	**0.000**
CUF - NBPT	0.982	0.991
CUF - PCU	0.979	0.750
CUF - Urea	**0.043**	0.899
NBPT - PCU	0.802	0.472
NBPT - Urea	0.141	0.992
PCU - Urea	**0.010**	0.238

Chapter 5. Summary and Conclusions

5.1. Introduction

Optimizing forest plantations is critical to meet current and future societal demand for wood products. Improving forest plantation productivity is essential to alleviate pressures to harvest natural forests that function in providing clean water and air, wildlife habitat, and biodiversity. The optimization of forest plantation productivity can be achieved through the development and implementation of site specific management and includes the proper identification and amelioration of nutrient deficiencies and imbalances to desired tree species. In the southern United States, nitrogen (N) is generally the most limiting nutrient to growth due to its disproportional requirement compared to other nutrients in photosynthesis. Although fertilization with N (and phosphorous - P) has improved production in southern plantation forests for decades, uncertainties exist to the pathways and mechanisms of applied fertilizer N cycling in these systems. An improved understanding of the ultimate fate of fertilizer N in loblolly pine (*Pinus taeda* L.) plantations is critical to improve site-specific management for enhancing economic and environmental fertilizer N stewardship in southern forest systems.

Improving the productivity of forest plantations is critical to meet the current and projected global demand for traditional and emerging wood-based markets (FAO 2015). The global optimization of forest plantations is critical to minimize the pressure on intensive wood harvesting in natural forests which provide clean water and air, wildlife habitat, food, carbon sequestration, and areas of biodiversity (Sedjo 1995, Fox 2000, Smethhurst 2014, WWF 2015). Improved forest plantation productivity can be achieved through the development and implementation of site specific forest management. The identification and amelioration of nutrient deficiencies and imbalances that limit tree growth is an integral component of site specific forest management. A fundamental understanding of the processes to improve nutrient availability and the cycling through plants and soils is critical to developing globally sustainable forest management practices for both plantation and natural forests.

In the southern United States, there are approximately 15.8 million ha of pine (*Pinus* spp.) plantations (Wear and Greis 2012). Nitrogen and P are generally the most limiting nutrients in the soils that support pine plantations in the southern United States. Both N and P are required more than other nutrients due to the disproportional demand in photosynthesis and foliar production (Chapin et al. 1986, Linder 1987, Vitousek and Howarth 1991). The addition of N and P containing fertilizers since the 1950s has generally increased pine plantation productivity in the southern United States by ameliorating these N and P nutrient deficiencies (Allen 1987, Fischer and Binkley 2000, Richter et al. 2000, Allen et al. 2005, Fox et al. 2007a, b). This finding has led to the annual N + P fertilization of approximately one third of pine plantations in the South (Albaugh et al. 2004), with additional nutrients applied when required on a site specific basis (Albaugh et al. 1998, Sampson and Allen 1999, Jokela and Martin 2000, Vogel and Jokela 2011, Carlson et al. 2014).

Despite significant forest productivity gains from fertilization, uncertainty remains to the ultimate fate of fertilizer N in these southern pine plantations (Fox et al. 2007a). For example, some stands are nonresponsive to N fertilization because the fertilizer N may be: 1) lost from the system via ammonia (NH_3) volatilization, nitrification-denitrification, or leaching; 2) immobilized in the soil by microbial communities or on soil exchange sites; 3) partitioned into other ecosystem components such as understory competition; or 4) the site may not be N deficient due to high rates of mineralization from decomposing organic matter (Pritchett and Smith 1975, Birk and Vitousek 1986, Martin et al. 1999, Amateis et al. 2000, Albaugh et al. 2004, Carlson et al. 2014). Additionally, stands may not respond because deficiencies of elements other than N create nutrient imbalances that limit growth (Vogel and Jokela 2011, Carlson et al. 2014), and these sites may respond to N fertilization if the required nutrients could be properly identified and effectively applied. The cumulative effect of reduced fertilizer N uptake by trees translates to a lower fertilizer nitrogen use efficiency (FNUE). An improved understanding of the pathways and mechanisms resulting in the partitioning of fertilizer N in these pine plantations is needed to improve FNUE to: 1) reduce negative environmental impacts; 2) improve productivity; and 3)reduce fertilizer inputs, all critical issues concerning fertilizer with N in production agroecosystems today (Zhang et al. 2015). The inability to accurately determine site responsiveness of loblolly pine (*Pinus taeda* L.) plantations to N fertilization hampers basic forest management decisions. Until the understanding of the mechanisms and interactions governing fertilizer N cycling on a site-specific basis improves, the ability to accurately predict sites responsive to N fertilization will be limited.

The primary objective of this research was to improve the understanding N cycling after fertilization in mid-rotation intensively managed loblolly pine plantations across the entire range where loblolly pine is operationally viable in the southern United States. Enhanced efficiency fertilizers were assessed to determine if there could be improvements to FNUE compared to urea, the conventional form of fertilizer N used in southern forestry. In Chapters 2, 3 and 4, the following questions were addressed: 1) Does fertilizer N loss, specifically ammonia (NH_3) volatilization, differ between conventional (urea) and enhanced efficiency N containing fertilizers (*see* Chapter 2); 2) Does the use of enhanced efficiency N containing fertilizers impact seasonal N uptake compared to urea of loblolly pines (*see* Chapter 3); and 3) Is there a difference in the ecosystem partitioning of fertilizer N among enhanced efficiency fertilizers and urea (*see* Chapter 4)? This research used N containing fertilizers enriched with the stable isotope ^{15}N to improve the quantification and understanding of fertilizer N cycling in loblolly pine plantations. The results from this research provides forest managers the biological foundation to improve their decisions concerning the economic feasibility of N fertilization, and whether using enhanced efficiency N containing fertilizers may improve FNUE in mid-rotation loblolly pine plantations across the entire southern United States.

This study encompassed diverse stand and site conditions across the primary ecophysiographic regions of the southern United States where loblolly pine plantations are operationally viable. Eighteen sites were selected from an existing network of forest thinning and fertilization studies in mid-rotation loblolly pine plantations across the South for a spring application of fertilizer N treatments. From these eighteen sites, a subset of six sites were chosen for a summer application of fertilizer N treatments. Six sites were installed during 2011 and twelve sites in 2012. The six sites with a summer fertilization were installed in 2011. The same six sites installed in 2011 were chosen for both a spring and summer fertilizer N application to assess fertilizer N loss and temporal N uptake of loblolly pine

5.2. Chapter Results and Discussion

The findings from this research highlight the ability to use enhanced efficiency fertilizers to improve fertilizer nitrogen use efficiency compared to urea. There are three primary conclusions from this research:

1) The enhanced efficiency fertilizers used in this research were equal in their ability to reduce fertilizer N loss through ammonia (NH_3) volatilization when compared to urea, regardless of the timing of application. Although NH_3 volatilization was

positively correlated with certain weather variables for urea, correlations were generally weak or negatively correlated with all enhanced efficiency fertilizers, indicating fertilizer N loss from enhanced efficiency fertilizers was independent from the weather conditions at the time of application (*see* Chapter 2).

2) The pattern of fertilizer N uptake by loblolly pines was similar for all fertilizer N treatments after a spring and summer fertilization. One exception was the PCU treatment, which had a lower N uptake pattern immediately following application, but was similar to the other fertilizer treatments towards the middle and end of the growing season. These results indicate that although release mechanisms and timing may differ between enhanced efficiency fertilizers and urea, this does not adversely affect the fertilizer N uptake of loblolly pine trees from EEFs compared to urea. Foliar development occurred earlier and increased in total amounts (flushes) at the end of the growing season for fertilized compared to unfertilized treatments. In addition, the amount of N originating from both the fertilizer and natural system increased in fertilized compared to unfertilized treatments (*see* Chapter 3).

3) More total fertilizer N was recovered from the entire ecosystem for all EEFs compared to urea. There were differences between enhanced efficiency N containing fertilizers and urea for certain ecosystem components, but few differences between individual EEFs. Most fertilizer N recovered was either in the loblolly pine tree or mineral soil, while other ecosystem components were minor sinks for the fertilizer N. There were also differences between individual EEFs and urea for certain loblolly pine tree components, indicating an increased FNUE for most EEFs compared to urea. (*see* Chapter 4).

An improved understanding of how conventional (urea) and enhanced efficiency fertilizer N cycles through mid-rotation southern loblolly pine forests was provided by this dissertation research. This research indicates enhanced efficiency N containing fertilizers can improve fertilizer nitrogen use efficiency when compared to urea, in intensively managed mid-rotation southern loblolly pine forests. Although there were clear differences between all the enhanced efficiency N containing fertilizers and urea, there were not clear differences between individual enhanced efficiency fertilizers.

When the results from individual Chapters are viewed collectively, it would appear that the release mechanisms have a short term influence on the ecosystem partitioning of fertilizer N. Urea rapidly releases N that is plant available, but the amount of urea-N lost through NH_3 volatilization can range from 5% to 90% and is generally dependent on weather conditions at the time of application (Kissel et al. 2004, 2009, Elliot and Fox 2014, *see* Chapter 2). Higher urea N losses after fertilization decrease stand FNUE for the stand. The effects of elevated losses of fertilizer N in forest plantations are similar to agriculture, which if inappropriately applied can create adverse environmental impacts, especially when viewed in the context of the entire life cycle of urea production (Galloway et al. 2003, Zhang et al. 2015). Yet because N fertilization in forestry occurs only once or twice during an entire stand rotation, adverse impacts associated with fertilizer N losses in forestry are primarily economic due to reduced fertilizer response and the time required to carry the costs of fertilization before obtaining a revenue stream (10 to 30 years, product dependent). This contrasts to agriculture where crops, N fertilization, and revenue occurs annually, and the environmental impacts associated with N fertilization have the potential to be more problematic.

Although high fertilizer N losses after urea application may translate to a lower fertilizer response for the stand, this response may not be immediately apparent. Although considerable urea N loss (26% to 49%) occurred after fertilization (*see* Chapter 2), fertilizer N uptake of urea N by loblolly pine continued over the entire growing season regardless of application season (*see* Chapter 3) which translated to a 30% to 35% fertilizer N uptake by loblolly pine at the end of a single growing season (*see* Chapter 4). This percentage of fertilizer N uptake by loblolly pine after urea fertilization was similar to findings in the literature (Mead and Pritchett 1975, Albaugh et al. 1998, 2004, Blazier et al. 2006). Conversely, fertilizer N loss after application of enhanced efficiency N containing fertilizers (4% to 26%) was considerably lower than urea (*see* Chapter 2). Although fertilizer N uptake patterns of most EEFs were similar to urea by loblolly pine over the entire growing season regardless of application season (*see* Chapter 3), fertilizer N uptake ranged from 30% to 50% for EEFs (*see* Chapter 4). Clearly and increase in FNUE occurred for EEFs compared to urea in these mid-rotation southern loblolly pine forests. Additionally, fertilizer N remaining in the soil was generally greater for EEFs (31% to 43%) compared to urea (21% to 28%). Future research will need to determine whether additional gains in stand FNUE occur from N uptake of fertilizer N that remained in the soil, or if the fertilizer N that remained in the soil is immobilized and unavailable for plant uptake.

Although the findings of this research have addressed specific fundamental questions concerning N cycling after fertilization in intensively managed mid-rotation loblolly pine forests across the southern United States, key questions remain. Directions of future research should include (Figure 5.1):

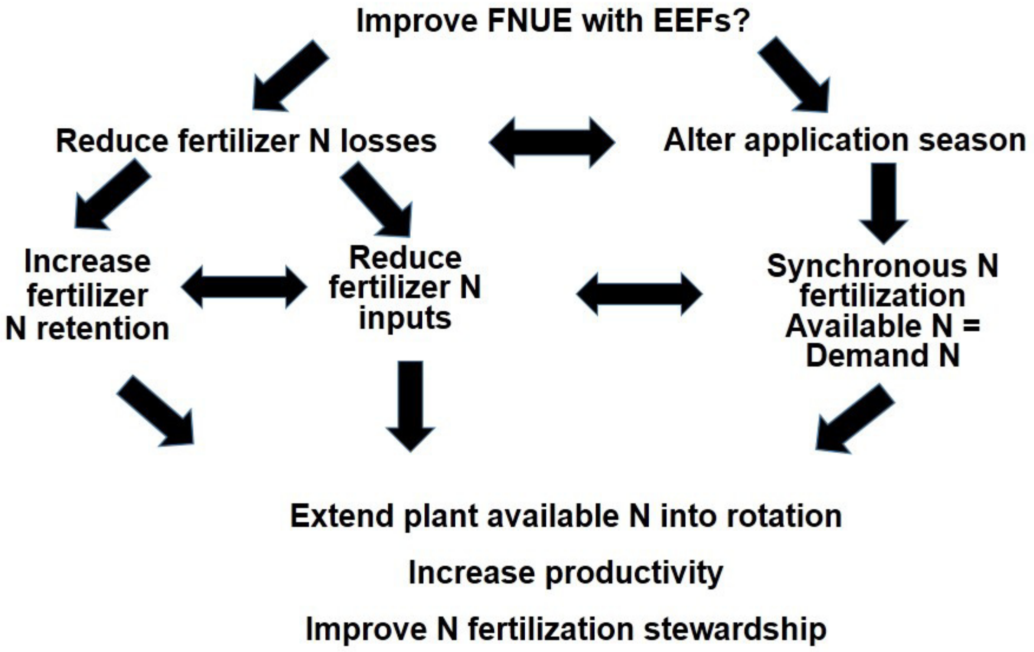
1) Loss mechanisms of fertilizer N during winter months. Although this research focused on N fertilization in spring and summer, most forest fertilization in the South occurs during winter months. Three questions specific to winter fertilization with N are: 1) does a larger percentage of fertilizer N loss from urea occur through

denitrification and/or leaching during the winter; 2) can enhanced efficiency fertilizers reduce losses during a winter application period, and 3) does N uptake by loblolly pines after a winter fertilization differ from spring and summer? This question is especially important for bedded sites where 50% of the site may be interbedded and inundated during the winter. Denitrification, as shown by Shreshta et al. (2014) could be an increasingly important loss pathway for fertilizer N after a winter fertilization with N.

2) Fertilizer application rates using enhanced efficiency fertilizers. This research has shown that EEFs can reduce fertilizer N losses, translating to increased fertilizer N remaining in the system. If fertilizer N application using EEFs could be reduced between 20% to 50%, the amount of fertilizer N lost following urea fertilization, could similar productivity gains occur with the current N fertilization rates of urea?
3) Long term cycling of fertilizer N originating from enhanced efficiency fertilizers. Because more fertilizer N remained in the loblolly pine system with EEFs compared to urea, what does this mean? Do the incremental gains in fertilizer N retention for the entire loblolly pine plantation ecosystem for EEFs compared to urea translate to a sustained release of fertilizer N into the stand rotation? If yes, does this continue to increase FNUE?
4) Productivity gains. Although there was a general trend in fertilizer N uptake for the entire tree for the EEFs compared to urea, does this translate to an increase in stand productivity, or is this luxury consumption for the trees with little gain in productivity? This study was co-located with numerous fertilizer and thinning trials, and the results from this research will need to be integrated with the results from these existing fertilizer and thinning trials.

The findings of this research provide forest managers the biological foundation of the positive attributes in using enhanced efficiency fertilizers to improve fertilizer nitrogen use efficiency in mid-rotation loblolly pine plantations across the southern United States when compared to urea, the current primary sources of fertilizer N in forestry. Forest managers will need to determine whether the positive attributes of enhanced efficiency fertilizers shown in this study are economic viable within the constructs of their land management. Future research will continue to refine the understanding of N cycling after fertilization with N through the aforementioned recommended research directions to achieve sustained incremental gains in productivity in loblolly pine plantations in the southern United States.

Figure 5.1. *Implication and directions of current and future research for improving the understanding of nitrogen cycling after fertilization with N containing fertilizers in mid-rotation loblolly pine plantations across the southern United States.*



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Appendix

Chapter 3

Figure A.3.1. *The N concentration (g kg^{-1}) values for flush 0, 1, and 2 for loblolly pine stands in the southern United States selected to evaluate temporal uptake of fertilizer nitrogen enriched with ^{15}N following a spring and summer application of urea or enhanced efficiency nitrogen fertilizers. The N application rate was 224 kg N ha^{-1}. N = 6.*

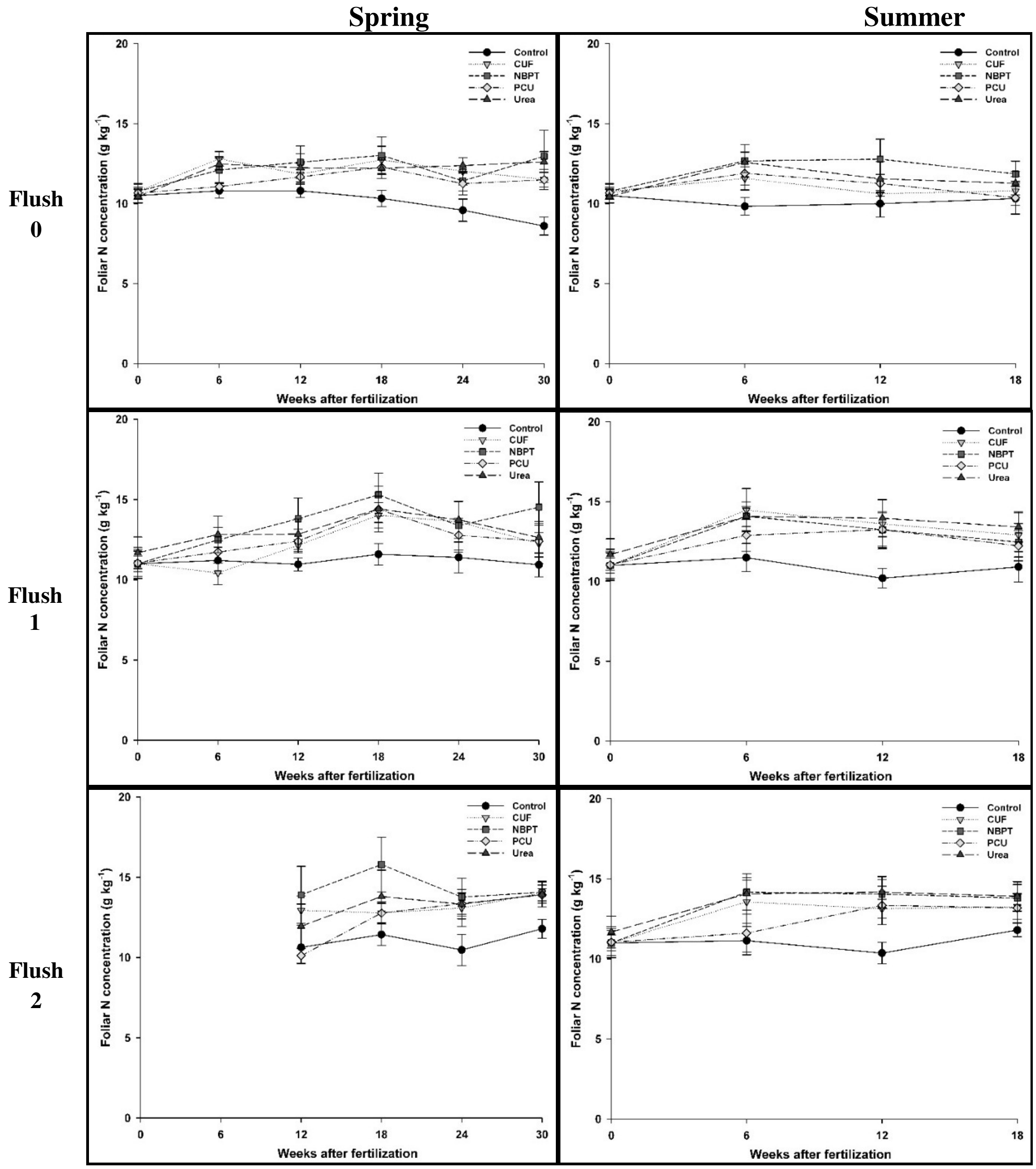

Figure A.3.2. *The* ^{15}*N (‰) values for flush 0, 1, and 2 for loblolly pine stands in the southern United States selected to evaluate temporal uptake of fertilizer nitrogen enriched with* ^{15}*N following a spring and summer application of urea or enhanced efficiency nitrogen fertilizers. The N application rate was 224 kg N ha*$^{-1}$*. N = 6.*

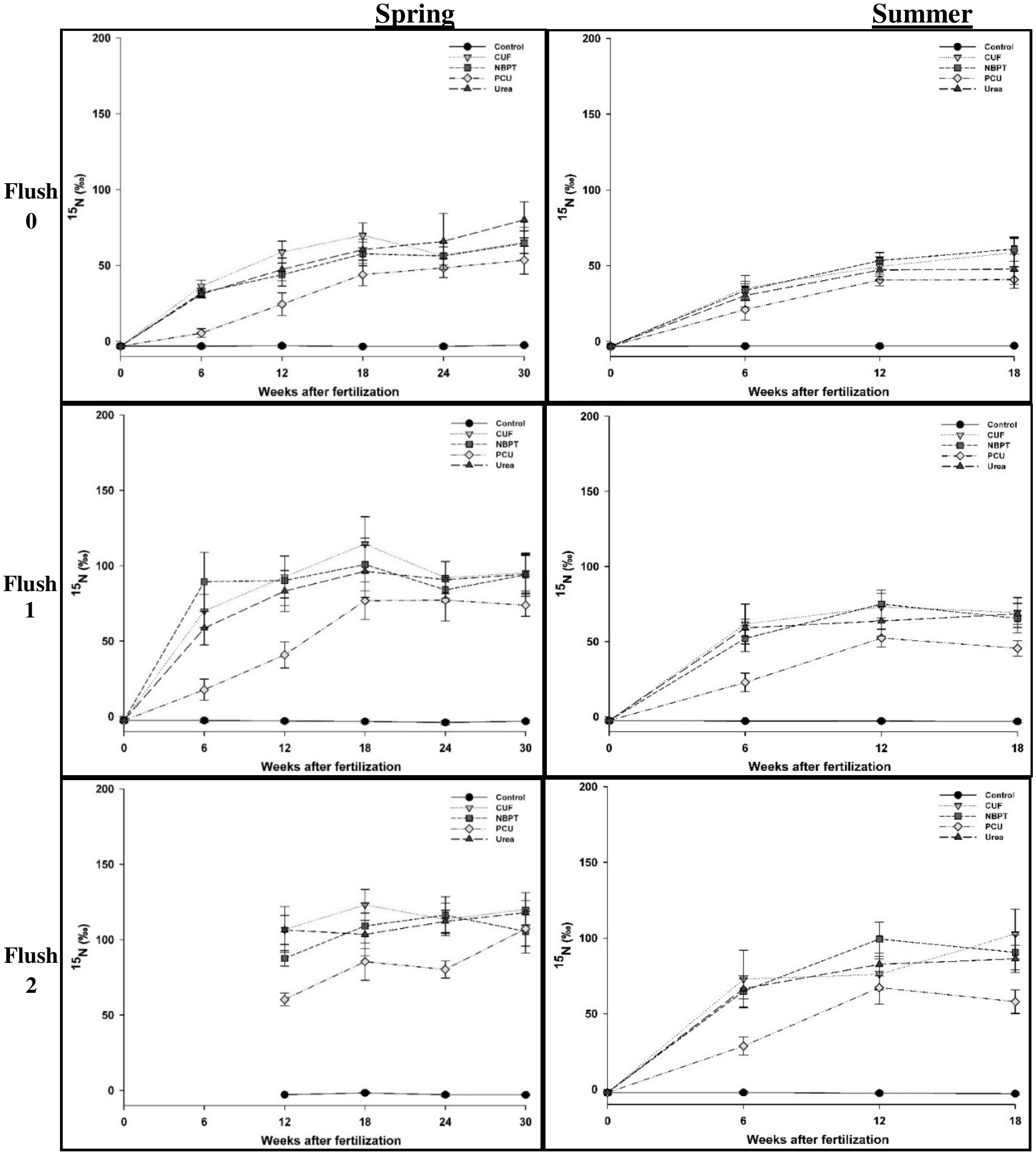

Table A.3.1 *P > | t | values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for N concentration (g kg^{-1}) between individual flushes (0, 1, 2) within sampling week (6, 12, 18, 24, 30) for loblolly pine stands in the southern United States selected to evaluate seasonal trends of fertilizer N uptake for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after a spring (2011) fertilization. Numbers in parentheses are the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 6.*

		CUF	**Control**	**NBPT**	**PCU**	**urea**
Week-Flush				p-value		
6-0	6-1	1.0000	1.0000	1.0000	1.0000	1.0000
6-0	12-0	1.0000	1.0000	1.0000	1.0000	1.0000
6-0	18-0	1.0000	1.0000	1.0000	1.0000	1.0000
6-0	24-0	1.0000	1.0000	1.0000	1.0000	1.0000
6-0	30-0	1.0000	1.0000	1.0000	1.0000	1.0000
6-1	12-1	1.0000	1.0000	1.0000	1.0000	1.0000
6-1	18-1	0.5650	1.0000	0.9779	0.9918	1.0000
6-1	24-1	0.8915	1.0000	1.0000	1.0000	1.0000
6-1	30-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-0	12-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-0	12-2	1.0000	1.0000	1.0000	1.0000	1.0000
12-1	12-2	1.0000	1.0000	1.0000	1.0000	1.0000
12-0	18-0	1.0000	1.0000	1.0000	1.0000	1.0000
12-0	24-0	1.0000	1.0000	1.0000	1.0000	1.0000
12-0	30-0	1.0000	0.9996	1.0000	1.0000	1.0000
12-1	18-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-1	24-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-1	30-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-2	18-2	1.0000	1.0000	1.0000	0.9990	1.0000
12-2	24-2	1.0000	1.0000	1.0000	0.9133	1.0000
12-2	30-2	1.0000	1.0000	1.0000	0.6253	1.0000
18-0	18-1	1.0000	1.0000	1.0000	1.0000	1.0000
18-0	18-2	1.0000	1.0000	1.0000	1.0000	1.0000
18-1	18-2	1.0000	1.0000	1.0000	1.0000	1.0000
18-0	24-0	1.0000	1.0000	1.0000	1.0000	1.0000
18-0	30-0	1.0000	1.0000	1.0000	1.0000	1.0000
18-1	24-1	1.0000	0.9956	1.0000	1.0000	1.0000
18-1	30-1	1.0000	1.0000	1.0000	1.0000	1.0000
18-2	24-2	1.0000	1.0000	1.0000	1.0000	1.0000
18-2	30-2	1.0000	1.0000	1.0000	1.0000	1.0000
24-0	24-1	1.0000	0.9996	1.0000	1.0000	1.0000
24-0	24-2	1.0000	0.9751	0.9972	1.0000	1.0000
24-1	24-2	1.0000	1.0000	1.0000	1.0000	1.0000
24-0	30-0	1.0000	1.0000	1.0000	1.0000	1.0000
24-1	30-1	1.0000	1.0000	1.0000	1.0000	1.0000
24-2	30-2	1.0000	1.0000	1.0000	1.0000	1.0000
30-0	30-1	1.0000	0.9952	1.0000	1.0000	1.0000
30-0	30-2	0.9793	0.6526	1.0000	0.9878	1.0000
30-1	30-2	1.0000	1.0000	1.0000	1.0000	1.0000

Table A.3.2. *P > | t | values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for ^{15}N values (‰) between individual flushes (0, 1, 2) within sampling week (6, 12, 18, 24, 30) for loblolly pine stands in the southern United States selected to evaluate seasonal trends of fertilizer N uptake for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after a spring (2011) fertilization. Numbers in parentheses are the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 6.*

		CUF	**Control**	**NBPT**	**PCU**	**urea**
Week-Flush				p-value		
6-0	6-1	0.9524	1.0000	0.0136	1.0000	0.9993
6-0	12-0	0.9997	1.0000	1.0000	1.0000	1.0000
6-0	18-0	0.6136	1.0000	0.9891	0.2336	0.9024
6-0	24-0	1.0000	1.0000	0.9961	0.0600	0.4856
6-0	30-0	0.6275	1.0000	0.3106	**0.0006**	**0.0004**
6-1	12-1	0.9997	1.0000	1.0000	0.9994	0.9973
6-1	18-1	**0.0438**	1.0000	1.0000	**0.0001**	0.2945
6-1	24-1	0.9997	1.0000	1.0000	**<.0001**	0.7105
6-1	30-1	0.9116	1.0000	1.0000	**<.0001**	0.1212
12-0	12-1	0.9142	1.0000	0.1661	1.0000	0.8264
12-0	12-2	**0.0286**	1.0000	0.1176	0.9703	0.0599
12-1	12-2	0.9998	1.0000	1.0000	1.0000	1.0000
12-0	18-0	1.0000	1.0000	1.0000	0.9997	1.0000
12-0	24-0	1.0000	1.0000	1.0000	0.5962	0.9858
12-0	30-0	1.0000	1.0000	0.9980	0.6342	0.2959
12-1	18-1	0.9954	1.0000	1.0000	0.1319	1.0000
12-1	24-1	1.0000	1.0000	1.0000	**0.0031**	1.0000
12-1	30-1	1.0000	1.0000	1.0000	0.2753	1.0000
12-2	18-2	1.0000	1.0000	1.0000	0.9967	1.0000
12-2	24-2	1.0000	1.0000	1.0000	0.9996	1.0000
12-2	30-2	1.0000	1.0000	1.0000	**0.0297**	1.0000
18-0	18-1	0.6501	1.0000	0.7309	0.9963	0.9775
18-0	18-2	0.1804	1.0000	0.3676	0.8188	0.7544
18-1	18-2	1.0000	1.0000	1.0000	1.0000	1.0000
18-0	24-0	1.0000	1.0000	1.0000	1.0000	1.0000
18-0	30-0	1.0000	1.0000	1.0000	1.0000	1.0000
18-1	24-1	0.9999	1.0000	1.0000	1.0000	1.0000
18-1	30-1	1.0000	1.0000	1.0000	1.0000	1.0000
18-2	24-2	1.0000	1.0000	1.0000	1.0000	1.0000
18-2	30-2	1.0000	1.0000	1.0000	0.9993	1.0000
24-0	24-1	0.9590	1.0000	0.9999	1.0000	1.000
24-0	24-2	**0.0438**	1.0000	**0.0176**	**0.0262**	0.3980
24-1	24-2	1.0000	1.0000	0.9927	1.0000	1.0000
24-0	30-0	1.0000	1.0000	1.0000	1.0000	1.0000
24-1	30-1	1.0000	1.0000	1.0000	1.0000	1.0000
24-2	30-2	1.0000	1.0000	1.0000	0.9738	1.0000
30-0	30-1	0.9989	1.0000	0.9978	1.0000	1.0000
30-0	30-2	0.0790	1.0000	0.7525	0.1001	1.0000
30-1	30-2	1.0000	1.0000	1.0000	0.9873	1.0000

Table A.3.3. *$P > | t |$ values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for N concentration (g kg^{-1}) between individual flushes (0, 1, 2) within sampling week (6, 12, 18) for loblolly pine stands in the southern United States selected to evaluate seasonal trends of fertilizer N uptake for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after a summer (2011) fertilization. Numbers in parentheses are the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 6.*

		CUF	**Control**	**NBPT**	**PCU**	**urea**
Week-Flush				p-value		
6-0	6-1	0.7654	1.0000	1.0000	1.0000	1.0000
6-0	6-2	0.9988	1.0000	1.0000	1.0000	1.0000
6-0	12-0	1.0000	1.0000	1.0000	1.0000	1.0000
6-0	18-0	1.0000	1.0000	1.0000	1.0000	1.0000
6-1	6-2	1.0000	1.0000	1.0000	1.0000	1.0000
6-1	12-1	1.0000	0.9998	1.0000	1.0000	1.0000
6-1	18-1	1.0000	1.0000	0.9996	1.0000	1.0000
6-2	12-2	1.0000	1.0000	1.0000	0.9614	1.0000
6-2	18-2	1.0000	1.0000	1.0000	0.9979	1.0000
12-0	12-1	0.7197	1.0000	1.0000	0.9989	0.9712
12-0	12-2	0.1430	1.0000	1.0000	0.9972	0.9163
12-0	18-0	1.0000	1.0000	1.0000	1.0000	1.0000
12-1	12-2	1.0000	1.0000	1.0000	1.0000	1.0000
12-1	18-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-2	18-2	1.0000	1.0000	1.0000	1.0000	1.0000
18-0	18-1	0.9939	1.0000	1.0000	1.0000	0.9923
18-0	18-2	0.9431	1.0000	1.0000	0.6735	0.8790
18-1	18-2	1.0000	1.0000	1.0000	1.0000	1.0000

Table A.3.4. *P > | t | values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for ^{15}N values (‰) between individual flushes (0, 1, 2) within sampling week (6, 12, 18, 24, 30) for loblolly pine stands in the southern United States selected to evaluate seasonal trends of fertilizer N uptake for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after a spring (2011) fertilization. Numbers in parentheses are the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 6.*

		CUF	**Control**	**NBPT**	**PCU**	**urea**
Week-Flush				p-value		
6-0	6-1	0.8369	1.0000	0.9992	1.0000	0.6771
6-0	6-2	0.1207	1.0000	0.4965	1.0000	1.0000
6-0	12-0	1.0000	1.0000	0.9900	0.9911	0.9995
6-0	18-0	0.6087	1.0000	0.2663	0.9517	0.9826
6-1	6-2	1.0000	1.0000	1.0000	1.0000	1.0000
6-1	12-1	1.0000	1.0000	0.9184	0.4330	1.0000
6-1	18-1	1.0000	1.0000	0.9999	0.4396	1.0000
6-2	12-2	1.0000	1.0000	0.1321	**0.0351**	0.9998
6-2	18-2	0.1242	1.0000	0.3964	**0.0042**	1.0000
12-0	12-1	0.9139	1.0000	0.9698	1.0000	0.9998
12-0	12-2	0.4816	1.0000	**0.0033**	0.7334	0.1299
12-0	18-0	1.0000	1.0000	1.0000	1.0000	1.0000
12-1	12-2	1.0000	1.0000	0.8689	1.0000	0.9969
12-1	18-1	1.0000	1.0000	1.0000	1.0000	1.0000
12-2	18-2	0.5059	1.0000	1.0000	1.0000	1.0000
18-0	18-1	1.0000	1.0000	1.0000	1.0000	0.9967
18-0	18-2	**0.0298**	1.0000	0.7010	0.8810	0.1391
18-1	18-2	0.4329	1.0000	0.9393	0.9997	0.9998

Table A.3.5. *P > | t | values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for mean foliar N concentration (%) and mean foliar ^{15}N (‰) for individual flushes (0, 1, 2) for individual sampling weeks (6, 12, 18, 24, 30) after a spring fertilization (2011) for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. The N application rate was 224 kg N ha^{-1}. N = 6.*

	Week															
	6		**12**			**18**			**24**				**30**			
	Flush															
	0	**1**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Contrasts	**N concentration (%)**															
Control-CUF	0.9999	1.0000	1.0000	1.0000	-	0.9999	0.9999	1.0000	0.9517	0.9913	0.8749	-	0.8072	1.0000	0.9960	-
Control-NBPT	1.0000	1.0000	1.0000	0.9569	-	0.9977	0.6614	**0.0359**	0.9999	0.9994	0.2842	-	**0.0212**	0.2497	0.9935	-
Control-PCU	1.0000	1.0000	1.0000	1.0000	-	1.0000	0.9942	1.0000	1.0000	1.0000	0.6509	-	0.8245	1.0000	0.9985	-
Control-urea	1.0000	1.0000	1.0000	1.0000	-	1.0000	0.9931	1.0000	0.7392	0.9757	0.6864	-	**0.0439**	1.0000	0.9984	-
CUF-NBPT	1.0000	0.9997	1.0000	1.0000	-	1.0000	1.0000	0.9996	1.0000	1.0000	1.0000	-	1.0000	0.9973	1.0000	-
CUF-PCU	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
CUF-urea	1.0000	0.9862	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
NBPT-PCU	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	0.9996	1.0000	1.0000	1.0000	-	1.0000	0.9995	1.0000	-
NBPT-urea	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
PCU-urea	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
	^{15}N (‰)															
Control-CUF	0.6786	**0.0002**	**0.0014**	**<.0001**	-	**0.0007**	**<.0001**	**<.0001**	**0.0168**	**<.0001**	**<.0001**	-	**0.0017**	**<.0001**	**<.0001**	-
Control-NBPT	0.9083	**<.0001**	0.1508	**<.0001**	-	**0.0283**	**<.0001**	**<.0001**	**0.0187**	**<.0001**	**<.0001**	-	**0.0022**	**<.0001**	**<.0001**	-
Control-PCU	1.0000	1.0000	0.9983	0.2806	-	0.4625	**<.0001**	**<.0001**	0.1403	**<.0001**	**<.0001**	-	**0.0546**	**<.0001**	**<.0001**	-
Control-urea	0.9373	**0.0080**	0.0598	**<.0001**	-	**0.0137**	**<.0001**	**<.0001**	**0.0009**	**<.0001**	**<.0001**	-	**<.0001**	**<.0001**	**<.0001**	-
CUF-NBPT	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
CUF-PCU	0.9897	0.0621	0.8939	**0.0414**	-	1.0000	0.9512	0.9485	1.0000	1.0000	0.9875	-	1.0000	1.0000	1.0000	-
CUF-urea	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
NBPT-PCU	0.9997	**<.0001**	1.0000	0.0793	-	1.0000	1.0000	1.0000	1.0000	1.0000	0.9448	-	1.0000	1.0000	1.0000	-
NBPT-urea	1.0000	0.9907	1.0000	1.0000	-	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
PCU-urea	0.9999	0.5909	1.0000	0.3810	-	1.0000	1.0000	1.0000	1.0000	1.0000	0.9944	-	1.0000	1.0000	1.0000	-

Table A.3.6. *P > | t | values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for the mean individual fascicle mass (g) and mean N content (mg) for individual flushes (0, 1, 2) for individual sampling weeks (6, 12, 18, 24, 30) after a spring fertilization (2011) for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. The N application rate was 224 kg N ha^{-1}. N = 6.*

Week	6		12			18			24				30			
Flush	0	1	0	1	2	0	1	2	0	1	2	3	0	1	2	3
Contrasts	**Individual Fascicle Mass (g)**															
Control-CUF	0.6206	0.2566	0.1204	0.9018	0.7416	0.6295	**0.0029**	**0.0181**	0.8945	**0.0043**	**0.0138**	-	0.4461	**0.0367**	**0.0003**	**-**
Control-NBPT	0.7835	0.2195	0.2138	0.5365	0.5525	0.9640	**0.0028**	0.0669	0.2996	0.0632	**0.0072**	-	0.5450	**0.0027**	**0.0081**	**-**
Control-PCU	0.3507	0.5927	0.2545	0.3761	0.8515	0.2929	**0.0027**	0.1504	0.2785	**0.0422**	0.1543	-	0.7613	**0.0063**	0.1717	-
Control-urea	0.8911	0.2023	0.1065	0.6407	0.8237	0.9165	**0.0030**	**0.0320**	0.1925	0.2153	0.0770	-	0.8272	**0.0088**	**0.0037**	**-**
CUF-NBPT	0.8252	0.9215	0.7503	0.6205	0.7457	0.5979	0.9910	0.6601	0.3650	0.2952	0.8103	-	0.8750	0.3359	0.3087	-
CUF-PCU	0.6588	0.5317	0.6718	0.4456	0.8371	0.1269	0.9820	0.3375	0.3409	0.3853	0.2829	-	0.6461	0.4978	**0.0207**	**-**
CUF-urea	0.7199	0.8819	0.3744	0.7312	0.8727	0.7055	0.9910	0.9081	0.2410	0.0952	0.4687	-	0.3278	0.5751	0.4546	-
NBPT-PCU	0.5084	0.4696	0.9159	0.7880	0.5756	0.3139	0.9910	0.6347	0.9626	0.8571	0.1900	-	0.7625	0.7748	0.1851	-
NBPT-urea	0.8903	0.9600	0.2294	0.8792	0.6071	0.8808	0.9820	0.7564	0.7876	0.5273	0.3356	-	0.4108	0.6864	0.7856	-
PCU-urea	0.4249	0.4396	0.1917	0.6740	0.9598	0.2480	0.9730	0.4247	0.8239	0.4172	0.7250	-	0.6019	0.9062	0.5107	-
	Individual Fascicle N content (mg)															
Control-CUF	0.2760	0.4598	0.4359	0.6096	0.8296	0.1704	**0.0013**	**0.0212**	0.2376	**0.0010**	**0.0068**	-	0.0821	**0.0502**	**0.0001**	**-**
Control-NBPT	0.4792	0.0887	0.7430	0.1137	0.5185	0.2843	**0.0002**	**0.0031**	0.8384	**0.0201**	**0.0011**	**-**	**0.0153**	**<.0001**	**0.0018**	**-**
Control-PCU	0.3771	0.5126	0.5239	0.2508	0.8952	0.9541	**0.0007**	0.0879	0.9127	**0.0312**	**0.0465**	-	0.1676	**0.0137**	**0.0514**	**-**
Control-urea	0.4868	0.0990	0.1945	0.2829	0.9188	0.3376	**0.0007**	**0.0099**	0.7243	**0.0474**	**0.0248**	-	0.1384	**0.0046**	**0.0010**	**-**
CUF-NBPT	0.6985	0.3087	0.6502	0.2764	0.5983	0.7599	0.6016	0.4144	0.3273	0.2974	0.5430	-	0.4733	**0.0229**	0.4171	-
CUF-PCU	0.8339	0.9298	0.8862	0.5187	0.6510	0.1886	0.8482	0.5301	0.2833	0.2231	0.4473	-	0.7133	0.5942	**0.0423**	**-**
CUF-urea	0.6895	0.3362	0.5970	0.5690	0.8743	0.6754	0.8341	0.6858	0.4054	0.1628	0.6213	-	0.7921	0.3554	0.5441	-
NBPT-PCU	0.8588	0.2695	0.7560	0.6531	0.3071	0.3106	0.7405	0.1597	0.9248	0.8581	0.1736	-	0.2794	0.0778	0.2151	-
NBPT-urea	0.9903	0.9544	0.3283	0.5993	0.4654	0.9098	0.7542	0.6925	0.8816	0.7194	0.2722	-	0.3276	0.1677	0.8369	-
PCU-urea	0.8492	0.2945	0.5023	0.9392	0.7435	0.3672	0.9856	0.3175	0.8070	0.8567	0.7895	-	0.9173	0.6940	0.1493	-

Table A.3.7. *P > ǀ t ǀ values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for the fertilizer N (mg) in individual fascicles (mg) for individual flushes (0, 1, 2) for week sampled (6, 12, 18, 24, 30) in loblolly pine stands of the southern United States selected to evaluate fertilizer N uptake for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N after a spring (2011) fertilization. Numbers in parentheses are standard error of mean. N application rate was 224 kg N ha^{-1}. N = 6.*

	Week															
Spring	**6**		**12**			**18**			**24**				**30**			
	Flush															
	0	**1**	**0**	**1**	**2**	**0**	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Contrasts																
Control-CUF	0.0914	**0.0131**	**0.0013**	**<.0001**	-	**0.0088**	**<.0001**	**<.0001**	**0.0306**	**<.0001**	**<.0001**	-	**0.0184**	**<.0001**	**<.0001**	**-**
Control-NBPT	0.1666	**<.0001**	**0.0073**	**<.0001**	-	**0.0286**	**<.0001**	**<.0001**	**0.0441**	**<.0001**	**<.0001**	-	**0.0030**	**<.0001**	**<.0001**	**-**
Control-PCU	0.8127	0.5196	0.1382	**0.0064**	-	0.1221	**<.0001**	**0.0011**	0.0854	**<.0001**	**0.0009**	-	**0.0394**	**0.0003**	**<.0001**	-
Control-urea	0.1528	**0.0056**	**0.0161**	**<.0001**	-	**0.0219**	**<.0001**	**<.0001**	**0.0223**	**<.0001**	**<.0001**	-	**0.0029**	**<.0001**	**<.0001**	**-**
CUF-NBPT	0.7515	**0.0162**	0.5421	0.2149	-	0.6447	0.8918	0.7727	0.8762	0.4131	0.2701	-	0.5125	0.1232	0.3026	-
CUF-PCU	0.1442	**0.0517**	**0.0623**	0.0606	-	0.2606	0.2098	0.0872	0.6463	0.2839	0.1054	-	0.7552	0.6161	0.0784	**-**
CUF-urea	0.7883	0.7420	0.3604	0.5082	-	0.7249	0.8157	0.0843	0.8970	0.4972	0.8087	-	0.5090	0.2708	0.7648	-
NBPT-PCU	0.2493	**<.0001**	0.2020	**0.0026**	-	0.5044	0.1653	0.0547	0.7616	0.7985	**0.0075**	-	0.3346	**0.0426**	0.4580	-
NBPT-urea	0.9615	**0.0356**	0.7583	0.5581	-	0.9128	0.7121	0.6059	0.7755	0.8884	0.1798	-	0.9957	0.4294	0.4630	-
PCU-urea	0.2304	**0.0243**	0.3301	**0.0127**	-	0.4375	0.3058	0.1614	0.5565	0.6925	0.1667	-	0.3320	0.2088	0.1420	-

Table A.3.8. *P > | t | values for fertilizer N treatments (control, CUF, NBPT, PCU, urea) contrasts for mean foliar N concentration (g kg^{-1}) and mean foliar ^{15}N (‰) for individual flushes (0, 1, 2) for individual sampling weeks (6, 12, 18) after a summer fertilization (2011) for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. The N application rate was 224 kg N ha^{-1}. N = 6.*

	Week										
	6			**12**				**18**			
	Flush										
	0	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Contrasts	**N concentration (g kg^{-1})**										
Control-CUF	0.9999	0.7097	0.9663	1.0000	0.4126	0.0726	-	1.0000	0.9977	1.0000	-
Control-NBPT	0.8032	0.9212	0.6707	0.8559	0.6778	0.2368	-	1.0000	1.0000	0.9968	-
Control-PCU	0.9975	1.0000	1.0000	1.0000	0.6772	0.7221	-	1.0000	1.0000	0.9998	-
Control-urea	0.8549	0.9259	0.7479	1.0000	0.2016	**0.0484**	-	1.0000	0.9282	0.9921	-
CUF-NBPT	1.0000	1.0000	1.0000	0.9945	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
CUF-PCU	1.0000	1.0000	0.9991	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
CUF-urea	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
NBPT-PCU	1.0000	1.0000	0.9282	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
NBPT-urea	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
PCU-urea	1.0000	1.0000	0.9582	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
	^{15}N (‰)										
Control-CUF	0.1028	**<.0001**	**<.0001**	**0.0002**	**<.0001**	**<.0001**	**-**	**<.0001**	**<.0001**	**<.0001**	**-**
Control-NBPT	0.1540	**0.0002**	**<.0001**	**<.0001**	**<.0001**	**<.0001**	**-**	**<.0001**	**<.0001**	**<.0001**	**-**
Control-PCU	0.9407	0.8772	0.5334	**0.0085**	**<.0001**	**<.0001**	**-**	**0.0432**	**0.0022**	**<.0001**	**-**
Control-urea	0.3323	**<.0001**	**<.0001**	**0.0006**	**<.0001**	**<.0001**	**-**	**0.0028**	**<.0001**	**<.0001**	**-**
CUF-NBPT	1.0000	1.0000	1.0000	1.0000	1.0000	0.9912	-	1.0000	1.0000	1.0000	-
CUF-PCU	1.0000	0.0879	**0.0156**	1.0000	0.9848	1.0000	-	0.9994	0.9945	0.2432	-
CUF-urea	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	-	1.0000	1.0000	1.0000	-
NBPT-PCU	1.0000	0.6456	0.1769	1.0000	0.9481	0.3059	-	0.9953	0.9999	0.9634	-
NBPT-urea	1.0000	1.0000	1.0000	1.0000	1.0000	0.9997	-	1.0000	1.0000	1.0000	-
PCU-urea	1.0000	0.1723	0.1149	1.0000	1.0000	1.0000	-	1.0000	0.9976	0.9985	-

Table A.3.9. *P > | t | values for fertilizer N treatment (control, CUF, NBPT, PCU, urea) contrasts for the mean individual fascicle mass (g) and N content (mg) for individual flushes (0, 1, 2) for individual sampling weeks (6, 12, 18) after a summer fertilization (2011) for loblolly pine stands in the southern United States selected to evaluate fertilizer N uptake of urea or enhanced efficiency N containing fertilizers enriched with* ^{15}N*. The N application rate was 224 kg N* ha^{-1}*. N = 6.*

Week

	6			**12**				**18**			
					Flush						
	0	**1**	**2**	**0**	**1**	**2**	**3**	**0**	**1**	**2**	**3**
Contrasts					**Individual Fascicles Mass (g)**						
Control-CUF	0.4466	0.0959	**<.0001**	0.3709	0.1007	0.1343	-	0.9959	**0.0419**	0.7713	-
Control-NBPT	0.7373	**0.0002**	**<.0001**	0.7464	**0.0151**	**0.0146**	-	0.9171	0.0978	**0.0176**	-
Control-PCU	0.4095	**0.0190**	**0.0009**	0.9868	0.1085	0.1496	-	0.5306	**0.0537**	0.2212	-
Control-urea	0.7763	**0.0006**	**0.0009**	0.8001	**0.0089**	**0.0080**	-	0.2728	**0.0090**	**0.0062**	-
CUF-NBPT	0.6694	**0.0283**	0.9833	0.2249	0.4060	0.3220	-	0.9212	0.6941	0.0359	-
CUF-PCU	0.9488	0.4760	0.2463	0.3797	0.9701	0.9539	-	0.5272	0.9125	0.3491	-
CUF-urea	0.6326	0.0572	0.2593	0.2523	0.3041	0.2248	-	0.2750	0.5405	**0.0137**	-
NBPT-PCU	0.6234	0.1318	0.2548	0.7340	0.3854	0.2950	-	0.4650	0.7768	0.2351	-
NBPT-urea	0.9591	0.7599	0.2681	0.9440	0.8424	0.8204	-	0.3203	0.3160	0.6967	-
PCU-urea	0.5877	0.2272	0.9746	0.7874	0.2871	0.2039	-	0.0871	0.4706	0.1166	-
					Individual Fascicle N Content (mg)						
Control-CUF	0.1263	**0.0021**	**0.0001**	0.7462	**0.0042**	**0.0244**	-	0.7193	**0.0148**	**0.0403**	-
Control-NBPT	**0.0497**	**0.0001**	**<.0001**	**0.0105**	**0.0017**	**0.0007**	-	0.4571	0.0623	**0.0060**	-
Control-PCU	0.0752	**0.0248**	**0.0419**	0.5903	**0.0105**	**0.0230**	-	0.8285	0.0725	**0.0117**	-
Control-urea	0.0576	**0.0003**	**0.0005**	0.4643	**0.0003**	**0.0004**	-	0.2517	**0.0026**	**0.0019**	-
CUF-NBPT	0.6533	0.4052	0.6687	**0.0508**	0.7649	0.2128	-	0.7104	0.5438	**0.0495**	-
CUF-PCU	0.7958	0.3700	**0.0540**	0.3897	0.7395	0.9811	-	0.5650	0.4980	0.4456	-
CUF-urea	0.7015	0.5562	0.7016	0.2926	0.4001	0.1488	-	0.4289	0.5260	**0.0195**	-
NBPT-PCU	0.8488	0.0866	**0.0197**	0.2645	0.5281	0.2215	-	0.3451	0.9434	0.2206	-
NBPT-urea	0.9473	0.8062	0.4183	0.3555	0.5864	0.8395	-	0.6735	0.2168	0.6929	-
PCU-urea	0.9009	0.1400	0.1194	0.8459	0.2420	0.1554	-	0.1735	0.1921	0.1075	-

Chapter 4

Figure A.4.1. *The fertilizer N recovery (% of fertilizer N applied) of the major ecosystem components (loblolly pine aboveground biomass, loblolly pine roots, litterfall, O horizon, mineral soil) for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N of urea or enhanced efficiency N fertilizers enriched with* ^{15}N*. Data represents spring application date (March-April 2011) for Study 1. Different letters represent significant differences at* $\alpha = 0.05$*. The N application rate was 224 kg N* ha^{-1}*. N = 5.*

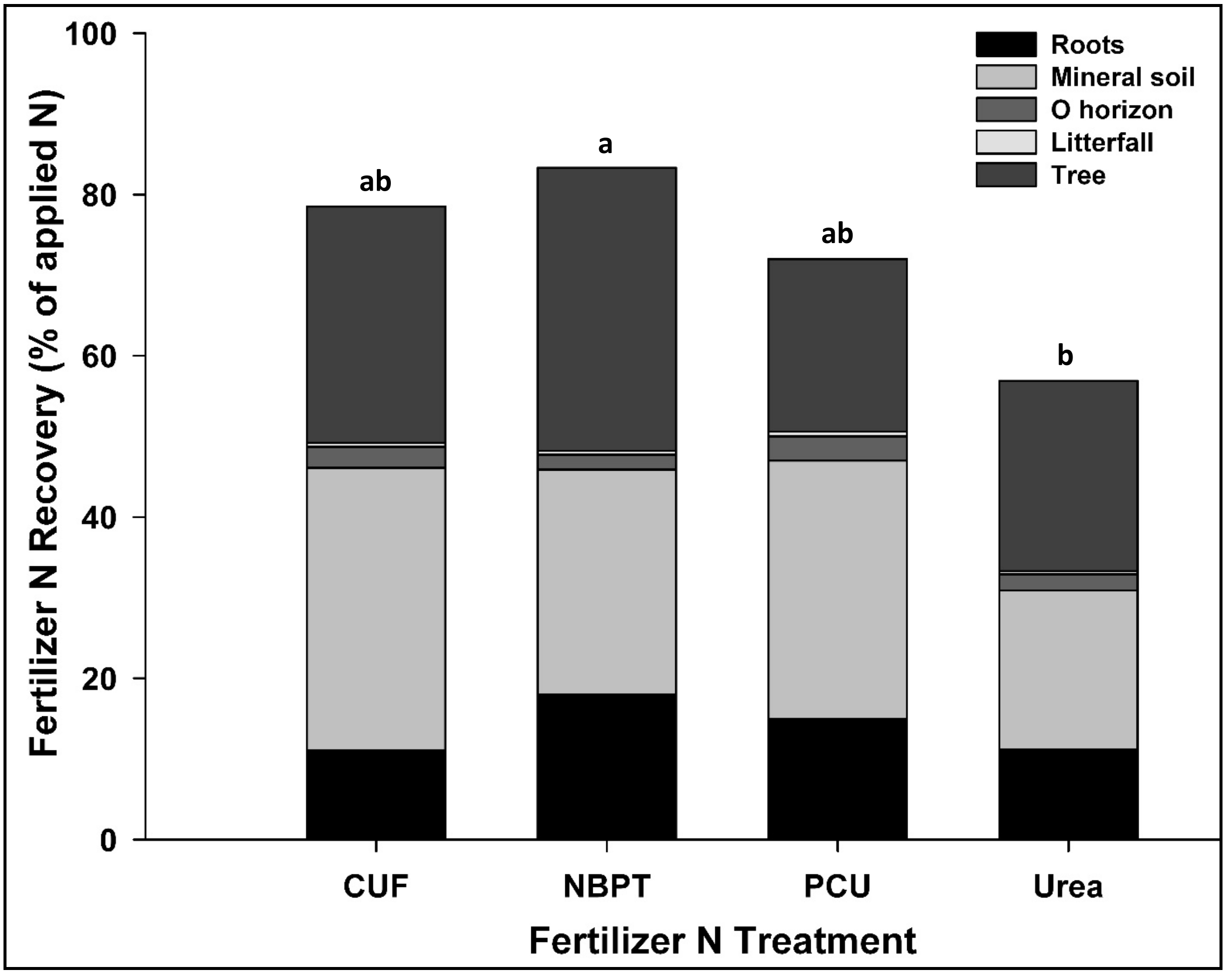

Figure A.4.2. *The fertilizer N recovery (% of fertilizer N applied) of the major ecosystem components (loblolly pine aboveground biomass, loblolly pine roots, litterfall, O horizon, mineral soil) for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N of urea or enhanced efficiency N fertilizers enriched with* ^{15}*N. Data represents summer application date (June 2011) for Study 1. Different letters represent significant differences at* $\alpha = 0.05$*. The N application rate was 224 kg N ha*$^{-1}$*. N=5.*

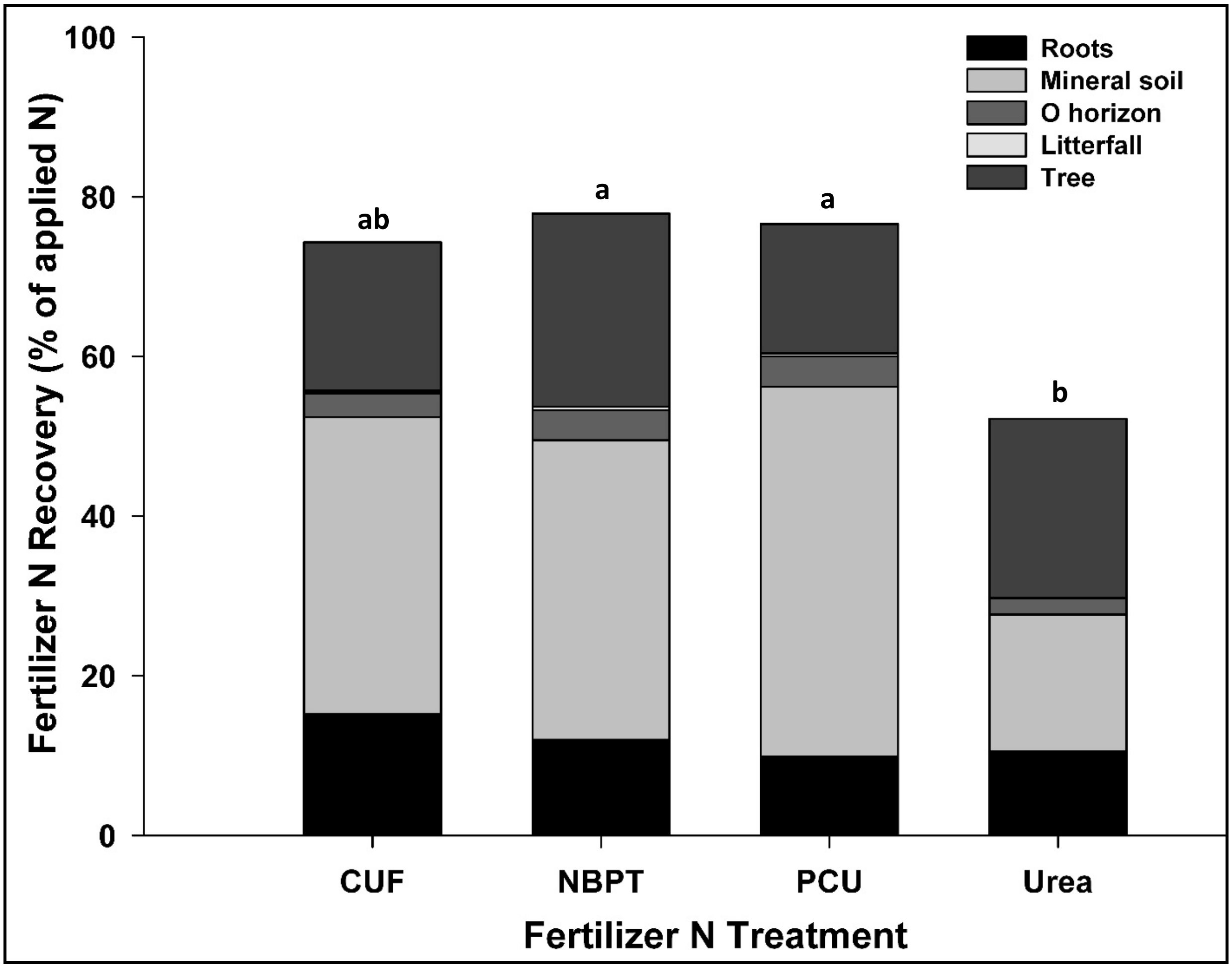

Table A.4.1. *Analysis of variance for treatment differences for individual component fertilizer N recovery (% of fertilizer N applied) for Study 1 (spring 2011) at mid-rotation loblolly pine plantations in the southern United States. N application rate was 224 kg N ha^{-1}. N = 10.*

Source	Degrees of Freedom	Adj. Sum of Squares	Adj. Mean Squares	F-Value	P-Value
Total Ecosystem Fertilizer N Recovery					
Treatment	4	19094	4773.6	12.52	**0.000**
Error	20	7624	381.2		
Total	24	26718			
Loblolly pine tree (foliage + fine branch + coarse branch + bark + CGR + PGR + fine roots + coarse roots					
Treatment	4	5413	1353.2	11.31	**0.000**
Error	20	2392	119.6		
Total	24	7805			
Soil (Organic Horizon + 0-15 cm mineral soil + 15-30 cm mineral soil)					
Treatment	4	4569	1142.3	7.46	**0.001**
Error	20	3064	153.2		
Total	24	7633			
Foliage					
Treatment	4	880.4	220.09	5.05	**0.006**
Error	20	871.0	43.55		
Total	24				
Fine Branches					
Treatment	4	14.87	3.7178	4.72	**0.008**
Error	20	15.76	0.7880		
Total	24	30.63			
Coarse Branches					
Treatment	4	154.98	38.745	12.76	**0.000**
Error	20	60.71	3.035		
Total	24				
Bark					
Treatment	4	5.263	1.3158	1.93	0.145
Error	20	13.641	0.6821		
Total	24	18.904			
Current Growth Ring					
Treatment	4	15.31	3.8272	6.16	**0.002**
Error	20	12.43	0.6217		
Total	24	27.74			
Previous Growth Rings					
Treatment	4	100.8	25.188	4.87	**0.007**
Error	20	103.4	5.172		
Total	24	204.2			
Fine Roots (< 2 mm)					
Treatment	4	638.7	159.66	5.38	**0.004**
Error	20	593.9	29.69		
Total	24	1232.5			
Coarse Roots (> 2 mm)					
Treatment	4	33.46	8.364	2.15	0.112
Error	20	77.72	3.886		
Total	24	111.18			
Litterfall					
Treatment	4	1.054	0.2634	0.93	0.468
Error	20	5.684	0.2842		
Total	24	6.737			

Source	Degrees of Freedom	Adj. Sum of Squares	Adj. Mean Squares	F-Value	P-Value
Organic Horizon (Oi + Oe + Oa)					
Treatment	4	26.08	6.519	1.86	0.158
Error	20	70.24	3.512		
Total	24	96.32			
0-15 cm Mineral Soil					
Treatment	4	2651	662.8	5.88	**0.003**
Error	20	2255	112.7		
Total	24	4906			
15-30 cm Mineral Soil					
Treatment	4	130.7	32.67	3.03	**0.042**
Error	20	215.5	10.77		
Total	24	346.2			

Table A.4.2. *Analysis of variance for treatment differences for individual component fertilizer N recovery (% of fertilizer N applied) for Study 1 (summer 2011) at mid-rotation loblolly pine plantations in the southern United States. N application rate was 224 kg N ha^{-1}. N = 10.*

Source	Degrees of Freedom	Adj. Sum of Squares	Adj. Mean Squares	F-Value	P-Value
Total Ecosystem Fertilizer N Recovery					
Treatment	4	18226	4556.5	9.40	**0.000**
Error	20	9694	484.7		
Total	24	27920			
Loblolly pine tree (foliage + fine branch + coarse branch + bark + CGR + PGR + fine roots + coarse roots					
Treatment	4	2420	605.10	6.71	**0.001**
Error	20	1804	90.22		
Total	24	4225			
Soil (Organic Horizon + 0-15 cm mineral soil + 15-30 cm mineral soil)					
Treatment	4	8243	2060.8	8.43	**0.000**
Error	20	4887	244.3		
Total	24	13130			
Foliage					
Treatment	4	466.2	116.56	3.12	**0.038**
Error	20	746.8	37.34		
Total	24				
Fine Branches					
Treatment	4	7.269	1.8173	2.66	**0.063**
Error	20	13.655	0.6827		
Total	24	20.924			
Coarse Branches					
Treatment	4	60.63	15.159	4.94	**0.006**
Error	20	61.35	3.067		
Total	24				
Bark					
Treatment	4	4.851	1.2128	2.32	0.092
Error	20	10.464	0.5232		
Total	24	15.315			
Current Growth Ring					
Treatment	4	14.42	3.604	2.71	0.059
Error	20	26.57	1.328		
Total	24	40.99			
Previous Growth Ring					
Treatment	4	77.85	19.462	3.04	**0.041**
Error	20	128.01	6.400		
Total	24	205.86			
Fine Roots (< 2 mm)					
Treatment	4	196.3	49.09	1.01	0.424
Error	20	968.4	48.42		
Total	24	1164.7			
Coarse Roots (< 2 mm)					
Treatment	4	9.520	2.3799	3.97	**0.016**
Error	20	11.995	0.5997		
Total	24	21.514			
Litterfall					
Treatment	4	0.5212	0.13031	1.46	0.251
Error	20	1.7824	0.08912		
Total	24	2.3037			

Source	**Degrees of Freedom**	**Adj. Sum of Squares**	**Adj. Mean Squares**	**F-Value**	**P-Value**
Organic Horizon					
Treatment	4	50.54	12.636	2.04	0.127
Error	20	123.69	6.184		
Total	24	174.23			
0-15 cm Mineral Soil					
Treatment	4	4473	1118.2	7.66	**0.001**
Error	20	2918	145.9		
Total	24	7391			
15-30 cm Mineral Soil					
Treatment	4	354.5	88.63	1.22	0.335
Error	20	1458.6	72.93		
Total	24	1813.1			

Table A.4.3. *P > | t | values for fertilizer N treatment (CUF, NBPT, PCU, urea) contrasts for primary individual component fertilizer N recovery (% of fertilizer N applied) for Study 1 (spring and summer 2011) at mid-rotation loblolly pine plantations in the southern United States. The N application rate was 224 kg N ha^{-1}. N =5.*

Differences in Fertilizer N Treatments	Spring 2011 Adjusted P-Value	Summer 2011 Adjusted P-Value
Total Ecosystem Fertilizer N Recovery		
Control – CUF	**0.000**	**0.001**
Control – NBPT	**0.000**	**0.001**
Control – PCU	**0.000**	**0.000**
Control - Urea	**0.006**	**0.044**
CUF - NBPT	1.000	0.997
CUF - PCU	0.967	0.972
CUF - Urea	0.322	0.536
NBPT - PCU	0.960	0.999
NBPT - Urea	0.304	0.344
PCU - Urea	0.682	0.231
Loblolly Pine Tree		
Control – CUF	**0.000**	**0.006**
Control – NBPT	**0.000**	**0.002**
Control – PCU	**0.003**	**0.010**
Control - Urea	**0.007**	**0.008**
CUF - NBPT	0.746	0.973
CUF - PCU	0.934	0.999
CUF - Urea	0.757	1.000
NBPT - PCU	0.307	0.915
NBPT - Urea	0.157	0.952
PCU - Urea	0.994	1.000
Litterfall		
Control – CUF	0.673	0.643
Control – NBPT	0.661	0.307
Control – PCU	0.387	0.329
Control - Urea	0.787	0.961
CUF - NBPT	1.000	0.973
CUF - PCU	0.987	0.980
CUF - Urea	1.000	0.951
NBPT - PCU	0.989	1.000
NBPT - Urea	0.999	0.684
PCU - Urea	0.956	0.712
Soil		
Control – CUF	**0.001**	**0.005**
Control – NBPT	**0.009**	**0.004**
Control – PCU	**0.002**	**0.001**
Control - Urea	0.078	0.327
CUF - NBPT	0.848	1.000
CUF - PCU	0.997	0.857
CUF - Urea	0.290	0.249
NBPT - PCU	0.960	0.901
NBPT - Urea	0.845	0.208
PCU-Urea	0.462	**0.039**

Table A.4.4. *P > | t | values for fertilizer N treatment (CUF, NBPT, PCU, urea) contrasts for individual component fertilizer N recovery (% of fertilizer N applied) for Study 1 (spring + summer 2011) at mid-rotation loblolly pine plantations in the southern United States. The N application rate was 224 kg N* ha^{-1}*. N =10.*

Differences in Fertilizer N Treatments	Adjusted P-Value	Differences in Fertilizer N Treatments	Adjusted P-Value	Differences in Fertilizer N Treatments	Adjusted P-Value
Foliage		***Current Growth Ring***		***Litterfall***	
Control – CUF	**0.001**	Control – CUF	**0.002**	Control – CUF	0.332
Control – NBPT	**0.000**	Control – NBPT	**0.000**	Control – NBPT	0.190
Control – PCU	**0.017**	Control – PCU	**0.003**	Control – PCU	0.083
Control - Urea	**0.001**	Control - Urea	**0.000**	Control - Urea	0.657
CUF - NBPT	0.795	CUF - NBPT	0.981	CUF - NBPT	0.998
CUF - PCU	0.880	CUF - PCU	1.000	CUF - PCU	0.951
CUF - Urea	1.000	CUF - Urea	0.835	CUF - Urea	0.982
NBPT - PCU	0.255	NBPT - PCU	0.950	NBPT - PCU	0.994
NBPT - Urea	0.875	NBPT - Urea	0.988	NBPT - Urea	0.908
PCU - Urea	0.802	PCU - Urea	0.745	PCU - Urea	0.714
Fine Branches		***Previous Growth Ring***		***Organic Horizon***	
Control – CUF	**0.000**	Control – CUF	**0.023**	Control – CUF	**0.043**
Control – NBPT	**0.000**	Control – NBPT	**0.000**	Control – NBPT	**0.041**
Control – PCU	**0.046**	Control – PCU	**0.002**	Control – PCU	**0.008**
Control - Urea	**0.016**	Control - Urea	0.101	Control - Urea	0.253
CUF - NBPT	0.995	CUF - NBPT	0.550	CUF - NBPT	1.000
CUF - PCU	0.464	CUF - PCU	0.885	CUF - PCU	0.971
CUF - Urea	0.715	CUF - Urea	0.973	CUF - Urea	0.917
NBPT - PCU	0.254	NBPT - PCU	0.974	NBPT - PCU	0.974
NBPT - Urea	0.470	NBPT - Urea	0.222	NBPT - Urea	0.912
PCU - Urea	0.994	PCU - Urea	0.544	PCU - Urea	0.594
Coarse Branches		***Fine Roots (< 2 mm)***		***0-15 cm Mineral Soil***	
Control – CUF	**0.000**	Control – CUF	**0.024**	Control – CUF	**0.000**
Control – NBPT	**0.000**	Control – NBPT	**0.005**	Control – NBPT	**0.000**
Control – PCU	**0.004**	Control – PCU	**0.027**	Control – PCU	**0.000**
Control - Urea	**0.001**	Control - Urea	0.061	Control - Urea	0.073
CUF - NBPT	0.996	CUF - NBPT	0.981	CUF - NBPT	0.999
CUF - PCU	0.733	CUF - PCU	1.000	CUF - PCU	0.960
CUF - Urea	0.985	CUF - Urea	0.996	CUF - Urea	**0.045**
NBPT - PCU	0.506	NBPT - PCU	0.974	NBPT - PCU	0.880
NBPT - Urea	0.899	NBPT - Urea	0.883	NBPT - Urea	0.082
PCU - Urea	0.953	PCU - Urea	0.997	PCU - Urea	**0.007**
Bark		***Coarse Roots (> 2 mm)***		***15-30 cm Mineral Soil***	
Control – CUF	0.102	Control – CUF	**0.039**	Control – CUF	**0.041**
Control – NBPT	**0.022**	Control – NBPT	**0.011**	Control – NBPT	0.207
Control – PCU	**0.019**	Control – PCU	0.179	Control – PCU	0.058
Control - Urea	0.338	Control - Urea	0.769	Control - Urea	0.389
CUF - NBPT	0.968	CUF - NBPT	0.987	CUF - NBPT	0.944
CUF - PCU	0.958	CUF - PCU	0.958	CUF - PCU	1.000
CUF - Urea	0.968	CUF - Urea	0.401	CUF - Urea	0.790
NBPT - PCU	1.000	NBPT - PCU	0.761	NBPT - PCU	0.974
NBPT - Urea	0.706	NBPT - Urea	0.173	NBPT - Urea	0.995
PCU - Urea	0.676	PCU - Urea	0.812	PCU - Urea	0.860

Table A.4.5. *P > | t | values for fertilizer N treatment (CUF, NBPT, PCU, urea) contrasts for individual component fertilizer N recovery (% of fertilizer N applied) for Study 1 (spring 2011) at mid-rotation loblolly pine plantations in the southern United States. The N application rate was 224 kg N ha^{-1}. N =5.*

Differences in Fertilizer N Treatments	Adjusted P-Value	Differences in Fertilizer N Treatments	Adjusted P-Value	Differences in Fertilizer N Treatments	Adjusted P-Value
Foliage		***Current Growth Ring***		***Litterfall***	
Control – CUF	**0.029**	Control – CUF	**0.013**	Control – CUF	0.673
Control – NBPT	**0.004**	Control – NBPT	**0.003**	Control – NBPT	0.661
Control – PCU	0.170	Control – PCU	**0.014**	Control – PCU	0.387
Control - Urea	**0.028**	Control - Urea	**0.006**	Control - Urea	0.787
CUF - NBPT	0.896	CUF - NBPT	0.963	CUF - NBPT	1.000
CUF - PCU	0.891	CUF – PCU	1.000	CUF - PCU	0.987
CUF - Urea	1.000	CUF – Urea	0.998	CUF - Urea	1.000
NBPT - PCU	0.401	NBPT - PCU	0.953	NBPT - PCU	0.989
NBPT - Urea	0.899	NBPT - Urea	0.997	NBPT - Urea	0.999
PCU - Urea	0.888	PCU – Urea	0.996	PCU - Urea	0.956
Fine Branches		***Previous Growth Ring***		***Organic Horizon***	
Control – CUF	**0.017**	Control – CUF	**0.021**	Control – CUF	0.231
Control – NBPT	**0.007**	Control – NBPT	**0.018**	Control – NBPT	0.573
Control – PCU	0.322	Control – PCU	**0.025**	Control – PCU	0.129
Control - Urea	0.164	Control - Urea	0.617	Control - Urea	0.477
CUF - NBPT	0.993	CUF - NBPT	1.000	CUF - NBPT	0.961
CUF - PCU	0.552	CUF – PCU	1.000	CUF - PCU	0.997
CUF - Urea	0.794	CUF – Urea	0.309	CUF - Urea	0.986
NBPT - PCU	0.320	NBPT - PCU	1.000	NBPT - PCU	0.851
NBPT - Urea	0.549	NBPT - Urea	0.280	NBPT - Urea	1.000
PCU - Urea	0.993	PCU - Urea	0.348	PCU - Urea	0.915
Coarse Branches		***Fine Roots (< 2 mm)***		***0-15 cm Mineral Soil***	
Control – CUF	**0.000**	Control – CUF	0.121	Control – CUF	**0.003**
Control – NBPT	**0.000**	Control – NBPT	**0.003**	Control – NBPT	**0.025**
Control – PCU	0.095	Control – PCU	**0.012**	Control – PCU	**0.006**
Control - Urea	**0.015**	Control - Urea	**0.053**	Control - Urea	0.157
CUF - NBPT	0.601	CUF - NBPT	0.443	CUF - NBPT	0.890
CUF - PCU	0.128	CUF - PCU	0.796	CUF - PCU	0.999
CUF - Urea	0.501	CUF - Urea	0.993	CUF - Urea	0.380
NBPT - PCU	**0.006**	NBPT - PCU	0.972	NBPT - PCU	0.963
NBPT - Urea	**0.042**	NBPT - Urea	0.695	NBPT - Urea	0.881
PCU - Urea	0.898	PCU - Urea	0.956	PCU - Urea	0.520
Bark		***Coarse Roots (> 2 mm)***		***15-30 cm Mineral Soil***	
Control – CUF	0.126	Control – CUF	0.386	Control – CUF	**0.036**
Control – NBPT	0.238	Control – NBPT	0.107	Control – NBPT	0.097
Control – PCU	0.371	Control – PCU	0.352	Control – PCU	0.131
Control - Urea	0.696	Control - Urea	0.950	Control - Urea	0.431
CUF - NBPT	0.996	CUF - NBPT	0.934	CUF - NBPT	0.988
CUF - PCU	0.962	CUF - PCU	1.000	CUF - PCU	0.964
CUF - Urea	0.740	CUF - Urea	0.800	CUF - Urea	0.633
NBPT - PCU	0.998	NBPT - PCU	0.952	NBPT - PCU	1.000
NBPT - Urea	0.910	NBPT - Urea	0.356	NBPT - Urea	0.888
PCU - Urea	0.979	PCU - Urea	0.764	PCU - Urea	0.942

Table A.4.6. *P > | t | values for treatment (CUF, NBPT, PCU, urea) contrasts for individual ecosystem component fertilizer N recovery (% of fertilizer N applied) for Study 1 (summer 2011) at mid-rotation loblolly pine plantations in the southern United States. The N application rate was 224 kg N ha^{-1}.*

Differences in Fertilizer N Treatments	**Adjusted P-Value**	**Differences in Fertilizer N Treatments**	**Adjusted P-Value**	**Differences in Fertilizer N Treatments**	**Adjusted P-Value**
Foliage		***Current Growth Ring***		***Litterfall***	
Control – CUF	0.121	Control – CUF	0.238	Control – CUF	0.643
Control – NBPT	**0.033**	Control – NBPT	0.173	Control – NBPT	0.307
Control – PCU	0.232	Control – PCU	0.308	Control – PCU	0.329
Control - Urea	0.077	Control - Urea	**0.036**	Control - Urea	0.961
CUF - NBPT	0.965	CUF - NBPT	1.000	CUF - NBPT	0.973
CUF - PCU	0.995	CUF – PCU	1.000	CUF - PCU	0.980
CUF - Urea	0.999	CUF – Urea	0.855	CUF - Urea	0.951
NBPT - PCU	0.842	NBPT - PCU	0.996	NBPT - PCU	1.000
NBPT - Urea	0.993	NBPT - Urea	0.927	NBPT - Urea	0.684
PCU - Urea	0.972	PCU – Urea	0.774	PCU - Urea	0.712
Fine Branches		***Previous Growth Ring***		***Organic Horizon***	
Control – CUF	0.081	Control – CUF	0.770	Control – CUF	0.350
Control – NBPT	0.065	Control – NBPT	**0.035**	Control – NBPT	0.150
Control – PCU	0.294	Control – PCU	0.133	Control – PCU	0.155
Control - Urea	0.239	Control - Urea	0.247	Control - Urea	0.717
CUF - NBPT	1.000	CUF - NBPT	0.303	CUF - NBPT	0.984
CUF - PCU	0.946	CUF – PCU	0.681	CUF - PCU	0.986
CUF - Urea	0.973	CUF – Urea	0.869	CUF - Urea	0.967
NBPT - PCU	0.913	NBPT - PCU	0.960	NBPT - PCU	1.000
NBPT - Urea	0.951	NBPT - Urea	0.836	NBPT - Urea	0.772
PCU - Urea	1.000	PCU - Urea	0.996	PCU - Urea	0.782
Coarse Branches		***Fine Roots (< 2 mm)***		***0-15 cm Mineral Soil***	
Control – CUF	**0.035**	Control – CUF	0.315	Control – CUF	**0.015**
Control – NBPT	0.174	Control – NBPT	0.679	Control – NBPT	**0.005**
Control – PCU	**0.013**	Control – PCU	0.836	Control – PCU	**0.001**
Control - Urea	**0.007**	Control - Urea	0.771	Control - Urea	0.635
CUF - NBPT	0.917	CUF - NBPT	0.966	CUF - NBPT	0.990
CUF - PCU	0.992	CUF - PCU	0.880	CUF - PCU	0.802
CUF - Urea	0.945	CUF - Urea	0.925	CUF - Urea	0.234
NBPT - PCU	0.712	NBPT - PCU	0.998	NBPT - PCU	0.965
NBPT - Urea	0.531	NBPT - Urea	1.000	NBPT - Urea	0.105
PCU - Urea	0.998	PCU - Urea	1.000	PCU - Urea	0.028
Bark		***Coarse Roots (> 2 mm)***		***15-30 cm Mineral Soil***	
Control – CUF	0.891	Control – CUF	**0.015**	Control – CUF	0.371
Control – NBPT	0.192	Control – NBPT	0.052	Control – NBPT	0.717
Control – PCU	0.091	Control – PCU	0.588	Control – PCU	0.322
Control - Urea	0.668	Control - Urea	0.614	Control - Urea	0.745
CUF - NBPT	0.651	CUF - NBPT	0.974	CUF - NBPT	0.974
CUF - PCU	0.410	CUF - PCU	0.263	CUF - PCU	1.000
CUF - Urea	0.992	CUF - Urea	0.245	CUF - Urea	0.966
NBPT - PCU	0.994	NBPT - PCU	0.577	NBPT - PCU	0.955
NBPT - Urea	0.879	NBPT - Urea	0.551	NBPT - Urea	1.000
PCU - Urea	0.666	PCU - Urea	1.000	PCU - Urea	0.943

Table A.4.7. *$P > | t |$ values for fertilizer N treatment (CUF, NBPT, PCU, urea) contrasts for individual component fertilizer N recovery (% of fertilizer N applied) for Study 2 (spring 2012) at mid-rotation loblolly pine plantations in the southern United States. The N application rate was 224 kg N ha^{-1}. N =12.*

Differences in Fertilizer N Treatments	Adjusted P-Value	Differences in Fertilizer N Treatments	Adjusted P-Value	Differences in Fertilizer N Treatments	Adjusted P-Value
Foliage		*Current Growth Ring*		*Litterfall*	
Control – CUF	**0.000**	Control – CUF	**0.001**	Control – CUF	**0.017**
Control – NBPT	**0.000**	Control – NBPT	**0.000**	Control – NBPT	**0.044**
Control – PCU	**0.000**	Control – PCU	**0.014**	Control – PCU	**0.004**
Control - Urea	**0.000**	Control - Urea	**0.006**	Control - Urea	**0.006**
CUF - NBPT	0.165	CUF - NBPT	0.402	CUF - NBPT	0.996
CUF - PCU	0.638	CUF - PCU	0.870	CUF - PCU	0.984
CUF - Urea	1.000	CUF - Urea	0.963	CUF - Urea	0.997
NBPT - PCU	**0.005**	NBPT - PCU	0.062	NBPT - PCU	0.898
NBPT - Urea	0.237	NBPT - Urea	0.122	NBPT - Urea	0.956
PCU - Urea	0.518	PCU - Urea	0.998	PCU - Urea	1.000
Fine Branches		*Previous Growth Ring*		*Organic Horizon*	
Control – CUF	**0.000**	Control – CUF	**0.000**	Control – CUF	0.191
Control – NBPT	**0.000**	Control – NBPT	**0.000**	Control – NBPT	0.254
Control – PCU	**0.001**	Control – PCU	**0.000**	Control – PCU	**0.053**
Control - Urea	**0.000**	Control - Urea	**0.005**	Control - Urea	0.902
CUF - NBPT	0.530	CUF - NBPT	0.916	CUF - NBPT	1.000
CUF - PCU	0.876	CUF - PCU	0.895	CUF - PCU	0.977
CUF - Urea	0.989	CUF - Urea	0.332	CUF - Urea	0.675
NBPT - PCU	0.103	NBPT - PCU	0.415	NBPT - PCU	0.946
NBPT - Urea	0.261	NBPT - Urea	0.061	NBPT - Urea	0.766
PCU - Urea	0.990	PCU - Urea	0.857	PCU - Urea	0.322
Coarse Branches		*Fine Roots (< 2 mm)*		*0-15 cm Mineral Soil*	
Control – CUF	**0.006**	Control – CUF	**0.000**	Control – CUF	**0.000**
Control – NBPT	**0.005**	Control – NBPT	**0.000**	Control – NBPT	**0.000**
Control – PCU	**0.021**	Control – PCU	**0.000**	Control – PCU	**0.000**
Control - Urea	**0.002**	Control - Urea	**0.000**	Control - Urea	**0.008**
CUF - NBPT	1.000	CUF - NBPT	0.589	CUF - NBPT	1.000
CUF - PCU	0.994	CUF - PCU	0.661	CUF - PCU	0.999
CUF - Urea	0.995	CUF - Urea	**0.001**	CUF - Urea	0.521
NBPT - PCU	0.986	NBPT - PCU	1.000	NBPT - PCU	0.995
NBPT - Urea	0.998	NBPT - Urea	0.083	NBPT - Urea	0.625
PCU - Urea	0.925	PCU - Urea	0.063	PCU - Urea	0.384
Bark		*Coarse Roots (> 2 mm)*		*15-30 cm Mineral Soil*	
Control – CUF	**0.008**	Control – CUF	**0.014**	Control – CUF	**0.041**
Control – NBPT	**0.000**	Control – NBPT	0.150	Control – NBPT	0.207
Control – PCU	0.102	Control – PCU	0.103	Control – PCU	0.058
Control - Urea	0.185	Control - Urea	0.600	Control - Urea	0.389
CUF - NBPT	0.819	CUF - NBPT	0.871	CUF - NBPT	0.944
CUF - PCU	0.864	CUF - PCU	0.935	CUF - PCU	1.000
CUF - Urea	0.712	CUF - Urea	0.359	CUF - Urea	0.790
NBPT - PCU	0.256	NBPT - PCU	1.000	NBPT - PCU	0.974
NBPT - Urea	0.148	NBPT - Urea	0.902	NBPT - Urea	0.995
PCU - Urea	0.998	PCU - Urea	0.825	PCU - Urea	0.860

Table A.4.8. *The mean ^{15}N (‰), N (g kg^{-1}) and fertilizer N recovery (% of fertilizer N applied) for individual ecosystem components for loblolly pine stands in the southern United States selected to evaluate ecosystem partitioning of fertilizer N for urea or enhanced efficiency N containing fertilizers enriched with ^{15}N. Data is for Study 1 (spring 2011, summer 2011). Different letters represent significant differences at $\alpha = 0.05$. Numbers in parentheses represent the standard error of the mean. The N application rate was 224 kg N ha^{-1}. N = 5.*

		Spring (n=5)			**Summer (n=5)**		
Ecosystem Component	**Treatment**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery**
Foliage	Control	*-3.47^{a} (0.96)*	12.4^{a} (0.3)	N/A	*-2.98^{a} (0.38)*	12.3^{a} (0.6)	N/A
	CUF	**106.4^{b} (8.2)**	14.2^{a} (0.6)	13.6^{a} (2.7)	**86.7^{b} (17.9)**	14.1^{a} (0.7)	9.8^{a} (3.4)
	NBPT	**110.1^{b} (14.3)**	15.0^{a} (1.2)	17.3^{a} (4.0)	**84.2^{b} (9.2)**	15.1^{a} (0.9)	12.3^{a} (4.2)
	PCU	**95.1^{b} (15.7)**	14.9^{a} (0.4)	9.8^{a} (1.2)	**72.7^{b} (9.0)**	14.0^{a} (0.5)	8.4^{a} (1.5)
	Urea	**105.4^{b} (10.1)**	14.7^{a} (0.5)	13.6^{a} (4.4)	**80.0^{b} (11.7)**	14.7^{a} (0.1)	10.7^{a} (2.5)
Fine Branches	Control	*-3.96^{a} (1.17)*	*4.8^{a} (0.2)*	N/A	*-2.13^{a} (0.91)*	**5.1^{a} (0.3)**	N/A
	CUF	**93.7^{b} (4.5)**	7.2^{ab} (0.5)	2.0^{a} (0.5)	**80.2^{b} (14.3)**	**7.5^{b} (0.6)**	1.4^{a} (0.4)
	NBPT	**88.8^{b} (9.6)**	**7.5^{b} (0.8)**	2.2^{a} (0.6)	**77.9^{b} (9.6)**	**7.2^{b} (0.5)**	1.5^{a} (0.6)
	PCU	**82.5^{b} (13.1)**	7.2^{ab} (0.3)	1.1^{a} (0.3)	**65.9^{b} (5.3)**	6.6^{ab} (0.4)	1.1^{a} (0.2)
	Urea	**83.4^{b} (6.7)**	6.8^{ab} (0.6)	1.3^{a} (0.4)	**66.8^{b} (16.5)**	6.4^{ab} (0.6)	1.1^{b} (0.4)
Coarse Branches	Control	*-2.38^{a} (0.98)*	3.7^{a} (0.6)	N/A	*-2.82^{a} (0.71)*	3.0^{a} (0.5)	N/A
	CUF	**69.2^{b} (6.5)**	3.4^{a} (0.7)	5.7^{ab} (1.1)	**47.7^{b} (6.8)**	3.3^{a} (0.5)	3.5^{a} (0.9)
	NBPT	**68.4^{b} (9.0)**	3.3^{a} (0.5)	*7.3^{a} (1.0)*	**51.3^{b} (7.7)**	3.3^{a} (0.3)	2.6^{a} (0.7)
	PCU	**52.0^{b} (7.9)**	3.1^{a} (0.3)	**2.9^{b} (0.4)**	**53.6^{b} (9.3)**	3.5^{a} (0.7)	4.0^{a} (1.1)
	Urea	**54.0^{b} (5.2)**	3.2^{a} (0.4)	**3.9^{b} (0.8)**	**57.4^{b} (3.8)**	3.2^{a} (0.3)	4.3^{a} (0.8)
Bark	Control	*-2.54^{a} (1.03)*	2.3^{a} (0.3)	N/A	*-2.99^{a} (1.87)*	2.1^{a} (0.2)	N/A
	CUF	**20.2^{b} (5.9)**	2.1^{a} (0.3)	1.3^{a} (0.7)	10.3^{ab} (2.5)	2.1^{a} (0.2)	0.4^{a} (0.2)
	NBPT	**19.0^{b} (6.0)**	2.7^{a} (0.5)	1.1^{a} (0.3)	**18.8^{b} (7.9)**	2.2^{a} (0.3)	1.0^{a} (0.4)
	PCU	17.3^{ab} (5.6)	2.5^{a} (0.1)	1.0^{a} (0.3)	**14.7^{b} (1.4)**	3.4^{a} (0.9)	1.2^{a} (0.5)
	Urea	14.6^{ab} (3.6)	2.4^{a} (0.2)	0.7^{a} (0.1)	**15.4^{b} (3.3)**	2.3^{a} (0.2)	0.6^{a} (0.1)
Growth Ring (CGR) year of sampling	Control	*-4.22^{a} (0.91)*	1.5^{a} (0.2)	N/A	*-3.41^{a} (0.78)*	1.2^{a} (0.2)	N/A
	CUF	**81.8^{b} (11.1)**	1.4^{a} (0.1)	1.8^{a} (0.4)	**65.8^{b} (9.2)**	1.7^{a} (0.2)	1.6^{a} (0.7)
	NBPT	**75.4^{b} (13.2)**	1.9^{a} (0.2)	2.1^{a} (0.3)	**70.5^{b} (8.8)**	1.9^{a} (0.1)	1.7^{a} (0.4)
	PCU	**65.2^{b} (13.2)**	1.9^{a} (0.3)	1.8^{a} (0.3)	**53.3^{b} (10.1)**	1.6^{a} (0.1)	1.5^{a} (0.5)
	Urea	**70.0^{b} (12.4)**	1.8^{a} (0.3)	2.0^{a} (0.5)	**70.91^{b} (8.43)**	2.2^{a} (0.3)	2.3^{a} (0.7)
Growth Rings (PGR) composited prior to year of sampling	Control	*-3.21^{a} (1.78)*	0.9^{a} (0.2)	N/A	*-3.55^{a} (1.61)*	1.0^{a} (0.2)	N/A
	CUF	**44.0^{b} (4.9)**	1.2^{a} (0.2)	4.9^{a} (0.8)	**27.3^{b} (3.6)**	1.1^{a} (0.3)	1.9^{a} (0.7)
	NBPT	**35.0^{b} (6.2)**	1.0^{a} (0.2)	5.0^{a} (1.6)	**38.2^{b} (8.1)**	1.5^{a} (0.3)	5.1^{a} (1.5)
	PCU	**33.1^{b} (7.7)**	1.2^{a} (0.3)	4.8^{a} (1.3)	**28.2^{b} (4.8)**	1.1^{a} (0.3)	4.0^{a} (1.6)
	Urea	**35.7^{b} (7.7)**	0.7^{a} (0.2)	2.1^{a} (0.4)	**27.5^{b} (4.6)**	1.2^{a} (0.3)	3.4^{a} (1.1)
Litterfall (Current Year)	Control	-2.23^{a} (0.93)	8.3^{a} (0.2)	N/A	-2.59^{a} (0.90)	7.8^{a} (0.8)	N/A
	CUF	36.4^{a} (14.1)	6.9^{a} (0.7)	0.5^{a} (0.2)	23.0^{a} (3.05)	5.4^{a} (0.9)	0.26^{a} (0.1)
	NBPT	33.6^{a} (6.5)	5.7^{a} (1.2)	0.5^{a} (0.2)	28.4^{a} (14.6)	6.1^{a} (1.4)	0.4^{a} (0.2)
	PCU	40.2^{a} (12.9)	6.6^{a} (1.1)	0.6^{a} (0.4)	27.4^{a} (11.1)	5.5^{a} (0.5)	0.4^{a} (0.2)
	Urea	33.9^{a} (10.3)	6.0^{a} (0.7)	0.4^{a} (0.2)	23.9^{a} (9.8)	4.2^{a} (1.0)	0.1^{a} (0.1)
Fine Roots (< 2 mm)	Control	*-0.10^{a} (0.19)*	11.7^{a} (0.7)	N/A	*3.1^{a} (2.1)*	13.0^{a} (0.9)	N/A
	CUF	**21.5^{b} (7.0)**	12.8^{a} (0.9)	8.8^{a} (2.8)	**26.7^{b} (2.1)**	11.7^{a} (0.9)	13.4^{a} (1.9)
	NBPT	**24.0^{b} (4.3)**	12.8^{a} (0.9)	14.7^{a} (3.8)	15.6^{ab} (4.9)	13.5^{a} (1.1)	10.5^{a} (3.0)
	PCU	**24.2^{b} (5.5)**	12.5^{a} (1.0)	12.6^{a} (1.8)	14.4^{ab} (1.8)	12.2^{a} (0.6)	9.2^{a} (1.8)
	Urea	14.0^{ab} (2.3)	12.2^{a} (1.1)	10.2^{a} (2.1)	14.1^{ab} (2.2)	12.2^{a} (0.7)	9.8^{a} (3.3)

		Spring (n=5)			**Summer (n=5)**		
Ecosystem Component	**Treatment**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery**	**^{15}N (‰)**	**N (g kg^{-1})**	**Fertilizer N Recovery**
Coarse Roots (> 2mm)	Control	*0.75^{a} (0.72)*	9.1^{a} (0.9)	N/A	*1.0^{a} (0.8)*	8.7^{a} (0.5)	N/A
	CUF	**16.3^{b} (4.3)**	8.8^{a} (0.7)	2.3^{a} (0.9)	**15.2^{c} (2.4)**	7.1^{a} (0.5)	1.8^{a} (0.6)
	NBPT	**15.2^{b} (2.2)**	7.0^{a} (0.2)	3. 3^{a} (1.5)	**12.9^{bc} (2.0)**	8.4^{a} (0.8)	1.5^{a} (0.3)
	PCU	**16.3^{b} (2.2)**	7.0^{a} (0.3)	2. 4^{a} (0.9)	**8.2^{b} (0.9)**	6.9^{a} (0.3)	0.7^{a} (0.3)
	Urea	8.4^{ab} (1.9)	6.9^{a} (0.5)	1.0^{a} (0.4)	**8.6^{b} (0.8)**	6.7^{a} (0.5)	0.7^{a} (0.2)
Organic Horizon	Control	*-2.36^{a} (0.52)*	8.0^{a} (0.6)	N/A	*-2.33^{a} (0.52)*	7.1^{a} (0.9)	N/A
	CUF	**85.1^{b} (20.5)**	9.0^{a} (0.7)	2.6^{a} (1.1)	**69.9^{b} (3.0)**	9.7^{a} (1.3)	3.0^{a} (1.2)
	NBPT	**51.7^{b} (9.5)**	8.5^{a} (0.7)	1.8^{a} (0.6)	**91.4^{b} (9.9)**	8.6^{a} (0.7)	3.8^{a} (1.4)
	PCU	**77.5^{b} (4.7)**	9.3^{a} (1.1)	3.0^{a} (1.2)	**101.6^{b} (13.3)**	9.4^{a} (1.4)	3.8^{a} (1.6)
	Urea	**53.3^{b} (13.6)**	8.8^{a} (0.8)	2.0^{a} (0.7)	**76.6^{b} (11.1)**	8.1^{a} (1.2)	2.0^{a} (0.6)
0-15 cm Mineral Soil	Control	*3.86^{a} (0.76)*	0.8^{a} (0.1)	N/A	*3.01^{a} (0.53)*	1.2^{a} (0.2)	N/A
	CUF	**17.6^{b} (3.3)**	1.1^{a} (0.3)	**28.5^{b} (6.6)**	**15.6^{b} (2.3)**	1.3^{a} (0.3)	*27.2^{a} (4.7)*
	NBPT	**14.4^{b} (1.7)**	1.1^{a} (0.3)	*22.4^{ab} (3.5)*	**17.7^{b} (2.8)**	1.1^{a} (0.3)	*30.7^{a} (7.6)*
	PCU	**16.4^{b} (3.8)**	1.4^{a} (0.4)	26.8^{ab} (4.6)	**19.7^{b} (4.3)**	1.2^{a} (0.4)	*35.7^{a} (6.9)*
	Urea	**10.8^{b} (1.9)**	1.1^{a} (0.4)	*16.1^{a} (6.1)*	**9.60^{b} (2.4)**	1.4^{a} (0.5)	**10.7^{b} (4.4)**
15-30 cm Mineral Soil	Control	**5.12^{a} (0.37)**	0.5^{a} (0.2)	N/A	5.80^{a} (0.7)	0.7^{a} (0.2)	N/A
	CUF	**11.0^{b} (1.8)**	0.7^{a} (0.1)	6.5^{a} (2.3)	12.7^{a} (4.5)	0.8^{a} (0.2)	10.0^{a} (6.6)
	NBPT	9.7^{ab} (1.1)	0.8^{a} (0.4)	5.5^{a} (1.7)	10.8^{a} (1.81)	0.7^{a} (0.2)	6.8^{a} (2.1)
	PCU	9.7^{ab} (1.3)	0.8^{a} (0.3)	5.2^{a} (1.3)	12.7^{a} (2.6)	0.9^{a} (0.4)	10.6^{a} (2.9)
	Urea	8.6^{ab} (1.1)	0.7^{a} (0.2)	3.6^{a} (1.1)	8.3^{a} (0.7)	1.1^{a} (0.6)	6.5^{a} (4.1)