

Effects of a Control Release Nitrogen Fertilizer and Thinning
on the Nitrogen Dynamics of a Mid-Rotation Loblolly Pine Stand
in the Piedmont of Virginia

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ABSTRACT

Nitrogen deficiency is characteristic of many mid-rotation loblolly pine (*Pinus taeda* L.) plantations in the Piedmont region of the southeastern USA. Fertilization with urea is the most common method used to correct this deficiency. Previous studies show that urea fertilization produces a rapid pulse of available nitrogen (N) with only a portion being utilized by plantation trees. Controlled release fertilizers release available N more slowly over a longer period of time and therefore may result in greater uptake efficiency. The objective of this study was to compare Nitroform®, a urea-formaldehyde controlled release N fertilizer versus urea and a control by measuring the effects of the two fertilizer treatments on N availability and loss as: total KCl extractable-N, total ion exchange membrane-N (IEM-N), N mineralization, and N volatilization, in a mid-rotation loblolly pine plantation in the Piedmont of Virginia. In addition, mid-summer and mid-winter fertilizations were compared to assess fertilizer uptake as a function of season. After the summer fertilization, Nitroform® significantly increased total KCl-extractable N, IEM-N, and N mineralization for two to three months over urea and the control. Three hundred times more N volatilized from urea than from controlled release Nitroform®. Interestingly, seven months after the summer application, the controlled release Nitroform® showed marked immobilization for three months while urea demonstrated greater N mineralization. After the winter application, fertilization with urea demonstrated greater soil inorganic N concentrations for two to three

months over Nitroform®, very little N was immobilized, and volatilization was only 10 times that of Nitroform®. After summer and winter fertilizations, both fertilizer treatments significantly increased soil inorganic N concentrations and N volatilization over controls, however did not significantly increase N mineralization over controls when average response was tested over the entire sampling period. In addition to the fertilizer effects measured, a thinning only treatment was also incorporated into this study with soil N-availability indices compared to a control with no thinning or fertilization. The results from the thinning only treatment demonstrated no significant increases over the control in total KCl extractable-N, IEM-N, N-mineralization, or N volatilization when average responses were tested over the entire sampling period.

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CHAPTER 1. INTRODUCTION

Planting of pine in the southeastern United States began during the 1920's, when a new demand for wood products and fiber began to outstrip the supply of a land base that had been subjected to a century of abusive agriculture and timber harvesting without subsequent reforestation (Fox et al., 2004). According to Wakely (1954), the first commercial planting operation took place in southern Louisiana, between 1925 and 1930, by the Great Southern Lumber Company. This was accompanied by over 1,000 hectares of southern pine being planted by the U.S. Forest Service in South Carolina and over 400,000 hectares by the Civilian Conservation Corps in the 1930's (Fox et al., 2004). Immediately following World War II, demand for wood products greatly increased (Binkley, 1986), and subsequently millions of acres of abandoned farmland were planted in southern pine and managed under the Soil Bank Program (USDA Forest Service, 1988). Since this time, global populations and per capita wood demand have increased, thus intensively managed southern pine plantations have become an important contributor to the world wood supply (Shepherd, 1993). Presently more than 12.1 million hectares of pine plantations are managed in the southeastern United States (Fox et al., 2004). On the land base of southeastern pine plantations, loblolly pine is the most widely grown commercial tree species (Albaugh et al., 2003; Fox et al., 2006).

Similar to the rise of pine plantations, research in forest nutrition began in the early 1900's, when several experiments on European species were presented at the 6th IUFRO conference in 1906 (Binkley, 1986). However, experiments specifically focused on the effects of nutritional amendments to southern pine plantations began in earnest after World War II (Binkley, 1986 ; Fox et al., 2004). Several early studies, including Laird (1972) and Pritchett et al. (1961), measured marked volume growth in Slash pine after fertilization with phosphorus (P). Research began to focus on nitrogen (N)

fertilization response after several studies showed conclusively that fertilization with N and P produced large volume gains throughout a rotation (Ballard, 1984). Pritchett and Smith (1969) reported that 48% of over 30 experiments on young pines in the SE coastal plain showed response to N fertilizers.

Since that time, it has been well established that Nitrogen is the nutrient that most often limits productivity in southern pine plantations (Allen and Ballard, 1983), and that response to fertilization depends on the ability of the site to supply the nutrients required for tree growth (Allen, 1987). Allen and Ducey (2001) recently concluded that even on sites with high nutrient availability, N and P concentrations are not sufficient for maintaining high growth rates without fertilization. The need to overcome these site deficiencies has led to the majority of industrial pine plantations being fertilized in the southeast (Vitousek et al., 1983). Generally, fertilization increases nutrient availability and uptake from mineral soil, resulting in higher N in foliage, greater leaf area, and greater net photosynthesis. This translates into greater above ground allocation of carbohydrates for wood production and less below-ground allocation for root growth (Fisher and Binkley, 2000). Economically, it has been shown that returns on investment greater than 15% can be achieved from fertilization on productive but N-limited sites throughout the southeast (Allen et al., 2005).

Pine plantations are examples of the domesticated forest, where site quality can be manipulated by silvicultural inputs (Stone, 1975). Moore and Allen (1999), state the main goal of intensive plantation management is to, “generate significantly improved biological and economic yield on a finite land base, while avoiding site degradation and minimizing off-site pollution”. After several decades of research providing evidence that production increases are based on increased resource availability, pine plantation resource management through fertilization has become a widespread practice, with more

than 600,000 hectares of southern pine plantations being fertilized on an annual basis (Allen and Ducey, 2001). The ability to manipulate site resource availability through silvicultural inputs has led to greater wood production per acre over shorter rotation lengths (Kelting et al., 1999). Silvicultural treatments of established stands usually lead to increased nutrient availability, either by reduced competition, or nutrient additions (McGoll and Powers, 1984). This concept is clearly shown in Nilsson and Allen (2003), where the combination of intensive silvicultural treatments including site preparation, continual herbicide application, and nutrient amendment, significantly increased standing volume after 18 years over any single treatment alone (Nilsson and Allen, 2003). The growth potential for plantations in southeastern USA is much higher than previously thought (Allen and Albaugh, 1999). The current challenge is to develop and implement the appropriate silvicultural systems to realize this potential in a cost effective and environmentally sensitive manner (Allen and Albaugh, 1999).

CHAPTER II. LITERATURE REVIEW

Pine Plantation Soil Nutrient Availability and Fertilization Practices

Introduction

Immediately after plantation establishment, soil nutrient availability is relatively high because site preparation operations have created an environment of increased temperature and moisture which facilitates nutrient release from incorporated organic matter, a condition referred to as the assart effect (Tamm, 1964; Kimmins, 1997). In addition, within the first four years of a plantation, the demand for nutrients by crop trees is relatively minimal due to their low leaf area, and low site occupancy (Allen et al., 1990). As stem growth and leaf area increase, stand crown closure occurs and the conditions conducive to high soil nutrient availability are no longer present (Piatek and Allen, 2001). In addition, there is high nutrient demand as nutrients are being rapidly sequestered by growing trees, and in the forest floor material (Binkley et al., 1995). During this stage of stand development, stand leaf area production and subsequent growth is regulated by the diminished available site resources (Miller, 1981). Here, fertilization becomes necessary to overcome soil nutrient deficiency and meet the nutritional demands of the growing trees. Allen (1987) states that for N, the period of maximum response to fertilization may be at or before this period of stand closure and may last well into the rotation. As stands age, growth has been observed to reach a peak and then decline (Assman, 1970), which is hypothesized to be a result of limited site resources (Binkley et al., 1995). However, Albaugh et al. (2004), concludes that continuous improved resource availability through mid-rotation fertilization may delay these expected growth reductions late in the rotation (Albaugh et al., 2004). Furthermore, mid-rotation fertilization response has been found on all but the driest or very best sites (Allen, 1985). Albaugh et al. (2003), studied fertilization effects in mid-rotation loblolly

plantations across the southeast and found that N and P were important limiting factors at all study sites on a variety of soil types and that all sites exhibited significant positive response to additions of N and P.

Soil Nitrogen Supply and Uptake

According to Fisher and Binkley (2000), nutrient supply can be viewed in three ways: 1) the mass of an element that is readily available in soil solution; 2) the mass of an element in labile pools that is accessible to microbes and plants ; and 3) the mass of an element actually acquired by growing plants. Nitrogen is especially important in plant nutrition as a component of amino acids, enzymes, proteins, and nucleic acids (Fisher and Binkley, 2000). In foliage, most of the nitrogen is present in the carboxylating enzyme RUBISCO, and higher nitrogen in leaves leads to higher RUBISCO concentrations and higher rates of photosynthesis (Fisher and Binkley, 2000). However, according to Vitousek et al. (1983), nitrogen is highly limiting in pine plantations because 1) only fixed forms of N are utilized by pines and microbial fixation of atmospheric N is not prevalent in the southeast, 2) nitrogen is highly mobile and lost through volatilization or leaching, and 3) less than 5% of organic N in soils is mineralized each year, and the resultant ammonium may be immediately utilized by soil microbes in the decomposition of organic matter and conversion to nitrate, or by competing vegetation. Therefore, most southern pine plantations are inherently N deficient (Dougherty, 1996).

After clear-cutting, N mineralization is increased (Vitousek, 1982), but nearly all the mineralized nitrogen is retained on site. The major mechanisms retaining this nitrogen are uptake by re-growing vegetation where N is bound as amide groups in proteins (Binkley, 1986), uptake by microbes during decomposition of organic material (immobilization), adsorption to cation exchange sites on clay particles which are

negatively charged as a result of broken edges and isomorphous substitution (Binkley, 1986), and low rates of nitrification (Vitousek and Matson, 1984).

As stands grow after planting, stemwood increases in volume while leaf biomass reaches an asymptote (Fisher and Binkley, 2000). Likewise, the total amount of N in trees increases with stand age and biomass (van den Driessche, 1984), while maximum foliage N concentrations accumulate early in the rotation and then decrease (Switzer et al., 1968). Switzer et al. (1968) report that in a 20 year old loblolly pine stand, more than 50% of N in standing biomass was in stemwood (Switzer et al., 1968). Switzer et al. (1968) show that in a 20 year loblolly plantation in Mississippi, 2300 kg-N ha⁻¹ of total ecosystem N was estimated on site. However, only 20% was in the organic fraction of trees and forest floor, while 80% was estimated to be in soil (Switzer et al., 1968).

Leaf litter also continues to increase in the forest floor throughout the rotation, which becomes the dominant pool of above ground N (Piatek and Allen, 2001). Wells and Jorgensen (1975) studied ecosystem N allocation in a 16 year old loblolly pine plantation, where they concluded that 13% of total ecosystem N was sequestered in standing biomass, another 13% was in the forest floor, and the remaining 74% of the ecosystem N was in long-term, unavailable soil organic matter (Wells and Jorgensen, 1975). Urrego (1993) also reported that the forest floor in southern pine stands was the dominant aboveground pool for N and P, accounting for 75% and 66%, respectively, of the total content in aboveground biomass.

Naturally occurring sources of nitrogen for growth in southeastern pine plantations include bulk precipitation and sedimentation of aerosols from the atmosphere which may amount to 6-10 kg-N ha⁻¹ year⁻¹ (Wells and Jorgensen, 1979), and biological fixation of N that may average 2 kg-N ha⁻¹ year⁻¹ (Vitousek et al., 1983). Therefore it is assumed that non-fertilized pine plantations retain net N inputs of approximately 8 kg-N

ha⁻¹ year⁻¹ (Vitousek et al., 1983). Paul and Juma (1981), studied N¹⁵ turnover rates in forest soil and divided total soil N into six pools:

1. N mineralized from microbial biomass
2. N from metabolites
3. N from the active humus fraction
4. N from the stabilized N pool
5. inorganic-N forms as a result of mineralization
6. N in biomass from decomposing litter

However, Rapp (1979) found that in one season only 1-2% of total soil N was mineralized. Therefore, in non-fertilized forest soils, the labile pool of nitrogen is typically 1 to 3 % of total soil nitrogen (Fisher and Binkley, 2000).

Pine Plantation Microbial Processes

It is generally accepted that the labile pool of Nitrogen in plantation soils is mainly controlled by microbially mediated processes (Fisher and Binkley, 2000; Pritchett and Fisher, 1987). Jansson (1958), in one of the first models of N transformations in the soil, stated that the ultimate step in the production of inorganic-N from organic residues was not unidirectional but rather was one step in the “continuous internal cycle” of N in the microbial biomass. N¹⁵ studies have shown that microbial uptake of N averages 75% to 95% of the N released through decomposition (Binkley, 1986).

The decomposition of annual leaf litter in the forest floor is a crucial step in the nitrogen cycling of pine plantations. According to Gosz (1984), litter decomposition serves two primary functions: the formation of soil organic matter and the mineralization of essential elements. Decomposition first relies on the fragmentation of larger materials by micro-, meso-, and macro-fauna (Fisher and Binkley, 2000). The smaller material and feces produced by these organisms are further broken down by microflora resulting in

soil organic matter integration. In acidic soils of pine plantations, Protozoa and Nematoda micro-fauna, and Enchytraeidae meso-fauna dominate this population (Gosz, 1984).

In southern pine soils, there are both autotrophic and heterotrophic bacteria. The autotrophic bacteria include *Nitrosomonas* and *Nitrobacter*, which reduce ammonium to nitrate, and nitrate to nitrite (Paul and Clark, 1989). Nitrate is highly mobile in soil, can be rapidly utilized by competing vegetation, leached, or lost as nitrogen gas in anaerobic conditions (Vitousek et al., 1983). In addition, the specialized nitrifying bacteria that oxidize ammonium to nitrate are not good competitors for ammonium, which leads to generally low levels of Nitrate in pine plantation soils (Binkley, 1986). Heterotrophic bacteria, the largest group of soil bacteria, require preformed organic compounds as sources of energy and carbon (Fisher and Binkley, 2000). These heterotrophic microbes are strong competitors for nitrogen due to their rapid growth and large surface to volume ratios (Vitousek et al., 1983).

Freshly fallen litter has a rapid initial increase in microbial populations which then dies away as the metabolism of sugars and amino acids is complete (Gosz, 1984). Soil organic matter is the principal source and sink of plant nutrients and the rate of decomposition of organic matter by microbes depends on the quality of the substrate (Fisher and Binkley, 2000). Past research has led to the general assumption that Carbon is usually plentiful to microbes however N and P are limiting (Gosz, 1984). Therefore, litter with low lignin and high N will decompose with N mineralization occurring, however litter with high lignin and low N will increase N immobilization in the soil (Binkley, 1986). Therefore, net immobilization of N and P will occur until the element is accumulated in quantities which do not limit decomposer growth, and then mineralization will occur (Gosz, 1984). Because of the high C:N ratio in forest litter, decomposition

proceeds slowly, leading to an increase in the carbonaceous materials which requires added nitrogen for microbial breakdown (Fisher and Binkley, 2000). Nutrient availability can place very substantial constraints on the decomposition rate of organic matter by its associated microflora (Rojas, 2005). Sanchez (2001) found that the quality of incorporated litter affected the rate of nutrient release. The incorporation of nutrients by means of fertilization may help microbes in the decomposition process and release of nutrients (Schmidt et al., 1999). Current research also suggests that total carbon may be abundant in the soil. However, microbial populations may be limited by the quantity of labile carbon (Boone, 1994; Compton and Boone, 2002; Gallardo and Schlesinger, 1995; Gurlevik et al., 2004; and Whalen et al., 2000). Therefore total C: N ratios may be too simplistic in explaining microbial mineralization or immobilization of N.

Soil temperature, moisture, and aeration are the major controlling factors for soil biological processes (Kelting et al., 1999). Paul and Clark (1989) showed that maximum microbial activity occurred between 25 and 35°C and decreased linearly on both sides of this range. In terms of soil moisture, Skopp et al. (1990) determined that maximum microbial activity occurred between water content levels of 55% and 61% of total soil porosity. Skopp et al. (1990), also show that soil water content is the main determinant of microbial activity due to its affect on oxygen and nutrient diffusion rates with soil.

Uptake of Nitrogen by Pine Roots

Nutrients arrive at the root surface by a mixture of mass flow with water or of diffusion along a concentration gradient (Nye and Tinker, 1977). Nitrate is transferred to roots mainly through mass flow, while ammonium is mainly through diffusion, making it less mobile (Bowen, 1984). In mature forests the bulk if not all of the N absorbed is ammonium (Attiwill and Leeper, 1987). Bowen (1984) summarized that the limiting

factor in the uptake of ions moving through diffusion is not the absorption capability of the root but the tortuosity of the path ions take towards the root.

The fine roots of trees turn over 2-5 times per season, involving the production of up to 10 tons $\text{ha}^{-1} \text{yr}^{-1}$ of dry matter (Cannell, 1989). The death of fine roots can represent the largest mortality loss and the greatest input of organic matter to the soil. Lee and Jose (2003) found no significant fertilization effect on fine root production, though an increasing trend was noted. The greatest fine root production was noticed during late spring and early summer (Lee and Jose, 2003). Previous research has shown that high input of N increases fine root mortality and decreases root production and longevity (Lee and Jose, 2003). Cannell (1989) states differing conclusion that improved nutrition decreases rates of fine root turnover, which can greatly increase the production of aboveground dry matter and wood. Allen and Albaugh (1999) hypothesized that increased total production efficiency resulted when more biomass was allocated to foliage and less to fine roots. Partitioning to wood can be increased most dramatically by decreasing rates of fine root turnover, which can be achieved by improving rates of nutrient and water supply to the roots (Cannell, 1989). Mycorrhizae also play an important role in nutrient movements to roots as they effectively increase the area of soil for nutrient accumulation, and exude compounds which solubilize immobilized nutrients (Bowen, 1984). Fertilizers have also been shown to increase mycorrhizal roots on nutrient poor sites (McGoll and Powers, 1984).

Pine Plantation Nitrogen Retranslocation

Considering microbial and forest floor immobilization of N, and the labile pool of N in forest soils being 1 to 3% of total soil N, internal cycling of Nitrogen is an important aspect of pine plantation nutrition. Switzer et al., (1968) found that total N requirement of a 20-year-old loblolly pine stand was around 70 kg ha^{-1} of which 38 kg was derived

from the soil (Switzer et al., 1968), while Binkley et al., (1995) later reported that typical N uptake rates in pine plantations were approximately $10 \text{ kg N ha}^{-1} \text{ year}^{-1}$, and Piatek and Allen (1999) show that N use in foliar biomass can reach $65 \text{ kg N ha}^{-1} \text{ year}^{-1}$, while Net N mineralization in mineral soil 0-15 cm in depth ranges from only 20 to $35 \text{ kg N ha}^{-1} \text{ year}^{-1}$. Several studies have shown that 20-60% of the required N for southern pine growth was derived from internal N re-translocation from senescing leaves to stems and back to new leaves (Binkley et al., 1995; Nambiar and Fife, 1991). Wells and Metz (1963) estimate that loblolly pine needles retranslocated 44, 38, and 58 % of their N, P, and K, respectively, prior to abscission. Switzer and Nelson (1972) estimated that 39% of N, 60% of P, 22% of K, 24% of Mg and 22% of S required by 20 year loblolly is obtained by retranslocation. A physiological explanation for the importance of retranslocation, is that the energy required for re-translocation of nutrients is 10% that of the energy that root uptake requires for uptake and assimilation of N (Fisher and Binkley, 2000).

The quantity of N cycling within the system is 9 times greater than the quantity being immobilized within the standing trees (Switzer et al., 1968). Since the forest floor also immobilizes great quantities of N, there must be a great amount of N cycling throughout the stand to meet the relatively small growing requirements of the trees (Switzer et al., 1968).

Measurements and Indices of Soil Nitrogen Availability

Introduction

Burger and Kelting (1996), state that tree growth should not be the only factor in determining site productivity changes from silvicultural treatments, instead, direct measurements on soil processes could provide more accurate portrayal of site response to management activities (Burger and Kelting, 1996). However, Mead (1984) stated that

considerable research was still required to develop a reliable index of the soil N available to trees, since the N cycle is variable across sites, and N pool sizes, as well as processes such as immobilization, mineralization, and losses must be considered (Mead, 1984). Furthermore, Polglase et al. (1992) argued that no *in situ* method can ever yield absolute and accurate measures of field N supply rates. While others argue that no soil measure of N has related well to fertilization response in pine forests of southeastern USA (Binkley et al., 1995; Hart and Binkley, 1985; Hart et al., 1986). Direct soil sampling not only measures soil nutrient supply, but instead provides an index of short-term nutrient availability (Wells and Allen, 1986). Several direct measurements of soil N availability are currently used in forest research, though none is believed to provide a complete understanding of soil N quantity, capacity, or intensity (Keeney, 1980). Most studies investigating fertilizer response in southern pine plantations utilize results from more than one method in order to draw meaningful conclusions.

In Situ N Mineralization Measurements

N mineralization is defined as the conversion of organic N to inorganic N as a result of microbial activity (Soil Science Society of America, 1987). N immobilization is defined as the inverse of mineralization, where inorganic N is converted the organic N form in microbial tissues (Soil Science Society of America, 1987). Organic compounds with C:N ratio less than 15:1 favor N mineralization while those with C:N ratios greater than 25:1 favor N immobilization (Schepers and Mesinger, 1994). N mineralization is microbially driven and therefore dependent on soil temperature, moisture, aeration, pH, and other physical and chemical factors.

Early methods for studying field N mineralization included 1) the buried bag incubation method (Eno, 1960; Vitousek and Matson, 1985), 2) Undisturbed soil columns

in plastic bags incubated under field conditions (Nadelhoffer et al., 1983), 3) Incubation of ion exchange resins placed in field (Binkley and Matson, 1983).

Raison et al. (1987), developed the *in situ* sequential pvc coring method because previous field estimates of N mineralization resulted in unknown effects of sample disturbance on the N mineralization measured. Likewise, laboratory estimates of mineralization on disturbed soils have been shown to be unreliable indices of field rates as well (Hart and Binkley, 1985)

Raison et al. (1987) argued that sequentially incubating *in situ* pvc tubes with caps prevents the uptake of mineral-N by roots and losses from leaching (Raison et al., 1987). Also, this method minimized soil disturbance throughout the sampling period, so disturbance effects on mineralization were minimized (Raison et al., 1987). Rice and Havlin (1994) also concluded that incubating disturbed soil samples results in overestimation of N mineralization and that measurements determined from disturbed soil samples did not correlate well to N uptake as estimated from field experiments. Raison et al. (1987) calculated net mineralization as the sum of changes in NH_4^+ -N and NO_3^- -N, expressed as concentrations or total amounts on an area basis The basic equations used in this methodology were:

$$\Delta \text{NH}_4^+\text{-N} = \text{net ammonification during incubation } (\text{NH}_4^+ \text{ incubated} - \text{NH}_4^+ \text{ initial})$$

$$\Delta \text{NO}_3^-\text{-N} = \text{net nitrification during incubation } (\text{NO}_3^- \text{ incubated} - \text{NO}_3^- \text{ initial})$$

$$\text{Nmin} = \Delta \text{NH}_4^+\text{-N} + \Delta \text{NO}_3^-\text{-N}$$

In order to test his methodology in forest soils, Raison et al. (1987), investigated the effects of sample disturbance on *in situ* N mineralization, the effects of sieving and increasing incubation temperature on laboratory N mineralization, the effects of galvanized cores on N mineralization in the laboratory, the effects of steel tubes on soil temperature, and the effects of incubation period length on *in situ* N mineralization.

The results of Raison et al. (1987), show that sieving can have a major effect on rates of N mineralization when performed prior to incubation, and the effect can either be positive or negative depending on the soil type.

Raison et al. (1987), suggest that *in situ* incubation periods should be site, seasonal, and treatment specific. If N mineralization is expected to be slow, the use of incubations less than 30 days would make detection of mineral N changes difficult. However, if more rapid mineralization is expected during the growing season or after fertilization, then a shorter incubation period may be more appropriate (Raison et al., 1987).

With regard to *in situ* N mineralization incubation results, Raison et al. (1987) found that within a few months of fertilization, a standard error of about 10% of the mean was typical. Mineralization and uptake of N, 12-14 months after fertilization, was also successfully measured and significant differences were demonstrated between treatments (Raison et al., 1987). Raison et al. (1987) argue that the fluxes estimated by sequential coring methodology could be used as quantitative measures. Results from the *in situ* N mineralization incubation showed N mineralization rates in control treatments to be $0.1 \text{ kg ha}^{-1} \text{ day}^{-1}$ and fertilized treatments to be $1.1 \text{ kg ha}^{-1} \text{ day}^{-1}$ (Raison et al., 1987).

Polglase et al. (1992) utilized *in situ* N mineralization cores inserted in the top 5 cm of mineral soil. Root severing removed root uptake effects and a cap on the cores removed leaching effects. Monthly *in situ* incubations occurred for 1 year, and monthly rates of net N mineralization were summed to yield annual totals.

Adams and Attiwill (1986), using *in situ* pvc cores capped with perforated sides, studied N mineralization in eucalyptus plantations in Australia. This study tested whether the methodology of *in situ* sequential coring proposed by Raison et al. (1987), affected N mineralization rates. Adams and Attiwill (1986) argue that moisture changes

inside cores with unperforated sides do not fluctuate similarly with bulk soil, and that N mineralization is not expected to be linear with time as stated by Raison et al. (1987). Results from Adams and Attiwill (1986) show that perforated cores over 4 weeks may equilibrate with bulk soil moisture and temperature environments. In a subsequent study, Adams et al. (1989), show N mineralization in temperate mature forests to be less than $10\text{kg-N ha}^{-1} \text{ year}^{-1}$, which is below the $16\text{-}100 \text{ kg-N ha}^{-1} \text{ year}^{-1}$ range reported in previous studies (Ducey and Allen, 2001 ; Gurlevik et al., 2004) .

Data showed that mineral N did not accumulate with time, rather rates of accumulation or depletion fluctuated in response to the changing environmental conditions during the incubation period, which was most notable in the 4-week period (Adams and Attiwill, 1986). The two week sample period in this study, showed no fluctuations in bulk soil or contained soil environments. The overall effect of incubation length on N mineralization was that measured rates of N mineralization tended to decrease as the period of incubation increased (Adams and Attiwill, 1986). It was argued that when using *in situ* method, measurements should be considered the concentrations of inorganic-N at discrete intervals which in turn measure the net balance between mineralization and immobilization (Adams and Attiwill, 1986).

Adams and Attiwill (1986), conclude that *in situ* incubations may affect rates of mineralization due to immobilization driven by the decomposition of severed roots with high C:N ratios. When incubation periods are long, both net mineralization and uptake will be underestimated (Adams and Attiwill, 1986). However, *in situ* N mineralization studies offer the possibility of obtaining the best estimate of the rate of N mineralization in forest soil (Adams et al., 1989). Furthermore, the effects of root severing and lack of C input from litterfall are diminished with shorter incubation periods (Adams et al., 1989). The emphasis should be on adequate replication at a time-scale which reflects the

balance between mineralization and immobilization (Adams et al., 1989). Adams et al. (1989) also conclude that the best estimate of N-mineralization will be obtained if incubations are for 2 weeks or less, as short incubations are necessary where N turnover is rapid or where disturbance effects on N mineralization are to be measured (Adams et al., 1989).

Schepers and Mesinger (1994) discuss the validity of in situ N mineralization methodologies, and state that these techniques have the advantage of incorporating all soil environment factors that affect N mineralization at a specific site, and also avoid the problems of extrapolating laboratory N mineralization estimates back to the field. However, they also state the limitations of field mineralization estimates include the dynamic nature of NO_3^- -N in the soil, the slowness of mineralization, and the natural spatial variability in the soil environment.

Estimating field mineralization using isolated intact soil cores is beneficial as it minimizes soil disturbance during sampling, and provides a clearly defined soil volume (Schepers and Mesinger, 1994). The advantage of covered soil tubes is that the effects of nitrogen leaching as result of precipitation and water movement downward through the bottom of the tube is minimized (Schepers and Mesinger, 1994).

Commenting on the buried bag N mineralization approach developed by Eno (1960), Schepers and Mesinger (1994) conclude that this method has advantages of eliminating nitrate leaching and exposure to field temperature conditions, however its disadvantages include an inability to mimic field soil moisture conditions, and gas exchange of O_2 and CO_2 throughout the incubation period.

Mattos et al. (2003), measured the effects of urea fertilization on N mineralization and microbial biomass N in a Citrus grove in norther Florida using Raison et al. (1987) methodology. Their results showed net immobilization around 3 mg-N kg^{-1} immediately

following fertilization with urea, accompanied by an increase in soil microbial biomass N around 11 mg-N kg⁻¹. Fertilization with urea positively affected immobilization and microbial biomass N over controls, though only after the first 15 days of sampling (Mattos et al., 2003).

Potential N Mineralization Indices

Potentially mineralizable N (N_o) refers to an active fraction of soil N that is responsible for the release of NO₃-N and NH₄-N through microbial activity (Kissel and Vigil, 1994). This method has traditionally been used for determination of N fertilization rates in agricultural soils. However, Kissel and Vigil (1994) argue that it may also be useful in estimating N mineralization processes in pine plantation soils as well. The Stanford and Smith (1972) method for determining potential N mineralization in the laboratory used field moist soil mixed with sand incubated under optimal temperature and moisture levels over a pre-determined period. The soil mixture was regularly leached which removed the NO₃-N and NH₄-N. The cumulative N mineralized with time is fitted to a single exponential model of $N_{min} = N_o(1 - e^{-kt})$.

According to Kissel and Vigil (1994), this method provides a laboratory index of potential N mineralization, however extrapolating laboratory results back to field estimates requires adjusting the model to fit actual field environmental conditions. Cabrera and Kissel (1988) tested Stanford and Smith's (1972) model against *in situ* N mineralization estimates in an agricultural setting and found that the potentially mineralizable N model overestimated field N mineralization from 114 to 343%. Kissel and Vigil (1994) that the overprediction was a result of soil disturbance as well as an improper model adjustment. It has also been concluded by other studies (Polglase et al., 1992 ; Lea and Ballard, 1982) that laboratory estimates such as potentially mineralizable

N estimates for forest soils have never been realized in the field because they are not responsive to site environmental factors through time.

Ion Exchange Membrane Soil Nitrogen Sampling

Ion exchange resins (IER) have long been used to estimate plant nutrient availability (Barrow and Shaw, 1977), as well as nitrogen mineralization (Hart and Binkley, 1985). Resins are very sensitive in detecting treatment effects in experiments (Binkley, 1986) and increases in nitrogen availability following harvesting (Binkley and Hart, 1989). In a study of 13 forest ecosystems, results of N accumulation in resin bags correlated well with other measures of N mineralization and dynamics (Binkley, 1986). Hart and Binkley (1985) show a 90% recovery of nitrate using 2M KCl extraction.

However, resin bags present a number of complications in that they alter water flow around and through the bag, as well as they are 3-dimensional in nature and therefore calculation of N per unit volume may be difficult (Subler et al., 1995). Also, a great deal of soil disturbance is encountered when utilizing the buried bag method.

Two-dimensional ion exchange membranes (IEM) overcome many of soil sampling problems, most namely that they minimize soil disturbance and may allow remeasurement of a specific point in soil through time (Johnson et al., 2005). Also, IEM measurements are sensitive to soil moisture and competition from microbes and roots (Johnson et al., 2005). As early as 1966, Vaidyanathan and Nye (1966) used ion-exchange papers to estimate effective ion diffusion rates in soil. Searl (1991) used IEMs to determine phosphate-extractable sulfate in soils, Cooperband and Logan (1994) used IEMs to measure soil-P in tropical silvo-pastoral systems. However there was no use of IEMs to measure N until 1995 (Subler et al., 1995).

Schnabel (1995) investigates the adsorption of nitrate and phosphate by two different resin materials, and their recovery using different extract solutions. Schnabel

(1995) shows that 2M KCl rendered the greatest recovery of nitrate from the resins during extraction.

Binkley and Matson (1983) used six laboratory methods for estimating N availability for comparison to ion exchange resin (IER)-N: 1) per-sulfate-peroxide digestion for total- N, 2) 1 M KCl extraction for initial extractable-N, 3) anaerobic incubation, 4) aerobic incubation, 5) boiling Water, and 6) autoclaving. IER were mixed bed cation and anion resins.

Binkley and Matson (1983) show that field IER-N estimates were greater than laboratory estimates. In addition, Binkley and Matson (1983) found significant treatment effects on field IER-N, whereas no treatment effects were found in laboratory-N estimates. Binkley and Matson (1983) concluded that field IER-N experiments demonstrate greater sensibility to on-site environmental characteristics and a more realistic estimate of field nutrient quantity, capacity, and intensity than do laboratory estimates.

DiStefano and Gholz (1986) used PVC cores with mixed-bed cation and anion exchange resins to measure mineralization in a 28 year old slash pine (*Pinus elliottii* L.) plantation in northern Florida. Using the exchange resins placed in the field and 2 N KCl extractions, this study measured much greater N concentrations in fertilized treatment areas than in controls (DiStefano and Gholz, 1986). Nitrate recovery by resins in the bottom of the cores increased by factor of 5 after fertilization, demonstrating the ability to capture leaching N. Ammonium also showed mobility with 50% of total extracted N being captured in the lower resin after fertilization. DiStefano and Gholz (1986) found initial IER-NH₄⁺ concentrations to be 8.4 mg-NH₄⁺ kg⁻¹, while after a 1 month incubation NH₄⁺ concentration was 17.5 mg-NH₄⁺ kg⁻¹. Net N mineralization was also measured

using this approach and was found to be $11.9 \text{ mg-N kg}^{-1} \text{ month}^{-1}$ during the growing season.

Subler et al. (1995), utilized buried 2-dimensional IEMs to determine the NO_3 uptake characteristics of IEMs, evaluate the utility of the IEM technique for *in situ* measurement of NO_3 availability and to investigate potential influence of the IEMs on soil N dynamics. Subler et al. (1995) showed that when a fresh IEM is placed in the soil, nutrient ions diffuse to the IEM and bind to its exchange sites, establishing a diffusion gradient in the soil. As the ions in the immediate vicinity of the soil are depleted, the diffusion gradient extends outward into the soil matrix, and the rate of diffusion approaches an equilibrium dependent upon the concentration of the ions in the soil matrix, the diffusive resistance of the soil, and the strength of the ion sink (Subler et al., 1995).

In this study Subler et al. (1995) used field soil in plastic cups with organic residue amendments of legume leaves, wheat straw, and legume leaves + wheat straw. Soils amended with legumes had greater initial N concentrations, more than half of which was ammonium. This study also showed that soil amended with wheat straw and legume leaves, with C:N ratios of 85 and 9 respectively, a period of initial N immobilization was followed by gradual increases in mineralization. IEM's also bound a large amount of NO_3 initially and decreased in NO_3 concentrations with longer incubation periods as the sink strength of membrane decreased. Therefore, IEM- NO_3 concentrations would not correlate well with soil NO_3 concentrations. However, Subler et al. (1995) argues that since IEMs are site-sensitive, they are still a good indication of plant available NO_3 ions in soil. This study showed that IEMs did not act as an infinite sink for NO_3 since the wheat straw amended soil exhibited NO_3 removal from IEM. NO_3 is bound to IEMs only by weak ionic bonds, and are freely exchangeable with other anions in the soil solution

(Subler et al., 1995). Subler et al. (1995) concludes that in the field, the influence of the IEM technique on soil processes may be relatively small as compared to other techniques that involve a great amount of soil disturbance (Subler et al., 1995).

Johnson et al. (2005), used PRSTM (Plant Root Simulator) membrane probes in a laboratory study to assess N status in prairie soil of the Pulaski series and to test PRS probe use against traditional resin and KCl extractable-N measurements. One month incubation periods were used, with sequential incubations for 3 months. Johnson et al. (2005) found NO₃-PRS was between 0.2 to 1.2 $\mu\text{mol cm}^2 \text{ day}^{-1}$, NH₄-PRS was between 0.005 to 0.01 $\mu\text{mol cm}^2 \text{ day}^{-1}$, and total N was between 0.4 to 1.2 $\mu\text{mol cm}^2 \text{ day}^{-1}$. Johnson et al. (2005), found that resin and extractable-N were significantly affected by moisture and temperature while PRSTM was only affected by moisture. Soil solution is the major vector of contact between soil exchange sites and resin surfaces (Johnson et al., 2005). This study concluded that the greater sensitivity of the probes and resins to soil moisture is more reflective of plant root activity and that PRSTM membrane probes provide a measure of integrated soil processes over time while mineral soil sampling merely a snapshot in time (Johnson et al., 2005).

Pine Plantation Response to Urea Fertilization

Urea is the most widely used source of N in forestry, because of its high N analysis (46-0-0), favorable physical properties, and low cost of application (Pritchett and Fisher, 1987). Urea is synthesized by the Haber-Bosch process, where N₂ and CH₄ are passed over a metal catalyst at high temperature and pressure to produce NH₃, which is then transformed into urea (NH₂)₂CO (Booze-Daniels and Schmidt, 2004). Urea is water soluble, and therefore is very reactive in forest floor and mineral soil after application. Urea hydrolysis is an enzymatically driven process, therefore, after application, the fate of urea-N is determined by several environmental characteristics including air

temperature, humidity, soil moisture (Wells and Allen, 1986), timing of rainfall (Kissel et al., 2004), and the amount of organic material in the forest floor (Hauck, 1968). If urea is not immediately incorporated into the mineral soil through dissolution by rainfall, urea reacts with water and urease, an enzyme naturally found in forest floor material to produce ammonium and carbonate ions. Upon further hydrolysis, the carbonate releases a hydroxyl ion which increases the pH of the soil environment immediately surrounding the urea molecule. It is at this point when either 1) urea nitrification may occur due to the stimulation of the nitrifying bacteria, *Nitrosomas*, by a higher pH (Hauck, 1968), or 2) ammonia volatilization may occur by the deprotonation of ammonium resulting in ammonia gas which diffuses out of the soil due to the difference in ammonia partial pressure between the soil and atmosphere. Other possible fates for urea-N include biological immobilization of NH_4^+ , chemical immobilization in humus, silicate clays, and on cation exchange sites (McGoll and Powers, 1984). Mineralization of soil organic N is frequently stimulated by addition of urea upon the formation of alkaline conditions in the soil (Hauck, 1968). This results from solubilization of humic compounds followed by increased biological activity (Viro, 1963). In highly leached and acid forest soils, ammonium can be the predominant N form after fertilization, as a result of low soil nitrifying capacity (Hauck, 1968). Binkley (1986) states that in general, after urea-N fertilizer application: 1) less than a quarter of applied N is taken up by trees ; 2) most of the fertilizer-N is immobilized in microbial biomass and soil organic matter, and 3) some portion of fertilizer-N is lost through leaching and volatilization. Typically, only 10-40% of added urea-N is acquired by vegetation (Hulm and Killham, 1988; Johnson et al., 1980) . Furthermore, fertilizer N recover may be as low as 23%, and the larger amount applied, the more inefficient the application (Bengston, 1981; Hauck, 1981; Ducey and Allen, 2001).

Rates of 100-300 kg-N ha⁻¹ plus 25 – 100 kg-P ha⁻¹ are commonly prescribed for N fertilization in mid-rotation, and multiple fertilizer applications are made throughout a rotation (Powers, 1984). Though nutritional studies show that photosynthesis may increase between 10% and 30% as a result of fertilization, and volume response is usually greater than 30%, the increased volume growth is more likely due to increased leaf area after fertilization (Vose and Allen, 1988). Net volume-growth responses to fertilization were shown to be greatest at moderate stocking levels of 20-28 m² ha⁻¹ of basal area for loblolly pine (Wells and Allen, 1986). At these levels, individual crowns can still expand and stand foliage biomass is sufficient to utilize the nutrient amendment. Thinning reduces overall stand volume and shifts the stand towards a greater density of larger trees (Fisher and Binkley, 2000). For residual trees to adequately respond to the thinning, adequate nutrition is needed to increase the canopy leaf area required for increased volume growth (Binkley, 1986). Volume responses to standard mid-rotation fertilization rates with urea have been shown to yield an increase in mean annual increment of 5.25 m³ ha⁻¹ year⁻¹ for at least 6 years (Allen, 1985). Similarly, in a south-wide regional fertilization response study in loblolly pine plantations, fertilization increased mean annual increment by 5.74 m³ ha⁻¹ year⁻¹ for 8 years (Forest Nutrition Cooperative, 1995). Mid-rotation fertilization can be viewed as an acceleration of stand development as response to fertilization shifts the stand diameter distribution toward fewer larger trees and in turn affects product classification (Allen, 1987). Morris and Myerscough (1991), also suggest that fertilization allows trees to grow larger per unit area before self thinning would occur. Fertilization late in the rotation can be very attractive financially because yields increase, the increased biomass may be in premium product categories, and the investment period is short (Binkley, 1986). However, typical rates of fertilization do not increase the N capital of a site and soil N availability is typically increased for only 1-2

years (Allen, 1987). Therefore, it is generally accepted that N fertilization can only be considered a treatment for enhancing short-term growth, but not for site quality amelioration (Miller, 1981 ; Vitousek et al., 1983 ; Allen, 1987 ; Forest Nutrition Cooperative, 2005b).

Fertilization Effects on Leaf Area Increment

Cannell (1989), states that the amount of dry matter produced by a forest stand is linearly related to the amount of light energy intercepted by the foliage canopy. The rate of growth in dry weight and leaf area of individual trees is also linearly related to the rate of N supply to the roots (Ingestad, 1982). Similarly, Duzan et al. (1982) also concluded that growth response to fertilization depends on increasing light interception, photosynthetic efficiency of leaves, and altered allocation among tissues. Light interception is principally a function of the amount of leaf area, and the duration of leaf area display (Allen and Albaugh, 1999). Brix (1983), estimated that 2/3 of increased growth response in Douglas-fir to N fertilization came from increased leaf area and light interception and 1/3 from increased photosynthetic efficiency. Pine plantation stands with well structured canopies, long-lived needles, favorable water balance, ample nutrients and high levels of solar radiation will develop the greatest LAI values. N fertilization was shown by Miller and Miller (1976) to increase leaf retention and LAI (Miller and Miller, 1976).

In the southeast, low nutrient availability has been shown to be the principal factor in causing suboptimal LAI (Colbert et al., 1990; Vose and Allen, 1988). Allen and Albaugh (1999) found that in southeastern pine plantations, low nutrient availability was the principal factor causing suboptimal levels of leaf area and therefore production. Therefore it is accepted that improving stand resource availability can dramatically improve stand productivity through the mitigation of suboptimal LAI status (Albaugh et

al., 1998; Colbert et al., 1990; Vose and Allen, 1988). This was demonstrated by Albaugh et al. (1998), in findings that both leaf area and growth efficiency in a pine plantation were increased with increased resource availability (Albaugh et al., 1998).

In an investigation at the SETRES study, Allen and Albaugh (1999) show that optimal nutrition amendment increased LAI and volume growth, while irrigation alone produced only minimal growth responses. Even with dramatic responses to resource additions, leaf area and volume growth remain strongly coupled throughout the six year study with leaf area accounting for ~90% of the variation in volume growth (Allen and Albaugh, 1999).

Rojas (2005) shows that fertilization increased soil nutrient availability resulting in greater leaf area production and stand growth, 2 to 5 years after planting. This study also showed that peak of leaf area increases sharply with average growing season available N. Enhanced nutrient availability through fertilization resulted in better foliar nutrition and foliar production (Rojas, 2005). As found in other studies (Jokela et al., 2004; Rojas, 2005; Sampson and Allen, 1999), a strong relationship between volume growth and leaf area was found by Rojas (2005), indicating that increased foliage was the primary response to nutrient additions. Rojas (2005), concluded that fertilization positively affected foliar N, foliage quantity, volume growth, litterfall, and litterfall N concentration, translating into greater forest floor N accumulation.

Albaugh et al. (2004) studied pine plantation responses to 9 years of receiving optimal water and nutrition of $100 \text{ kg N ha}^{-1} \text{ year}^{-1}$ resulting in average foliar N level of 1.35%. This study showed that 9 years after yearly N additions, fertilized plots significantly increased LAI over control plots. The results from the fertilized stands in this study demonstrated 3 times the total standing biomass and 2 times basal area at age 16 than control plots (Albaugh et al., 2004). It is now generally accepted that much of

the variation in wood production can be accounted for by variation in light interception (Allen and Albaugh, 1999).

Fertilization Effects on Litterfall and the Forest Floor

Nutrient accumulation, distribution, and cycling within forest ecosystems closely parallels that of dry matter (Switzer and Nelson, 1968). Most fertilizer N retained in plantations of the temperate and boreal zone eventually resides in the forest floor (Miller, 1981). Accumulations of 100-400 kg ha⁻¹ N and up to 35 kg ha⁻¹ P in forest floors have been reported for various 15- to 20- year-old loblolly pine stands in the southeastern USA (Jorgensen et al., 1980; Switzer and Nelson, 1972; Wells and Jorgensen, 1975). Piatek and Allen (2001), found total N and P accumulation in the forest floor to be 193-270 kg N ha⁻¹ and 5.2-7.0 kg P ha⁻¹, depending on treatment. Nutrient inputs to forest floors in annual litter fall in this study were 20 kg N and 1 kg P ha⁻¹ year⁻¹ at age 15. These accumulations seem to represent nutrient input in litter fall throughout the stand's life.

Piatek and Allen (2000), measured 4500 - 4800 kg ha⁻¹ year⁻¹ of fresh litterfall added to the forest floor in a mid-rotation loblolly plantation. Fresh litter was determined to be a source of labile C for microbial use that is readily colonized (Piatek and Allen, 2001). Thus, the mechanism for the sink activity of these forest floors may include microbial growth and translocation upward, from older litter to fresh litter and to labile C sources (Piatek and Allen, 2001). Therefore, Piatek and Allen (2001) found that N and P may cycle from 1- and 2-year-old litter to fresh litter, and little or no N and P may be available for release at the bottom of the forest floor. Chadwick et al. (1998) had previously shown that further translocation of N and P may occur as growth and migration of fungal hyphae or as diffusion from higher to lower concentration along continuous water films. Movement of N in fungal filaments from mineral soil to the forest floor has also been described (Hart and Firestone, 1991).

Rojas (2005), found significant effects of fertilization on litterfall, with fertilized plots accumulating 227% more litter than control plots. Greater litterfall containing greater amounts of N translated into greater N accumulation in the forest floor effectively lowering the C/N ratios (Rojas, 2005). McGoll and Powers (1984), also show that adding N to the forest floor decreases the C:N ratio of litter and humus and which accelerated decomposition. This was shown in Piatek and Allen (2001), who found that after 26 months, all leaf litter in the forest floor had lost 55% of its original biomass. Additions of NH_4 fertilizers such as urea generally increase numbers and activity of ureolytic bacteria and fungi in the litter and humus, and the production of NO_3 indicating rapid microbial response to excess NH_4 (McGoll and Powers, 1984). Though N fertilization may increase N accumulation in the forest floor, Piatek and Allen (2001) summarize that forest floor could be considered an N sink since fresh litterfall demonstrated net immobilization and little net N mineralization occurred from old litter, thus preventing net release of N and P from the forest floor

Fertilization Effects on Microbial Activity and N Availability

Silvicultural manipulations have been shown to alter microbially driven soil processes such as nitrogen mineralization, immobilization, nitrification, and denitrification (Allen, 1987; Burger and Pritchett, 1988; Vitousek and Matson, 1985; Li et al., 2003). Total soil N declines as soil N accumulates in vegetation and forest floor (Richter et al., 2000), while soil C increases. The gradual increase in C:N ratio suggest that the potential for N immobilization increases with stand development (Gurlevik et al., 2004). The significance of immobilization in C-rich forest soils has been previously demonstrated (Raison et al., 1992; Vitousek and Matson, 1984; Vitousek and Matson, 1985). Bauhus and Khanna (1999) reported that about 800 kg-C ha^{-1} , 150 kg-N ha^{-1} , and 45 kg-P ha^{-1} could be stored by microbial populations found in the forest floor and top

horizons of the mineral soil in an average forest ecosystem. It has also been shown that changes in pH greatly affect microbial communities, with more acidic or alkaline soils favor fungal growth more than bacterial growth (Parr, 1968). Parr (1968) states that in the case of urea application on acidic forest soils, an alkaline environment forms around the pellets, possibly raising the pH of forest floor to become more beneficial for bacteria.

Conflicting studies showing increase and decrease in microbial biomass in response to N fertilization (Lee and Jose, 2003). Lee and Jose (2003) studied the effects of continual N fertigation on 7 year old loblolly plantations on Redbay sandy loam soils of Northern Florida. Lee and Jose (2003) found that soil microbial biomass C was significantly negatively affected by N fertilization. Lee and Jose (2003) found microbial biomass C was strongly correlated to SOM and soil pH. Lee and Jose (2003) found that loblolly pine soils were more acidic after 7 years of litterfall than a nearby hardwood site. Lee and Jose (2003) suggest that there is an optimal pH range of 6 ± 0.4 , where microbial activity could be the highest. Humus is divided into the active, passive, and slow fractions.

Li et al. (2005) investigated the effects of balanced and optimal fertilization on soil microbial biomass C and N using chloroform fumigation methods, as well as microbial functional diversity using the Biology micro plate method. After fertilization, their results show significantly greater microbial C and N in fertilized plots than in control plots, immediately following the fertilization (Li et al., 2005). This is in contrast to previous studies (Lee and Jose, 2003) that show negative effects on microbial biomass C, though in these studies balanced nutritional amendments were not provided. Li et al. (2005) also show a seasonal effect on microbial biomass C and N, where an increase in microbial biomass occurred in early spring translating into higher rates of net N mineralization. Fertilization did not affect functional diversity of microbes in the

rhizosphere, however microbial selection of C compounds was affected by fertilization (Li et al., 2005).

The relationship between immobilization and mineralization is crucial in nutrient availability in forest systems (Gosz, 1984). Vitousek (1982) showed a strong positive relationship between the N content in litterfall and N mineralization rates. Mineralization depends upon chemical composition of the substrate as C:N, lignin and cellulose contents (Khanna and Ulrich, 1984). The ammoniacal-N from this process is what is utilized by trees (Cole, 1981) and microbes (Jansson, 1958). Reich et al. (1997) showed that wood production increased with net N-mineralization, with net N-mineralization explaining 50% of variation in wood production.

Several studies address the effect of N fertilization on the microbially driven processes of immobilization and mineralization. In many studies, fertilization was shown to increase N concentration in soil and net mineralization (Maimone et al., 1991). Raison et al. (1992) showed 2-3 fold increases in net N mineralization in radiata pine (*Pinus radiata* L.) when fertilized with 400 kg-N / ha for 4 years. Raison et al. (1992) also found that a continued higher rate of N mineralization was attributed to the remineralization of immobilized fertilizer N and decomposition of fine roots with high N content.

Vitousek and Matson (1985), through the use of N¹⁵ labeled ammonium sulfate fertilizer application, traced nitrate-N and ammonium-N processes as uptake, mineralization, immobilization, leaching, and denitrification. Vitousek and Matson (1985) found that soils from plots without herbicide or logging residue removal immobilized >90% of added N¹⁵ within 28 days, while those with residue removal and herbicide treatments immobilized <70%. The seasonal course of net nitrogen *in situ* was primarily controlled by soil temperature and moisture. *In situ* mineralization increased

from March through May or June as the soil warmed in both years, then declined precipitously as the soil dried later in the summer (Vitousek and Matson, 1985). The results clearly illustrated that microbial immobilization and mineralization controlled nitrogen pool sizes and losses.

Polglase et al. (1992), studied the release of N and P from litter from organic matter in a six year old loblolly pine stand, as affected by cultural treatments. The methodology for this study included both laboratory potential N mineralization and in situ field N mineralization estimates utilizing the sequential coring method of Raison (1987). Pre-treatment mineral soil concentrations of inorganic N in bulk soil were found to be quite low in this study, at less than $1.3 \text{ mg-N kg}^{-1}_{\text{soil}}$. The results of the in situ N mineralization study were used to calculate specific N mineralization, which is field annual net N mineralized as a percent of total soil N. The greatest annual rates of specific N mineralization were shown to occur in plots receiving a vegetation control plus fertilization treatment, though this effect was not significant. Polglase et al. (1992) also found that fertilizer did not cause an increase in the N concentrations of the Oi horizon needles. Since no inorganic N was detected, Polglase et al. (1992) argue that no inorganic N was immobilized during the initial stages of decomposition. Polglase et al. (1992) further state that these results demonstrate the inability of fertilizer to elicit a change in the chemistry or turnover rates of N throughout the decomposition profile.

Fox (2004), applied urea at incremental rates from 0 to 600 kg N ha^{-1} repeatedly at annual and 5 year intervals over 6 years and studied the effects on N mineralization potential. The repeated N fertilization increased N mineralization potential up to application rates of 450 kg N ha^{-1} , with higher rates decreasing N mineralization (Fox, 2004). Soil pH dropped following fertilization, with the greatest drops occurring in the greatest application rates indicating higher nitrification and base cation leaching with

higher application rates (Fox, 2004). The results from this study also demonstrated that growth of trees increased after several smaller applications of fertilizer over a period of years instead of after just one application at a high rate. Likewise N mineralization was found to respond more positively after several applications at lower rates over time than after just one dose (Fox, 2004).

Gurlevik et al. (2004), investigated vegetation and fertilization effects on the N cycle of a 14 year old loblolly stand in the piedmont of North Carolina. The stand was fertilized with 224 kg ha⁻¹ Urea plus 56 kg ha⁻¹ TSP in the late spring and monthly field N mineralization was measured using the sequential coring method with capped pvc tubes as described in Raison (1987). In addition laboratory aerobic N mineralization incubation took place monthly for 14 months. Gurlevik et al. (2004) found total KCl extractable-N ranging from 1.8 kg ha⁻¹ in controls to 2.8 in vegetation control and from 4.2 to 18 kg ha⁻¹ vegetation control plus fertilization treatments. This study also measured *in situ* N mineralization ranging from -5 to 14 kg ha⁻¹ month⁻¹. There was a significant effect of sampling date with greater N mineralization occurring during summer months. Fertilization plus vegetation control was found to consistently have greater N mineralization than other treatments (Gurlevik et al., 2004). In terms of net annual mineralization, Gurlevik et al. (2004) found that fertilization only increased N mineralization 24% over control, vegetation control 58% over control, and fertilization plus vegetation control 330% over control. Net N mineralization rates in this study were 22 kg ha⁻¹ year⁻¹ in control treatments, similar to those reported by Piatek and Allen (1999), and 96 kg ha⁻¹ year⁻¹ in the fertilization and vegetation control plots. These findings were reported to be in the 16 – 100 kg ha⁻¹ year⁻¹ range of N uptake by growing trees as reported by Ducey and Allen (2001). The results from this study also demonstrated that fertilization alone increased N mineralization by 19% over controls

(Gurlevik et al., 2004). Gurlevik et al. (2004), speculate that the increased N mineralization was derived from previously immobilized fertilizer N. After fertilization, Gurlevik et al. (2004) measured 5 immobilization dates the first year and 2 the second year similar to trends found by others (Mudano, 1986; Raison et al., 1992; Vitousek and Matson, 1985). Raison et al. (1992) showed that after an initial net immobilization of 37% of 400 kg-N ha⁻¹, the fertilized plots showed marked increases in N mineralization over controls for 4 years. However, the vegetation control only treatment in this study, increased annual net N mineralization more than fertilization relative to control, as shown in other studies (Li et al., 2003). Gurlevik et al. (2004) attribute this effect to the increase in soil temperature, reductions in immobilization, and vegetation uptake. Gurlevik et al. (2004) also show that using the assumption of a Q¹⁰ of 2 for net mineralization from (Stanford et al., 1973), then the approximate 2 degree C higher temperature measured in summer in the vegetation control plots explained about 30% of the increase in net N mineralization with vegetation control.

Gurlevik et al. (2004) explained that the strong positive interaction between vegetation control and fertilization found in their study was likely caused by a reduction in bioavailable Carbon with vegetation control and an increase in mineral N with fertilization. Gurlevik et al. (2004) also showed that N mineralization may also vary with seasonal changes in labile C inputs. Other research has documented seasonal variation in light fraction-C (Boone, 1994), potentially mineralizable C, dissolved organic C and fine root turnover (Gurlevik et al., 2004). Studies have shown that increased bioavailable-C inputs can result in greater immobilization of N (Compton and Boone, 2002; Gallardo and Schlesinger, 1995; Whalen et al., 2000). Gurlevik et al. (2004) concluded that vegetation-derived labile C is an important driver for net N mineralization.

Schimmel and Bennett (2004), challenge the classical paradigm that plants only uptake mineral N, resulting from nitrogen mineralization by soil microbes, and that N mineralization is the key event in the N cycle making mineral N bio-available. For several years, N mineralization assays were considered adequate measures of plant-available N (Adams et al., 1989; Nadelhoffer et al., 1983). Concerns about the methodology, especially the lack of roots, and disturbance effects caused N mineralization assays to be viewed as an index of available N (Hart et al., 1994). After several inconsistent studies on N mineralization in forest soils (Nadelhoffer et al., 1983; Polglase et al., 1992), concerns about N mineralization being the key aspect of N availability arose. These concerns lead to conclusions that trees may utilize organic-N in the soil instead of strictly mineral N left over by microbial scavenging for energy (Schimmel and Bennett, 2004). Also, that trees may, in certain circumstances, outcompete soil microbes for nutrients.

Trees may outcompete microbes for resources through two means. Primarily, through the use of mycorrhizae in their direct roles of decomposition and organic nutrient uptake (Attiwill and Leeper, 1987; Smith and Read, 1997) , and also through the interception of nutrients as they diffuse from microsites of high N availability to microsites of low N availability (Burger and Jackson, 2003).

The new paradigm of N availability recognizes that the critical point in the N cycle is the depolymerization of N-containing compounds. Polymers are not immediately bioavailable because they are too large (Chapin et al., 2002). They are first broken down by extracellular enzymes to release monomers such as amino acids and nucleic acids that are broadly bioavailable and may be used by plants or micro-organisms (Schimmel and Bennett, 2004).

Schimmel and Bennett (2004), describe four scenarios along a gradient of bioavailable soil N: 1) in extremely N-poor ecosystems, both plants and microbes may rely on organic compounds for their N ; 2) in greater N available systems, microbes will be less N limited and begin to mineralize N within the N-rich microsites, which would then diffuse away from the N-rich microsites into the N-poor microsites. The diffusing ammonium would be available and needed by plants and microbes, and thus plant and microbes are actively competing for the excess ammonium diffusing through the soil. 3) as overall N availability increases further, microbial N demand is met by local organic sources, decreasing their dependence on N diffusing from N-rich sites, and thereby decreasing competition between plants and microbes and increasing available N access for growing plants. In these conditions, mineralization would increase and ammonium would dominate in soil N pools, similar to moderately fertile temperate forests (Pastor et al., 1984). Since the generation of organic-N monomers increases along the gradient, it is quite possible that plant use of amino acids for N would continue even at high available-N levels.

In low soil N environments, mycorrhizae may be important in acting directly as decomposers by producing the exoenzymes that break down organic polymers, a mycorrhizal fungus enhances the likelihood that it will capture the monomers released and thus provide a more direct conduit between polymers and plants (Schimmel and Bennett, 2004).

For studies on the high-N side or those comparing systems across wide N gradients, net mineralization should correlate broadly with plant uptake and nutrient status (Schimmel and Bennett, 2004). Net mineralization retains its utility as an index of N availability but only as an indirect index, measuring something that is related to but not actually the key step regulating the cycle (Schimmel and Bennett, 2004). In

understanding C dynamics, models have always treated polymer breakdown as the rate-limiting step of overall decomposition and C flow to microorganisms (Schimmel and Bennett, 2004). The concept of N mineralization as the driving process in the N cycle, is shifting to one where exoenzyme-driven depolymerization is seen as the rate-limiting step in the generation of bioavailable-N (Chapin et al., 2002).

Fertilization Effects on Nitrogen Volatilization

Volatile losses from forest soil after surface-applied urea have ranged from 5-10% (Volk, 1961) to 20-40% (Nommik, 1973; Watkins et al., 1972), and in agricultural soils from 25 to 47% (Scharf and Alley, 1988). Volatile losses from non-fertilized forest soils are likely to be insignificant because conditions generally prevailing in these soils do not favor the formation of gaseous forms of nitrogen (Pritchett and Fisher, 1987).

Volatilization of free ammonia is insignificant below pH 7 and most forest soils are much too acid for losses from this route (Pritchett and Fisher, 1987). Ammonia volatilization is driven by the difference in ammonia partial pressure between the atmosphere and that in equilibrium with the moist soil (Mattos et al., 2003). Ammonium is converted to ammonia in significant amounts only at pH values above approximately 8 (Bates and Pinching, 1950). A sufficient number of hydroxyl ions are generated during urea hydrolysis to temporarily raise the surface pH of soils to 8 or 9 (Doak, 1952).

Incorporation of urea into the soil has been shown to effectively lower volatilization (Ernst and Massey, 1960; Fenn and Kissel, 1976). Rapid volatilization from urea generally occurs when soils are moist (Ferguson and Kissel, 1986). However, moisture from dewfall and humid air can be enough to stimulate ammonia volatilization on dry soils (Hargrove et al., 1977; Reynolds and Wolf, 1987). Rainfall and irrigation can both positively and negatively affect volatilization. Craig and Wollum (1982) showed that rainfall of 1 inch or more effectively washed urea into mineral soil, after which point,

ammonia volatilization was diminished. However, light rainfalls have been shown to stimulate volatilization by moistening the soil surface and promoting urease activity (Craig and Wollum, 1982; McInnes et al., 1986). Wind also dramatically increases volatilization rates by carrying ammonia away from the volatilizing surface, effectively increasing diffusion gradients (Kissel et al., 1977). Though wind may temporarily increase volatilization, it may also decrease volatilization over a longer period by drying the soil surface (Ferguson and Kissel, 1986). Volatilization is positively correlated to soil buffering capacity (Ferguson et al., 1984) and negatively correlated to CEC (Volk, 1959). In order to minimize ammonia loss after fertilization efforts have been made by adding compounds that inhibit the activity of urease in soil, hinder the diffusion of urea, or a combination of both (Nommik, 1973).

Nommik (1973) investigated the effects of urea pellet size on ammonia loss, and the effect of adding phosphoric acid or boric acid on ammonia loss. This study took place in a 9 year old scots pine (*Pinus sylvestris* L.) stand, fertilized at a rate of 200 kg N ha⁻¹. Nommik (1973) designed static ammonia sorbers that were installed in 3x3 meter plots that were covered with plastic 1-1.5 meters above ground. Nommik (1973), found that the increased pellet size decreased ammonia loss over a 28 day period, with small-pellet urea volatilizing 24% of applied N while large-pellet urea volatilized 20% of applied N. Also, Nommik (1973) showed that the amount of N volatilized decreases over time. Phosphoric acid amended urea (95% urea + 5% H₃PO₄) showed significant reductions in volatilization. Nommik (1973) determined that Phosphoric acid, effectively lowers the pH of the soil and inhibits urease activity in pellets and surrounding soil. A 5% addition of Boric acid to the urea pellets also significantly reduced ammonia loss. However, Boric acid, as a weak acid, did not actually inhibit urease activity through lowering soil pH, but acted as a mild, nonspecific, metabolic inhibitor (Nommik, 1973).

Bremner and Douglas (1971) had also previously shown that dihydric phenols and quinone additions to urea were the most effective at reducing ammonia loss. Through the use of N^{15} labeled urea, Nommik (1973) also showed that 92% of the ammonia trapped was derived from the applied urea, and an unexpected 8% of ammonia trapped was from native soil N. The ammonia volatilization from control plots in this study was found to be minimal at 0.7% (Nommik, 1973).

Beauchamp et al. (1978), used a series of glass suction flasks mounted on a mast at several heights above the ground to study volatilization of sludge material applied in the field. Flasks were filled with glass beads containing Phosphoric acid and incubated for 2 hour intervals on selected days after ammoniacal sludge application. Beauchamp et al. (1978), measured an exponential decay in ammonia volatilized through time and also determined that half of the ammoniacal N volatilized was captured between 3.6 and 5 days. Air temperature was highly correlated to ammonia flux rates, especially in the two or three days immediately following the application (Beauchamp et al., 1978).

Marshall and Debell (1980), investigate four methods for measuring ammonia volatilization after fertilization in forest soils and reported that lowest ammonia losses from urea fertilizer were estimated when using a closed-static chamber method. Artificial conditions created in such a closed environment were determined to hinder ammonia diffusion from the soil surface to the absorption discs and may have resulted in ammonia re-adsorption by soil (Marshall and Debell, 1980).

There have been few studies of ammonia loss after fertilization in southern pine plantations (Kissel et al., 2004). The existing studies such as Craig and Wollum (1982) found 3% losses in winter and 13% in spring. Volk (1970), and Boomsma and Pritchett (1979), found volatilization losses ranging from 1 to 10 % after fertilization. Previous studies also showed that with rainfall, urea may be dissolved quickly and moved into the

soil (Paramasivan and Alva, 1997). However, rainfall increases soil moisture, which also increases urea hydrolysis (Kissel and Cabrera, 1988). Faster urea hydrolysis at the soil surface would in turn increase the rate of ammonia loss from fertilization (Moe, 1967).

Kissell et al. (2004) studied a 24 year old loblolly pine plantation growing on Cecil sandy loam. This study utilized field based volatilization chambers which used soil that was cut out of the forest floor. Nine chambers in a line were covered under a canvas roof at 3m in height. Rainfall simulators were used to add varying amounts of water to the measurement plots after fertilization at different times. A laboratory study was also initiated to investigate more small scale changes in rainfall timing effects. Results showed that rainfall occurring on the same day of urea application reduced ammonia losses to less than 1% of applied urea during Aug. 9 to Sept. 7 (Kissel et al., 2004). However, when urea was allowed to slowly dissolve into the forest floor material by morning dew and rainfall was added on day 5 during the March through May trials, ammonia losses were significantly greater with increasing rainfall added. Up to 45% was lost after 28 days when 40mm of rain was applied on day 5 (Kissel et al., 2004). The greatest volatilization rates, which were 58% of applied urea-N, occurred during the 58 day incubation period from Sept. 16 – Nov. 14, when rain was applied on day 16 or 30. The greater volatilization during this period was attributed to higher air temperatures, humidity, and greater natural rainfall (Kissel et al., 2004).

One explanation for the increased volatilization after slow dissolution of urea into the forest floor is that abiotic immobilization occurs in forest floor which may prevent ammonia incorporation into mineral soil, as shown by other studies (Axelsson and Berg, 1988; Johnson et al., 2000). Kissel et al. (2004) concluded that if rain dissolves the urea pellets, a greater amount is incorporated into the mineral soil, however if morning dew is

allowed to slowly dissolve the pellets, more is retained by the forest floor and may be volatilized up to 58 days post fertilization (Kissel et al., 2004).

Mattos et al. (2003), investigated N volatilization in a Florida sandy entisol under a citrus plantation after winter fertilization with urea or ammonium nitrate at a rate of 230 g N per tree, and constant soil moisture control at field capacity. This study utilized Nommik et al. (1973) semi-open static volatilization chambers but were outfitted with internal fans for simulation of forest floor windspeed. Results from this study showed that volatilization of urea was much greater than with ammonium nitrate throughout study. Volatilization for UR was 12.8% with the fan off and 33% with fan on (Mattos et al., 2003). Volatilization was significantly affected by soil and air temperature (Mattos et al., 2003). Urea also changed the acidity of soil to a pH of 9, while ammonium nitrate remained around native soil pH of 6.9

Pine Plantation Response to Controlled Release Nitrogen Fertilization

Fertilization with urea has consistently shown advantageous growth responses, both early and late in a rotation. However, urea also has several properties making it inefficient in its delivery to crop trees. Hauck (1968) stated that N recovery in crop trees after fertilization was accepted at 50 – 75% and that losses of 10-20% of urea-N would always be unavoidable. These properties include rapid water solubility leading to leaching losses (Booze-Daniels and Schmidt, 1997; Fisher and Binkely, 2000), and volatilization losses (Nommik, 1973; Kissel et al., 2004). In addition, the rapid N-release pattern of urea far exceeds the needs of soil microbes and plant roots (Hauck, 1968), and does not match the relatively slow and prolonged N-uptake of crop trees throughout a growing season (Voigt, 1968). Ecosystem retention of fertilizer nutrients is important, not only for extending the period of plantation growth response, but also for minimizing contamination (McGoll and Powers, 1984). Understanding of these urea inefficiencies

has led to the development of less soluble forms of fertilizer N. It was postulated that by using N fertilizers with slow release characteristics, the inefficiencies of soluble nitrogen could be avoided allowing larger N fertilization rates at planting which in turn could reduce application costs and prolong fertilization effects (Smith et al., 1971). Also, slow release fertilizers were hypothesized to be more efficient because their release characteristics would more closely match the uptake requirements of slow growing pine (Hauck, 1968).

Controlled-release fertilizers differ from conventional forms of fertilizer in that the majority of nutrients are not available immediately following application but released slowly over time (Jacobs et al., 2003b). The first control release N product developed was ureaformaldehyde, commercially known as ureaform, and was mainly investigated for use in turfgrass (McIlvanie et al., 1980), ornamental (Mikkelsen et al., 1994), nursery (Jacobs et al., 2003b), or seedling N fertilization. The ureaforms range from highly condensed, polymeric urea-formaldehyde resins to products containing a high percentage of short-chain, water-soluble molecules and unreacted urea which readily release crop-available N (Hauck, 1968). One example of commercially available ureaform is Nitroform®, produced by Nu-Gro Technologies, Incorporated. During manufacturing, the urea-formaldehyde reaction produces alkylated urea polymers of differing molecular weights and polymer chain lengths (Goertz, 1993). The polymer chain length is the mechanism by which nutrient release is regulated over time with longer chain length polymers being water insoluble and requiring microbial activity for decomposition and N release (Jacobs et al., 2003b). Because of the dependence on microbial activity, N release from ureaform is also dependent upon the environmental conditions that regulate microbial activity such as soil moisture, pH, and temperature (Allen, 1984; Jacobs et al., 2003a). Another method of control release technology is that of the coated ureas,

including Osmocote®, Prokote Plus®, and sulfur-coated urea. These coated urea products have also been used in the horticulture and forest industry, with several studies and literature reviews focusing on their use (Mikkelsen et al., 1994; Army, 1963; Scharf and Alley, 1988). However, the focus of this literature review will be on the ureaform and methylene urea class of control release N fertilizer.

Ureaform, a “sparingly” soluble ureaformaldehyde contains less than 15% unreacted urea with long chain polymers such as trimethylene urea and others (Booze-Daniels and Schmidt, 1997). Methylene ureas, which were developed in the 1960's and 1970's, contain intermediate length polymers with 15-30% unreacted urea and can be made with various amounts of water insoluble nitrogen compounds (Booze-Daniels and Schmidt, 1997). May and Posey (1956), tested pine seedling growth after fertilization with a single application of 250 kg ha⁻¹ ureaform-N or 376 kg ha⁻¹ of ammonium nitrate distributed over 8 sequential applications. Their results showed no statistical difference in seedling growth, and concluded that ureaform would be operationally and economically feasible when fertilizing seedlings (May and Posey, 1956). Austin and Strand (1960) established a series of trials in the 1950's which demonstrated an increase in Douglas-fir seedling survival at plantation establishment after fertilization with ureaform. Bengtson and Voigt (1962) showed in a laboratory setting that ureaformaldehyde leaches much less readily than ammonium nitrate, and recommended its use in areas of high rainfall or tree nurseries.

Voigt (1968), suggested that the application of nutrient sources with slow release characteristics might increase pine growth response. However, he predicted that the effects on nutrient cycling would be difficult to predict since the release would be controlled by decomposition rate in forest soils (Voigt, 1968). A substantial amount of ureaform-N may be tied up in soil organic matter or incorporated into microbial cellular

structures, and therefore remineralization is required before the N becomes available to the crop (Hauck, 1968). This reversion of immobilized N from ureaform into organic matter forms which are successively more resistant to mineralization, may further reduce the efficiency of slow-release N in meeting the N requirements of crop trees (Hauck, 1968).

Hadas et al., (1975) studied the mineralization of ureaform in the field during the three weeks following application, in small plots of a banana plantation at two different seasons of the year. During winter, Hadas et al. (1975) found an increase of both ammonium and nitrite in the soil which persisted for 2 weeks. During summer application, only a small increase in ammonium was noted for 2 days. Hadas et al. (1975) concluded that the differences in mineralization were due to changes in microbiological activity and mineralization of nitrogen from sources other than the fertilizer.

Duffy (1977) investigated ureaform fertilization on juvenile loblolly pine on sandy and loamy sites in Mississippi to determine if its slow release of N would produce a response similar to that of several small applications of ammonium nitrate.

Ureaform was applied as 300 kg-N ha^{-1} at one application, while ammonium nitrate was applied at three annual application times summing to 300 kg-N ha^{-1} after three years. All fertilized plots produced greater foliage biomass in comparison to control plots, however, on the loamy site, the ammonium nitrate application produced statistically more foliage than ureaform, while on the sandy site no differences were found in foliage production (Duffy, 1977).

Maritikainen (1984), studied nitrification in forest soils after fertilization with urea and ureaform and found significant nitrification pulses after fertilization with urea, and no nitrification from ureaform. However, a significant ammonium accumulation was found in ureaform fertilized plots (Maritikainen, 1984). This study shows that in acid

soils, ureaform provided the greatest amounts of extractable ammonium and least amount of nitrification, concluding that in low pH forest soils, nitrification of ureaform is inhibited by higher levels of ammonium or by enzyme activity of mycorrhizae (Maritikainen, 1984).

In a laboratory setting, Christianson et al., (1988) studied mineralization and nitrification of the various water-soluble components of ureaform over a 35 day period using of N¹⁵ and high pressure liquid chromatographic techniques. Christianson et al., (1988) show that only the longer chain polymer fractions of ureaform, including dimethylenetriurea and triuret, showed any slow-release characteristics, whereas other more water-soluble fractions mineralized rapidly. No mineralization of the less soluble fractions of ureaform were measured during this experiment. Christianson et al., (1988), also found that due to their reduced solubility, ureaform exhibited a 37% reduction in volatilization loss over urea.

Praveen-Kumar and Brumme (1995) also tested the mineralization of ureaform-N consisting of differing chain lengths and reported a delay period in the mineralization for all alkylated urea fractions. However, after the delay period, mineralization occurred as rapidly as for urea (Praveen-Kumar and Brumme, 1995). They found the mineralization delay period generally increased with increasing chain length as follows: methylurea < 1,3-dimethylurea < ethylurea < butylurea < 1,1-dimethylurea , 1,3-diethylurea (Praveen-Kumar and Brumme, 1995).

It is currently accepted that control-release N fertilizers with long chain polymers such as ureaform, can be beneficial in nursery or early plantation settings because of lower water solubility and salt index (Pritchett and Fisher, 1987). It has also been shown that ureaform-N fertilizers can provide a long term N source (Booze-Daniels and Schmidt, 1997), and better coincide with plant nutritional needs (Hauck, 1985).

However, control-release fertilizers are high in price, ranging from 2 to 6 times greater per unit N than traditional N fertilizers (Hauck, 1968). Their successful use in mid-rotation southern pine fertilization must be based on demonstrated uptake use efficiency (Hauck, 1968), and their ability to minimize loss through N-volatilization and leaching.

CHAPTER III.

EFFECTS OF A CONTROLLED RELEASE NITROGEN FERTILIZER AND THINNING ON THE NITROGEN DYNAMICS OF A MID-ROTATION LOBLOLLY PINE PLANTATION IN THE PIEDMONT OF VIRGINIA

Abstract

Nitrogen deficiency is characteristic of many mid-rotation loblolly pine (*Pinus taeda* L.) plantations in the Piedmont region of the southeastern USA. Fertilization with urea is the most common method used to correct this deficiency. Previous studies show that urea fertilization produces a rapid pulse of available Nitrogen (N) with only a portion being utilized by plantation trees. Controlled release fertilizers release available N more slowly over a longer period of time and therefore may result in greater uptake efficiency. The objective of this study was to compare Nitroform®, a urea-formaldehyde controlled release N fertilizer versus urea and a control by measuring the effects of the two fertilizer treatments on N availability and loss as: total KCl extractable-N, total ion exchange membrane-N (IEM-N), N mineralization, and N volatilization, in a mid-rotation loblolly pine plantation in the Piedmont of Virginia. In addition, mid-summer and mid-winter fertilizations were compared to assess fertilizer uptake as a function of season. After the summer fertilization, Nitroform® significantly increased total KCl-extractable N, IEM-N, and N mineralization for two to three months over urea and the control. Three hundred times more N volatilized from urea than from controlled release Nitroform®. Interestingly, seven months after the summer application, the controlled release Nitroform® showed marked immobilization for three months while urea demonstrated greater N mineralization. After the winter application, fertilization with urea demonstrated greater soil inorganic N concentrations for two to three months over Nitroform®, very little N was immobilized, and volatilization was only 10 times that of

Nitroform®. After summer and winter fertilizations, both fertilizer treatments significantly increased soil inorganic N concentrations and N volatilization over controls, however did not significantly increase N mineralization over controls when average response was tested over the entire sampling period. In addition to the fertilizer effects measured, a thinning only treatment was also incorporated into this study with soil N-availability indices compared to a control with no thinning or fertilization. The results from the thinning only treatment demonstrated no significant increases over the control in total KCl extractable-N, IEM-N, N-mineralization, or N volatilization when average responses were tested over the entire sampling period.

Introduction

A high efficiency and increase in production of fiber and wood products is a crucial part of forest management across the southeast. One aspect of management that is essential to efficient production of loblolly pine plantations is nutrition management. Nitrogen deficiencies have been shown to severely limit growth in pine plantations in Virginia and across the southeast (Jokela, et al., 1991). Nitrogen fertilization is required in most loblolly pine plantations to obtain optimal growth rates. Fertilization with 100 kg-N ha⁻¹ has been shown to increase production of Piedmont middle rotation pine stands by 17.5 m³ ha⁻¹ over five years (Allen and Ballard, 1983). This increase in production is therefore important for an increased financial return. Internal rate of return from fertilizer investments can range from 15% to 20% (Fox, et al., 2004). Realizing this enhanced production and greater return on investment, approximately 600,000 hectares were fertilized with nitrogen on an annual basis in the southeast during 2005 (Forest Nutrition Cooperative, 2005b).

The most commonly used N fertilizer in forestry is urea, primarily due to its low cost. However, uptake efficiency by pines in plantations may be as low as 25% of

applied N (Binkley, 1986). The pathways of N-loss following fertilization that lead to this low tree uptake efficiency, occur through several processes: 1) ammonia volatilization (Nommik, 1973; Kissel et al., 2004), 2) uptake by competing vegetation (Smethurst and Comerford, 1993), 3) microbial immobilization (Fisher and Binkley, 2000; Schimmel and Bennett, 2004), and 4) nitrate leaching (Binkley, 1986). There is great opportunity to increase southern pine production and financial return by improving the uptake efficiency. One possible method of achieving this goal is through the use of controlled release fertilizer.

Traditional water soluble urea fertilization provides a rapid release of N over a short period of time. Mudano (1986) reported that surface soil available N declined to pre-fertilization levels after 160 days. These short-lived high levels of available N have been shown to exceed the maximum uptake rate of trees (Voigt, 1968; Binkley et al., 1999). Furthermore, the increased growth from fertilization has been shown to decline to pre-treatment levels over a 5 to 8 year period (Ballard, 1984). The ability of controlled release fertilizers to slowly release N over a much longer period would be more in phase with the N demand and uptake by crop trees throughout the growing season, resulting in greater efficiency of fertilizer uptake (Hauck, 1985). It has been shown in horticulture and nursery studies, that controlled release N fertilizers increase N retention in soils, N uptake in plants, and decrease N loss through leaching (Alexander and Helm, 1990; Rose, 2002). Enhanced uptake may allow for a lower application rate of fertilizers applied to pine plantations during mid-rotation, decreasing overall costs, while at the same time realizing a greater increase in production of crop trees. Although, the cost of controlled release N fertilizer is 2-3 times greater on a per ton basis than traditional urea, it may be economically viable because the trees might uptake a greater amount of N due to the increased N availability in the soil. There has been little research investigating the soil,

tree, and mid-rotation stand responses to controlled release N fertilizers that would lead to increased uptake efficiency, greater production, and greater financial return on investment. This study investigated the ability of Nitroform®, a ureaformaldehyde controlled release N fertilizer, to successfully increase soil N retention in a mid-rotation loblolly pine plantation in the Piedmont of Virginia. The effects from controlled release Nitroform® fertilization were compared to those of a traditional water soluble urea fertilization, as well as to a non-fertilized control. Because a thinning operation was also implemented in this study, the effects of thinning alone without fertilization on soil N availability will also be tested against a non-thinned, non-fertilized control.

The following null hypotheses were tested in this study:

HO₁: The effect of thinning only without fertilization on soil N availability will be no different than the control.

HO₂: The soil inorganic N availability after fertilization with controlled release Nitroform® will be no different than the soil inorganic N availability after fertilization with urea, and the effect of the fertilizer treatments on soil inorganic N availability will be no different than the control.

HO₃: Soil N mineralization after fertilization with controlled release Nitroform® will be no different than soil N mineralization after fertilization with urea, and the effect of the fertilizer treatments on soil N mineralization will be no different than the control.

HO₄: Nitrogen volatilization after fertilization with controlled release Nitroform® will be no different than fertilization with urea, and the effect of the fertilizer treatments on N volatilization will be no different than the control.

Materials and Methods

Site Description

The study site was located on the Reynold's Homestead Forest Resources Research Forest in the Piedmont physiographic province of Patrick County, VA (36°39' N, 80°90' W). The soils are highly weathered, kaolinitic, thermic, Typic Kanhapludults of the Cecil and Lloyd soil series. Due to soil erosion from past agricultural use, the top 15 cm of soil consisted of a very shallow sandy loam A horizon (≤ 7.5 cm) over an exposed Bt clay horizon. The pre-treatment stand was a 4 ha, 22-year-old loblolly pine plantation, machine planted in rows at a 2.4 m x 1.8 m spacing in 1982 following a shear, pile, and disc site preparation treatment (Fox, 1984). The average stand basal area was $34 \text{ m}^2 \text{ ha}^{-1}$ and site index (base age 25 years) was 20.4 m.

Experimental Design and Treatments

The experimental design was a randomized complete block design with four blocks of six treatments. During July of 2003, twenty-four, 0.04 hectare measurement plots centered within 0.09 hectare treatment plots were established in the stand. The treatment plots were separated by a distance of 6 to 9 meters. Total height, length of live crown, and DBH were measured for each tree within the treatment plots. The plots were then blocked according to pre-treatment basal area and height to account for any residual growth differences since the time of stand initiation.

After the plots were established, all arborescent understory vegetation was removed within each plot manually using a chainsaw. The competing vegetation included both hardwoods and wildling Virginia pine (*Pinus virginiana* L.). In addition, herbaceous understory vegetation and smaller arborescent competing vegetation was removed chemically using a foliar application of 5% Round-Up (Monsanto Co., St. Louis, MO) with a backpack sprayer. One-hundred percent of the competing vegetation was controlled within each treatment plot throughout the duration of the study period.

The six thinning, fertilizer type, and fertilization timing treatments were randomly assigned to the measurement plots according to the following regime: 1) no thinning / no fertilization (Check), 2) thinning only (TH), 3) thinning + 224 kg-N ha⁻¹ as urea + 28 kg-P ha⁻¹ as TSP in summer (URS), 4) thinning + 224 kg-N ha⁻¹ as control release ureaform + 28 kg-P ha⁻¹ as TSP in summer (CRS), 5) thinning + 224 kg-N ha⁻¹ as urea + 28 kg-P ha⁻¹ as TSP in winter (URW), and 6) thinning + 224 kg-N ha⁻¹ as control release ureaform + 28 kg-P ha⁻¹ as TSP in winter (CRW).

At the time of study installation, the average basal area for the stand ranged from 27 - 45 m² ha⁻¹, which is generally considered overstocked for loblolly pine plantations. At this high level of stocking, little growth response would be expected to occur following fertilization (Duzan et al., 1982). A low-thinning operation was implemented in a portion of the treatment plots. These plots were thinned to a basal area of 25.3 m² ha⁻¹ plus or minus 2.3 m² ha⁻¹. Residual basal area was left relatively high to reduce the potential damage from ice storms that are common in this region. Before thinning, the trees to be harvested from each treatment plot were marked with the following priorities: 1) removal of intermediate or suppressed trees, 2) appropriate residual basal area, and 3) even spacing left between residual trees. The thinning operation took place in the treatment plots from July 15 – 30, 2004, and was performed by manually felling the marked trees using chainsaws. The felled trees were bucked, limbed, and left on the forest floor within each treatment plot.

Nitrogen was applied in each fertilization treatment plot at a rate of 200 kg-N ha⁻¹. The summer fertilization treatment occurred on August 1, 2004, and the winter fertilization treatment on February 1, 2005. The two granular N-fertilizers used in this study were a traditional urea-N fertilizer (Southern States Cooperative, Inc., Richmond, VA), with a formulation of 46-0-0, and a controlled release-N fertilizer with a

formulation of 38-0-0. The controlled release-N fertilizer was Nitroform[®] - Blue Chip (Nu-Gro Corporation, Grand Rapids, MI), in which the 38%-N fraction consists of 4.5% immediately available water soluble urea, 6.9% slowly water soluble coated urea, and 26.6% water insoluble ureaform. The ureaform portion of Nitroform[®] is manufactured by reacting urea with formaldehyde, resulting in urea bound to long chain carbon polymers and requiring microbial breakdown for N release (Nu-Gro, 2004). Therefore the ureaform may provide a slower release of N through time, as well as a labile carbon source for microbial utilization.

Phosphorus was applied in each fertilization treatment plot at a rate of 28 kg-P ha⁻¹ as TSP (Southern States Cooperative, Inc., Richmond, VA), on the same dates as N fertilization. Both N and P fertilizers were broadcast by hand in each fertilization treatment plot so that the fertilizer evenly covered the forest floor throughout the plot.

Tree Growth Increment Response

Every tree in each measurement plot was measured at the time of plot installation in July of 2004, and two growing seasons after treatment in January of 2006. Three measurements were taken on each tree as follows: 1) diameter at breast height (DBH), 2) height to the base of live crown (HBC) and, 3) total height (HT). From these three measurements, basal area per acre (BA), trees per acre (TPA), and length of live crown (LC) were calculated. In addition, a growth (GRW) variable was calculated for each measurement, as the difference between the 2006 and 2003 measurements. All tree measurements and calculated variables were averaged to provide a plot mean for each response variable.

Foliar Nutrient Sampling

Foliage was sampled in each plot on four dates: 1) February 1-7, 2004, 2) January 1-7, 2005, 3) July 1-7, 2005, and 4) January 1-7, 2006. In each plot, five

dominant or co-dominant trees were selected and marked. The same five trees from each plot were sampled throughout the study in order to minimize between tree variability. The terminal end of a lateral branch in the upper 1/3 of the canopy was shot out of each sample tree and 20 intact, disease free fascicles were removed, including the bundle sheaths, and placed in a labeled paper bag. The 20 fascicles from the five trees in each plot were composited to create a plot sample of 100 fascicles. The foliage samples were transported in a cooler to the laboratory where they were dried in a forced air drying oven at 70° C for 10 days. The oven-dried plot samples were then weighed and ground in a Wiley® mini-mill to pass through a 1mm screen.

The ground foliage was analyzed for macro- and micro-nutrient cations according to Jones, et al (1973), by first drying ground samples in a 65° C oven for 24 hours, and subsequently weighing 0.5 gram samples into glass ignition tubes. The foliage was then ashed in a 932° C muffle furnace for 12 hours, allowed to cool, then digested in 6 N HCl solution for 30 minutes. The extracts were filtered through Whatman #42 filter paper and analyzed for P, K, Ca, Ba, Mg, S, B, Fe, Al Mn using an Inductively Coupled Plasma Spectrometer (SPECTRO Analytical Instruments GmbH and Co., Kleve, Germany) at the Virginia Tech University Soil Testing Laboratory. The dried and ground foliage was also analyzed for total Carbon and Nitrogen using a high temperature dry combustion analyzer (VarioMAX CNS analyzer, Elementar Analysensysteme GmbH., Hanau, Germany).

Leaf Litter

In order to examine nutrient cycling and ascertain leaf area index, leaf litter fall was collected monthly from September 1 to February 1, in 2004-2005, which corresponded to the 2003 cohort, and 2005-2006, which corresponded with the 2004 cohort. In each plot, five litter traps were placed in a consistent evenly spaced pattern,

and secured with stakes to the forest floor. The traps were 0.2 m² plastic bins with holes drilled in the bottom to allow for water drainage. The total ground area covered by the traps was 1 m².

The litter from traps was composited by plot in the field and placed in labeled paper bags. The litter was dried in a forced air drying oven at 70° C for 10 days. Litterfall dry weight totals for each plot were obtained by compositing the oven dried foliage from each of the six month collection dates in a cardboard box and weighing the total seasonal litterfall on a digital balance. Using the assumption that loblolly pine drops approximately one half of its foliage during the dormant season, the litterfall dry weight was used in coordination with loblolly pine specific leaf area values derived by Vose and Allen (1988), to estimate total canopy leaf area of the previous growing season.

In addition, from each plot's oven dried seasonal litterfall collection, 100 intact fascicles were randomly sampled, weighed, and ground using the same procedures as described for foliar nutrient content. The ground litterfall was then processed and analyzed in the lab for total micro- and macro-nutrient cations using an ICP spectrometer, as well as for total nitrogen and carbon using high temperature dry combustion.

Canopy Leaf Area Index

Leaf area index (LAI) was determined in each plot using hemispherical canopy photography and image post-processing with Hemi-view software (Delta-T Devices, Ltd., Cambridge, UK). Photographs were taken with a Nikon Cool-pix 950 digital camera using a hemispherical fish-eye lens. The camera was secured to a self-leveling mount (Delta-T Devices, Ltd., Cambridge, UK) and attached to a tripod. In the field, the tripod was placed over the center of each measurement plot and raised so that the fish-eye lens was 1.4 meters above the forest floor. The camera leveling mount was then oriented

towards magnetic North, adjusted so the lens was plumb and level, and one photograph was taken with the camera facing skyward.

The photographs were taken in black and white, with the aperture set to infinity, and without the use of a flash bulb. All pictures were taken when the sky conditions were suitable including dawn, dusk, or when the sky was completely and evenly overcast. The dates of photograph acquisition were: 1) March 23, 2004 ; 2) July 1, 2004 ; 3) August 18, 2004 ; 4) January 14, 2005 ; 5) September 23, 2005 ; and 6) January 4, 2006.

After each set of photographs was acquired, the images were analyzed with the Hemi-view software. Within the software each photograph was registered and a skymap aligned with the North marker on the leveling mount that was captured in each photograph. The radius of the skymap was adjusted to include only trees within each measurement plot, however this radius was held constant across all measurement plot photographs. For analysis, the Default Simple Solar Model was used, with the geographical coordinates and date of photo acquisition properly adjusted for the study site. As found in previous studies (Jonkheere, et al., 2004; Chen, et al., 1997; Coops, et al., 2004) the adjustment of brightness threshold levels within the software was the main determinant in LAI values. These levels were adjusted according to the Delta-T manual (Delta-T Devices, Ltd., Cambridge, UK), and technical support suggestions, so that the faintest leaves in each photo would be classified as “obstructed”, yet small canopy gaps remained classified as “visible”. For photographs taken at the same time during the same day, the threshold values were held constant across all measurement plot photos.

Total KCl Extractable-N and In Situ N Mineralization

Nitrogen mineralization was measured *in situ* in each plot using sequential coring methods similar to Raison, et al. (1987) and Gurlevik, et al. (2004). Cores were made of polyvinyl chloride pipe, 3.8 cm inside diameter, and 20.3 cm in length with one end

beveled to a sharp edge for easy soil penetration and root severing. At random locations within each measurement plot, two cores were inserted adjacent to one another, vertically into the mineral soil, and to a depth of 15 cm. Caps were placed on the cores to prevent precipitation from entering, but loosely enough to allow for gas exchange within each core. One core was immediately removed and transported in a cooler to the laboratory for processing. The second incubation core remained in place for 14 days. After the 14 day incubation period, this core was removed for laboratory processing. Sequential *in situ* incubations were implemented monthly at two random locations within each measurement plot, beginning July 1, 2004, and continuing through October 1, 2005. During the first 4 months after each fertilization date, 14 day incubations occurred sequentially, with the insertion of a new set of incubation cores on the same date that the previous set was removed, therefore two sequential incubations periods occurred within one month. However, following this 4 month period of sequential 14 day incubations, the frequency of sampling was reduced to include only one 14 day incubation per month, followed by a 14 day period of no incubations.

The soil was removed from each core, and sieved with a #10 mesh screen to remove coarse fragments. The sieved soil was then placed in plastic sample bags and stored at 4° C until extraction, which took place within 3 weeks of soil collection. Five grams of field moist soil from each sample bag was dried in a 105°C forced air oven for 24 hours, and subsequently weighed to determine gravimetric water content. A second 5 gram sample of field moist soil was placed in a centrifuge tube with 50 ml of 2 M KCl and shaken on a reciprocating shaker for 1 hour. After shaking, the samples were centrifuged for 10 minutes, filtered through Whatman #42 paper, and the extracts frozen in scintillation vials. All extracts were analyzed colorometrically for nitrate (US EPA Method 353.2) and ammonium (US EPA Method 350.1) using a TRAACS 2000 Auto

Analyzer (SEAL Analytical, Mequon, WI). N concentration data from each initial soil core was subtracted from its respective incubated core data to estimate *in situ* N-mineralization. Data from the initial cores also served as an estimate of total KCl extractable-N as NH_4^+ and NO_3^- .

At each sampling date in the mineralization study, *in situ* soil temperature was measured using a digital thermometer and soil volumetric water content was measured using a digital soil moisture meter (HydroSense®, Campbell Scientific Inc., Ogden, Utah) inserted 15 cm into the mineral soil immediately adjacent to the incubation cores. These soil temperature and moisture values were also supplemented by hourly air temperature, soil temperature, precipitation, and soil moisture measurements from the Soil Climate Analysis Network (SCAN) weather station located less than 0.4 km from the study site, operated by the Natural Resources Conservation Service (Figures 1, 2, and 3).

A comparison of the SCAN weather station mean monthly precipitation during the 1-year sampling period of this study with the monthly mean, maximum, and minimum precipitation over the previous 5 years (NOAA, National Climate Data Center) shows that during the study period there were no anomalous precipitation patterns (Figure 1). The trends in measured *in situ* soil temperature in the upper 15 cm of mineral soil followed closely the trends in SCAN soil temperatures to the same soil depth (Figure 2). However, it is interesting to note that during the summer months, *in situ* soil temperature measurements under a forest canopy were 0-3 C° lower than the SCAN soil temperatures in an open field environment (Figure 2). Lastly, the trends in measured *in situ* volumetric water content (θ) and soil sample gravimetric water content also followed closely the trends in SCAN volumetric water content (Figure 3). The differences in SCAN and *in situ* θ were similar to those of soil temperature, with *in situ* θ being consistently less than SCAN θ , perhaps due to rainfall interception from the forest canopy at the study site

(Figure 3). Changes in soil θ also followed trends in precipitation with increased soil VWC immediately following precipitation events, and marked decreases in soil θ after prolonged periods without precipitation events, most notable from April to June 2005 (Figure 3).

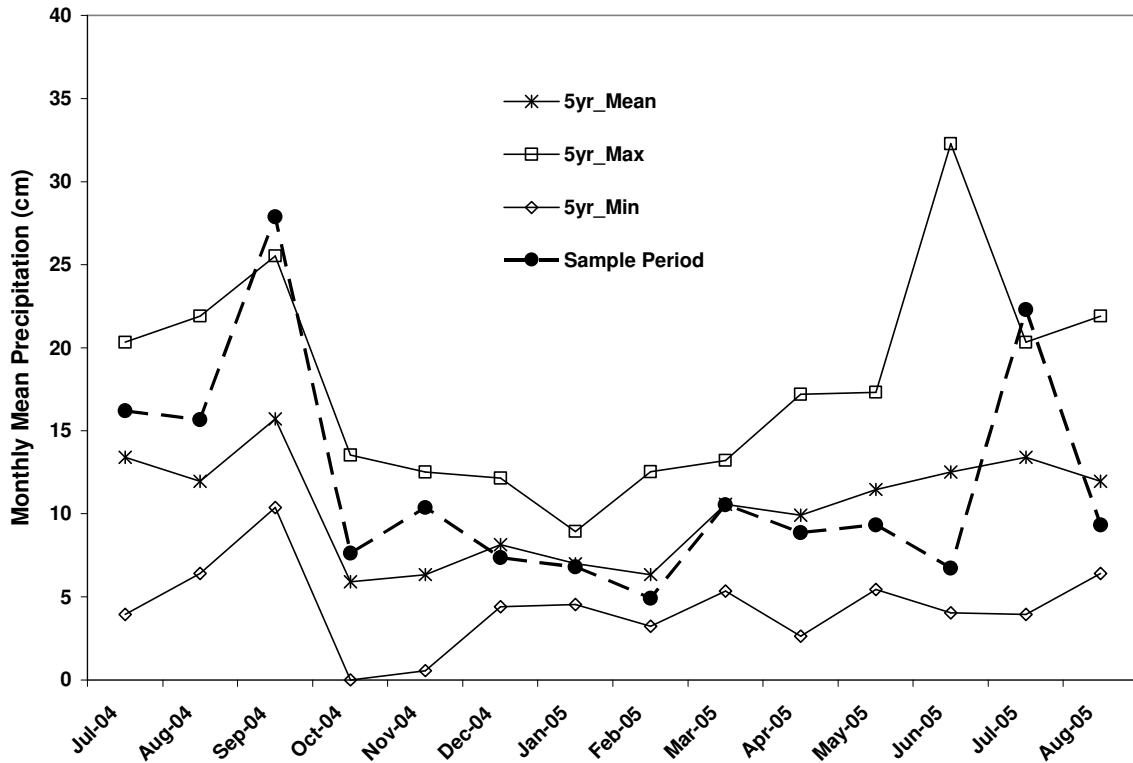


Figure 1. Comparison of 1-year sample period mean monthly precipitation (NRCS Soil Climate Analysis Network) and 5 year Mean, Maximum, and Minimum precipitation (NOAA National Climate Data Center) in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

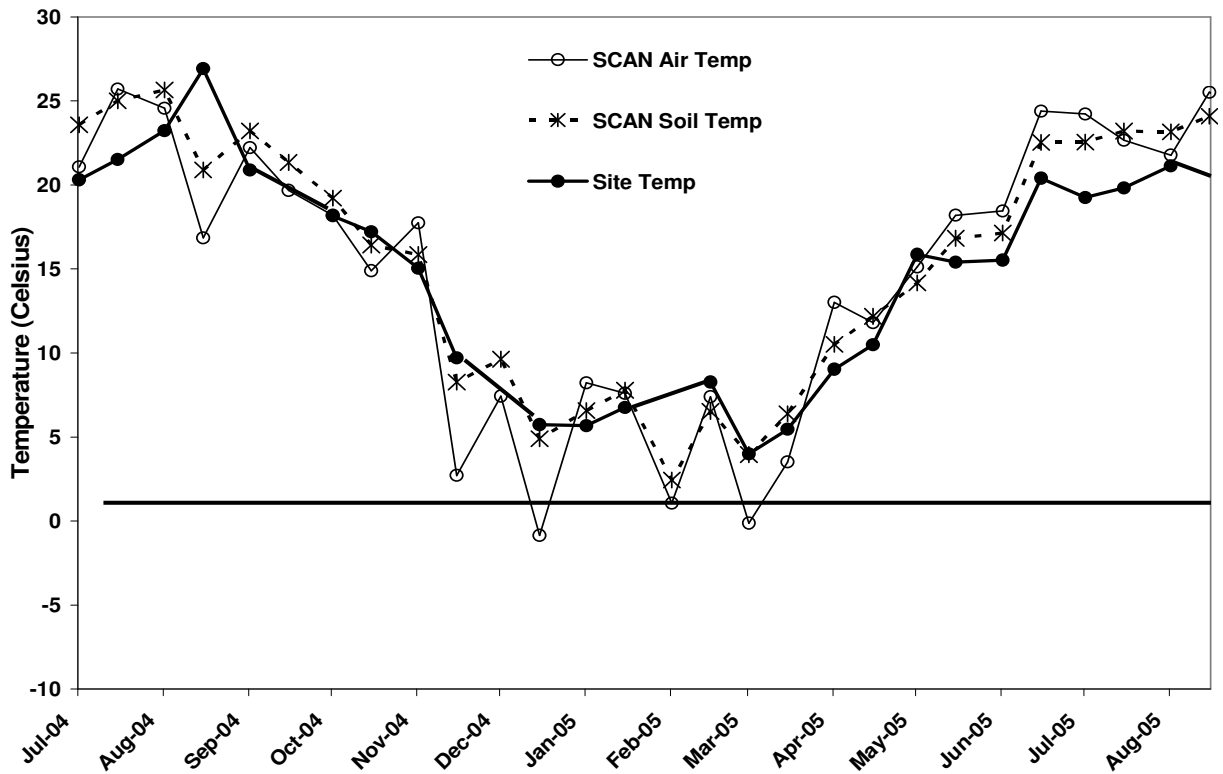


Figure 2. Comparison of 1-year sample period SCAN soil temperature and air temperature with concurrently measured site soil temperature measured in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

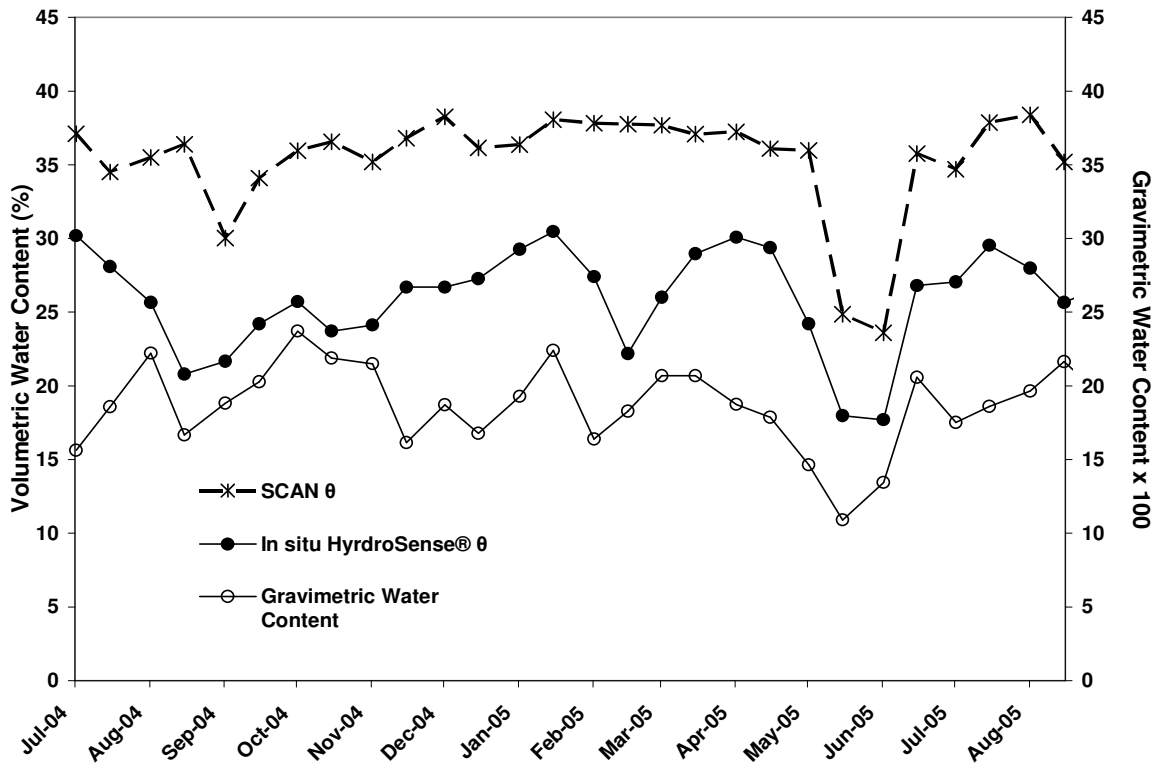


Figure 3. Comparison of 1-year sample period SCAN soil volumetric water content (θ) with concurrently measured *in situ* soil θ measured with Hydrosense® Moisture Meter, and laboratory gravimetric water content from soil collected in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

Ion Exchange Membrane-N

In situ ion exchange membrane-N (IEM-N) was measured in all measurement plots following procedures similar to those in Cooperband and Logan (1994) and Huang, et al., (1996). Cation and anion exchange membrane sheets (Ionics Inc., Watertown, MA), were first cut into 79 cm² squares. This size is equivalent to 6.32 grams of dry resin with a capacity of 1.77 cmol_c anion / cation exchange capacity. Cation and anion membrane squares were kept separate, washed with de-ionized water, and soaked inside plastic carboys containing 1 M NaCl solution for 24 hours before use in the field. Just before insertion into the mineral soil, membranes were removed from the salt solution and washed with de-ionized water. Two sets of membranes were installed at random

locations within each measurement plot during the same sampling period as the *in situ* N-mineralization cores.

Within each set, cation and anion membranes were inserted horizontally below the O horizon and vertically in the A horizon. Membranes were inserted horizontally by cutting a square of O horizon material down to the mineral soil with a serrated stainless steel spatula, carefully lifting the intact O material, and placing the membrane flush against the mineral soil beneath it. Membranes were inserted vertically in the A horizon soil from 3 cm to 12 cm deep by opening a vertical slit with the stainless steel spatula and placing the membrane into the mineral soil at the specified depth. The slit was firmly closed from either side with the spatula to ensure proper contact between membrane and soil. Within each membrane set, the cation and anion membranes were situated adjacent to one another with 7 to 12 cm between each membrane. The location of each membrane was marked with a pin flag and cable-ties for relocation. After a 14-day incubation period, the membranes were carefully removed from their locations, and soil adhering to the membranes was removed. Individual membranes were then sealed in labeled plastic bags and stored at 4° C until extraction, which took place less than 3 weeks from field removal.

At the time of extraction, each membrane was placed in a centrifuge tube with 25 mL of 1 M KCl and shaken on a reciprocating shaker for 1 hour. The membranes were then removed and the extract filtered through Whatman #42 paper and frozen in scintillation vials. All extracts were analyzed colorometrically for nitrate (US EPA Method 353.2) and ammonium (US EPA Method 350.1) using a TRAACS 2000 Auto Analyzer (SEAL Analytical, Mequon, WI).

After extraction, the membrane squares were washed separately with de-ionized water and re-charged with 1 M KCl solution. In order to maintain constant exchange

capacity over the entire sampling period, six sets of membrane squares were used and kept separated, so that no set of membrane squares was incubated, extracted, and recycled more than 4 times.

Nitrogen Volatilization

In order to determine the ammonia volatilization following fertilization, closed static ammonia sorbing systems were installed similar to those described in Nommik (1973) and Mattos, et al. (2003). The systems were constructed using sections of polyvinyl chloride pipe, 30.5 cm in diameter, 35.6 cm in length, and 729.67 cm² in internal cross-sectional area. The chambers were beveled on the bottom end and inserted to a depth of 10 cm into the forest floor.

Disks of polyurethane foam (Hibbco Plastics, Yadkinville, NC), 2 cm in thickness, 30.5 cm in diameter, with a density of 0.03 g cm⁻³, were first cleaned with a 10% HCl solution, thoroughly washed with de-ionized water, and left to air dry. After the initial cleaning, the foam discs were weighed and then placed in sterile plastic bags containing 150 mL of a 2.25 mol L⁻¹ H₂PO₄ plus 4.0% glycerol solution. After soaking, the discs were weighed to determine the volume of acid solution retained in the foam. All foam discs held between 115 mL and 150 mL of the acid solution. The discs were transported to the field in their respective sealed plastic bags and two disks were mounted horizontally in each PVC chamber, with the edges of the foam discs resting on internal mounts placed at 10 cm and 20 cm above the forest floor. A lid was placed on top of each PVC chamber to prevent rainfall and atmospheric-N inputs. The lower disks served to absorb gaseous ammonia volatilized from the soil and forest floor, while the upper disks served to absorb any atmospheric-N inputs. Two volatilization chamber systems were installed in each of the measurement plots and fertilizer was added to the forest floor within the chambers of the fertilized plots at a rate of 448 kg-N ha⁻¹. In order to

ensure the exact rate of fertilizer being applied within each chamber, the chambers were capped with a lid while the entire treatment plot was fertilized by hand broadcasting . After the entire treatment plot was fertilized, the volatilization chamber caps were removed and the fertilizer was added separately at the 448 kg-N ha⁻¹ rate, which was 3.27 grams of N per cylinder. Beginning at the time of fertilization, foam discs were incubated, removed, and replaced with freshly soaked foam after 1, 3, 5, 7, 9, 19, and 29 days. These sampling periods were chosen based on previous studies which demonstrated the majority of volatilization occurs within the first 10 days after fertilization (Nommik, 1973; Mattos, et al, 2003).

The disks, once removed were sealed in their respective plastic bags and stored at 4° C until extracted, which occurred less than 3 weeks after field removal. At the time of extraction, the foam discs were first weighed to obtain the volume of solution remaining in the foam discs after field incubation. The extraction then proceeded as a 3 stage process. First, 100 mL of 1M KCl was added to each bag containing a previously incubated foam disc. The foam, bag, and KCl solution were vigorously shaken for at least 10 minutes, usually resulting in the bulk of KCl solution being absorbed by the foam disc. The foam disc was then removed from the bag and the solution was thoroughly squeezed from the foam into a beaker. Secondly, the disc was placed back into its respective bag and the previous extraction process repeated with a second 100 mL aliquot of 1 M KCl solution. Lastly, after the second foam extraction, and with the foam removed from the bag, 50 mL of 1 M KCl solution was added to the bag, shaken, and poured into the same beaker. After the extraction process, the final volume of extract was brought to 500 mL by adding additional aliquots of 1 M KCl. The foam extract was then filtered through Whatman #2 paper and frozen in 250mL scintillation vials.

Before analysis, 10 mL sub-samples of the foam extract were buffered with 5 mL of 1 M NaOH solution, bringing solution pH to 7 +/- 0.5 pH. This allowed for the samples to be analyzed colorometrically for ammonia (US EPA Method 350.1) using the TRAACS 2000 Auto Analyzer (SEAL Analytical, Mequon, WI).

After the foam was extracted, it was soaked in a 10% HCl solution, washed thoroughly with de-ionized water, and air dried. This allowed for their re-use at the second fertilization date.

Statistical Analysis

The experimental design for the study was a randomized complete block design with four blocks of six treatments, resulting in a total of twenty-four measurement plots. The plots were blocked on pre-treatment basal area and dominant height. The six thinning and fertilization treatments were randomly assigned to the measurement plots according to the following regime: 1) no thinning / no fertilization (Check), 2) thinning only (TH), 3) thinning + 224 kg-N ha⁻¹ as urea + 28 kg-P ha⁻¹ as TSP in summer (URS), 4) thinning + 224 kg-N ha⁻¹ as control release ureaform + 28 kg-P ha⁻¹ as TSP in summer (CRS), 5) thinning + 224 kg-N ha⁻¹ as urea + 28 kg-P ha⁻¹ as TSP in winter (URW), and 6) thinning + 224 kg-N ha⁻¹ as control release ureaform + 28kg-P ha⁻¹ as TSP in winter (CRW). The following four null hypotheses were tested in this study:

HO₁: The effect of thinning only without fertilization on soil N availability will be no different than the control.

HO₂: The soil inorganic N availability after fertilization with controlled release Nitroform® will be no different than the soil inorganic N availability after fertilization with urea, and the effect of the fertilizer treatments on soil inorganic N availability will be no different than the control.

HO₃: Soil N mineralization after fertilization with controlled release Nitroform® will be no different than soil N mineralization after fertilization with urea, and the effect of the fertilizer treatments on soil N mineralization will be no different than the control.

HO₄: Nitrogen volatilization after fertilization with controlled release Nitroform® will be no different than fertilization with urea, and the effect of the fertilizer treatments on N volatilization will be no different than the control.

For each measured soil response to the fertilization treatments, means profiles and standard errors of the means were calculated at each sampling date (Figures 1 – 8). Prior to statistical analysis, outliers were determined with Dixon’s rejection criteria test (Dean and Dixon, 1951; Rouessac and Rouessac, 1989).

Dixon’s rejection criteria test is based on the following calculation:

$$Q_{\text{calculated}} = \frac{|\text{value in question} - \text{its closest neighbor}|}{(\text{largest value} - \text{smallest value})}$$

The $Q_{\text{calculated}}$ value is then compared to tabulated Q_{critical} values at the 95% confidence interval, which are dependent upon the number of measurements in the data set. If $Q_{\text{calculated}}$ is greater than Q_{critical} , then the data point classified as an outlier was removed from the data set. Only one data point was removed from any data set using this process.

The repeated measures statistical analyses were performed using the methods described in Littell, et al. (1998), utilizing the SAS mixed model procedure “proc mixed” in coordination with the “repeated” statement (SAS Institute, Cary, NC). Mixed model repeated measures analysis was used because non-equal covariance was prevalent across the timeline of this study for every dependent variable measured. In addition, this approach is most appropriate for the analysis of repeated measures on the same experimental units through time, as it assumes that correlation exists between the response measurements throughout the sampling period and thus calculates correct

standard error terms during hypothesis testing (Littell et al. 1998; Meredith and Stehman, 1991)

Before the response data was analyzed with the above procedure, the appropriate covariance structure was chosen from three possibilities: 1) the compound symmetry matrix, 2) the unstructured covariance matrix, and 3) the autoregressive covariance matrix (Littell et al., 1998). If the convergence criteria was not met or the appropriate matrices were not found to be positive definite while using a particular covariance structure, that possibility was eliminated (SAS Institute, Cary, NC). A further elimination occurred by comparing the REML log likelihood, Akaike information criteria, and the Schwarz Bayesian criterion parameter outputs for each data set and the covariance structure which yielded the lowest parameter values was chosen for the final analysis (Littell et al., 1998). For the total KCl extractable-N, total N-mineralization, and total IEM-N analyses, the autoregressive covariance structure was determined the most appropriate. For the total N-volatilization analysis, the compound symmetry covariance structure was determined the most appropriate.

Within this mixed model repeated measures approach, main effects treatment contrasts were performed to test the null hypotheses in two steps. First, to investigate treatment response trends across the entire sampling period, main effects treatment contrasts were performed for the average treatment response through time (Littell et al., 1998). Secondly, main effects treatment contrasts were also performed for each treatment response combination at each sampling date. Significance was accepted at the α (0.05) level and the $P > |t|$ values for each main effects treatment contrast are reported for both types of analysis.

RESULTS

Total KCL Extractable-N

KCl extractable-NO₃⁻ levels in the mineral soil were dynamic throughout the study (between 0.1 mg kg-soil⁻¹ and 30 mg kg-soil⁻¹). However, the overall trends in total KCl extractable-N reflected the result of the changes in available NH₄⁺ in the mineral soil. Therefore, it was not necessary to individually show the NH₄⁺ and NO₃⁻ results. Rather, the total KCl extractable-N data, computed as the sum of NO₃⁻ and NH₄⁺ was presented because it most clearly displayed the treatment effects. (The results for mineral soil NO₃⁻ and NH₄⁺ concentrations are presented graphically as means profiles with accompanying standard error bars in Appendix A)

Total KCl extractable-N varied between 5 and 15 mg kg-soil⁻¹ during the entire study period in the Check and TH plots (Figure 4). There were no significant differences in total KCl extractable-N between the Check and TH treatments for the analysis of average response over the entire sampling period. However, there were two dates when Check and TH were significantly different (Table 1).

Table 1. Pr>|t| values for treatment contrasts of total KCl Extractable-N means averaged over a 1-year sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

| Comparison | Estimate | Error | DF | t Value | Pr > t |
|------------|----------|--------|----|---------|---------|
| Check-TH | -0.4321 | 1.1646 | 6 | -0.37 | 0.7234 |
| URS-CRS | -7.149 | 2.4461 | 6 | -2.92 | 0.0265 |
| URS-TH | 5.5348 | 1.4286 | 6 | 3.87 | 0.0082 |
| CRS-TH | 12.6831 | 2.1993 | 6 | 5.77 | 0.0012 |
| URW-CRW | 12.3845 | 3.933 | 6 | 3.15 | 0.0198 |
| URW-TH | -17.5867 | 3.813 | 6 | -4.61 | 0.0036 |
| CRW-TH | -5.2022 | 1.2827 | 6 | -4.06 | 0.0067 |

Table 2. Pr>|t| values for treatment contrasts of total KCl Extractable-N means at each sampling date for a 1-year sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

| Comparison | Check-TH | URS-CRS | URS-TH | CRS-TH | URW-CRW | URW-TH | CRW-TH |
|------------|----------|---------|--------|--------|---------|--------|--------|
| 1-Jul | 0.600 | 0.883 | 0.318 | 0.509 | 0.528 | 0.593 | 0.755 |
| 1-Aug | 0.303 | 0.483 | 0.68 | 0.579 | | | |
| 15-Aug | 0.17 | 0.613 | 0.566 | 0.793 | | | |
| 1-Sep | 0.914 | 0.034 | 0.061 | 0.001 | | | |
| 15-Sep | 0.968 | 0.045 | 0.077 | 0.002 | | | |
| 1-Oct | 0.209 | 0.095 | 0.019 | 0.003 | | | |
| 15-Oct | 0.265 | 0.101 | 0.001 | 0.001 | | | |
| 1-Nov | 0.89 | 0.008 | 0.709 | 0.002 | | | |
| 15-Nov | 0.861 | 0.018 | 0.708 | 0.006 | | | |
| 15-Dec | 0.809 | 0.233 | 0.786 | 0.153 | 0.818 | 0.879 | 0.423 |
| 1-Jan | 0.947 | 0.491 | 0.617 | 0.319 | 0.986 | 0.972 | 0.976 |
| 15-Jan | 0.886 | 0.676 | 0.098 | 0.201 | 0.996 | 0.983 | 0.956 |
| 1-Feb | 0.083 | 0.035 | 0.001 | 0 | 0.381 | 0.456 | 0.671 |
| 15-Feb | 0.044 | 0.245 | 0 | 0.004 | 0.408 | 0.506 | 0.631 |
| 1-Mar | 0.191 | 0.26 | 0.001 | 0.007 | 0.747 | 0.537 | 0.599 |
| 15-Mar | 0.287 | 0.122 | 0.006 | 0.003 | 0.82 | 0.572 | 0.53 |
| 1-Apr | 0.003 | 0.837 | 0.404 | 0.522 | 0.472 | 0.465 | 0.91 |
| 15-Apr | 0.686 | 0.665 | 0.377 | 0.977 | 0.002 | <.0001 | <.0001 |
| 1-May | 0.568 | 0.445 | 0.034 | 0.81 | <.0001 | <.0001 | 0.323 |
| 1-Jun | 0.512 | 0.662 | 0.065 | 0.651 | <.0001 | <.0001 | 0.131 |
| 1-Jul | 0.623 | 0.971 | 0.533 | 0.725 | 0.119 | 0.022 | 0.222 |
| 1-Aug | 0.58 | 0.917 | 0.37 | 0.574 | 0.305 | 0.031 | 0.037 |

Summer Fertilization

Fertilization with urea and controlled release Nitroform® increased total KCl extractable-N with peak values reaching 25 mg kg-soil⁻¹ and 45 mg kg-soil⁻¹ respectively (Figure 4). Both URS and CRS were found to be significantly greater than TH for the analysis of average response over the entire sampling period, indicating greater average N-availability after fertilization for one year (Table 1). However, the analysis of response at each sampling date reveals that the URS treatment was significantly different than TH at five sampling dates after fertilization (Table 2). At these significant dates, URS total KCl extractable-N concentrations in the mineral soil were 1.5 to 2 times greater than TH. The CRS treatment was significantly different than TH at ten sampling dates after

fertilization (Table 2). These significant dates included six consecutive sampling dates from September 1 through November 15, 2004, as well as four consecutive sampling dates from February 1 through March 15, 2005. At these significant dates, CRS total KCl extractable-N concentrations in the mineral soil ranged from 3 to 4 times greater than TH. Main effects contrasts between URS and CRS treatment responses were found to be highly significant for the analysis of average response over the entire sampling period ($P=0.027$)(Table 1), with greater total KCl extractable-N occurring in the CRS treatment as compared to the URS treatment. The analysis of responses at each sampling date reveals that CRS total KCl extractable-N was significantly greater than URS on November 1 and November 15, 2004, as well as February 1, 2005. At these three dates, CRS was 1.5 to 2 times greater than URS. There were four consecutive sampling dates immediately following fertilization from September 1 to October 15, 2004 where CRS was notably greater than URS, however significance was at the $\alpha < 0.1$ level.

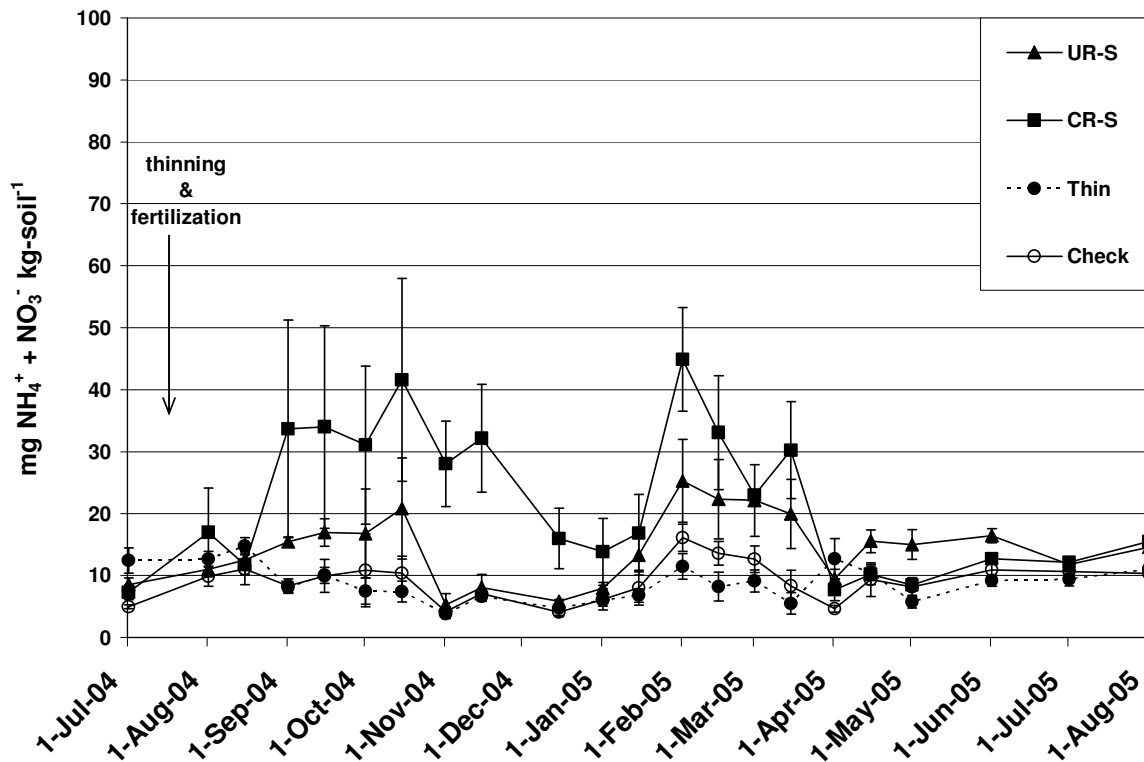


Figure 4. Total KCl extractable-N from the surface 15 cm of the mineral soil in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha⁻¹.

Winter Fertilization

Fertilization with urea and controlled release Nitroform® in the winter also increased total KCl extractable-N (Figure 5). Total KCl extractable-N reached a peak of 38 mg kg-soil⁻¹ in the CRW treatment and a peak of 82 mg kg-soil⁻¹ in the URW treatment following fertilization. Both URW and CRW were found to be highly significantly different than TH for the analysis of average response over the entire sampling period, indicating greater average N-availability after fertilization for eight months (Table 1). The analysis of response at each sampling date reveals that the URW treatment was significantly different than TH at five consecutive sampling dates in the growing season from April 15 through August 1, 2005 (Table 2). At these significant dates, URW total KCl extractable-N concentrations in the mineral soil were 7 to 9 times

greater than TH. The CRW treatment was significantly different than TH at only two sampling dates, April 15 and August 1, 2005 (Table 2). At these significant dates, CRW total KCl extractable-N concentrations in the mineral soil were 2 to 3.5 times greater than TH. Main effects contrasts between URW and CRW treatment response over the entire sampling period were found to be highly significant, indicating greater average N-availability in URW over eight months following fertilization (Table 1). The analysis of response at each sampling date reveals that URW total KCl extractable-N was significantly greater than CRW on April 15 and May 1. At these three dates, URW was 3 to 6 times greater than CRW. On July 1, 2005, URW KCL extractable-N response was 2 times greater than CRW, though not significant ($P=0.119$).

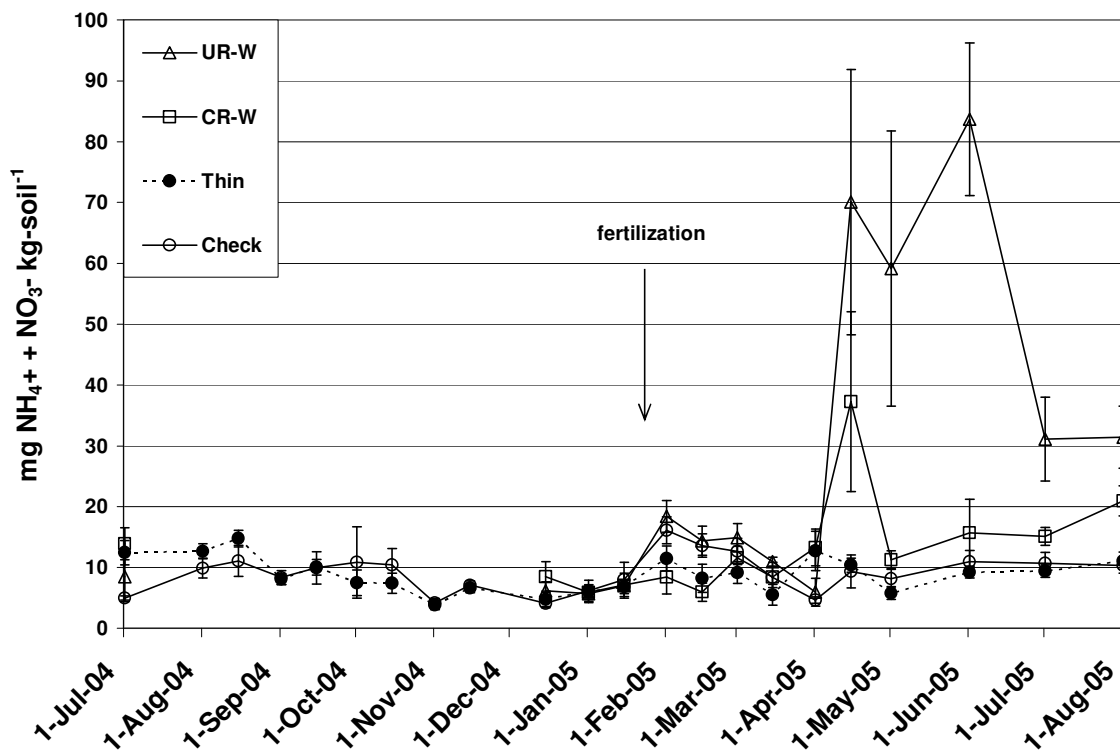


Figure 5. Total KCl extractable-N from the surface 15 cm of the mineral soil in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha⁻¹.

Total Ion Exchange Membrane-N

The methods utilized in this study to measure ion exchange membrane Nitrogen (IEM-N) response to fertilization, allowed for the analysis of NO_3^- and NH_4^+ , in both the O horizon and the A-horizon. IEM- NO_3^- concentrations in both the O horizon and A horizon were low throughout the study ($\leq 1 \text{ mg NO}_3^- \text{ m}^2 \text{ day}^{-1}$), therefore, the IEM- NO_3^- was summed with IEM- NH_4^+ , for an IEM-N result for both horizons. In addition, trends in IEM-N were also similar in the O horizon and the A horizon, and therefore their resultant sum as total IEM-N was used for repeated measures analysis, as it most clearly displayed the overall response after fertilization. (The results for O-Horizon NO_3^- and NH_4^+ , and A horizon NO_3^- and NH_4^+ concentrations are presented graphically as means profiles with accompanying standard error bars in Appendix C.

Throughout the study period, total IEM-N in the Check and TH treatments were less than $2 \text{ mg-N m}^2 \text{ day}^{-1}$ (Figure 6), and there were no significant differences between these treatments for either the analysis of average response over the entire sampling period or the analysis of response at individual sampling dates (Tables 3 and 4).

Table 3. Pr>|t| values for treatment contrasts of total IEM-N means averaged over a 1-year sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha^{-1} .

| Label | Estimate | Error | DF | t Value | Pr > t |
|------------|----------|--------|----|---------|---------|
| Check-Thin | 0.1153 | 0.2531 | 6 | 0.46 | 0.6647 |
| URS-CRS | -1.7634 | 0.6478 | 6 | -2.72 | 0.0345 |
| URS-Thin | 2.3512 | 0.3816 | 6 | 6.16 | 0.0008 |
| CRS-Thin | 4.1145 | 0.5567 | 6 | 7.39 | 0.0003 |
| URW-CRW | 1.2718 | 0.8565 | 6 | 1.48 | 0.1881 |
| URW-Thin | -6.9298 | 0.7876 | 6 | -8.8 | 0.0001 |
| CRW-Thin | -5.4298 | 0.6672 | 6 | -8.14 | 0.0002 |

Table 4. Pr>|t| values for treatment contrasts of total IEM-N means at each sampling date for a 1-year sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

| Comparison | Check-TH | URS-CRS | URS-TH | CRS-TH | URW-CRW | URW-TH | CRW-TH |
|------------|----------|---------|--------|--------|---------|--------|--------|
| 1-Jul | 0.612 | 0.852 | 0.895 | 0.894 | 0.824 | 0.853 | 0.906 |
| 1-Aug | 0.835 | 0.031 | <.0001 | 0.249 | | | |
| 15-Aug | 0.515 | 0.771 | <.0001 | <.0001 | | | |
| 1-Sep | 0.616 | 0.636 | <.0001 | 0.000 | | | |
| 15-Sep | 0.494 | 0.000 | 0.004 | <.0001 | | | |
| 1-Oct | 0.181 | 0.002 | 0.991 | 0.000 | | | |
| 15-Oct | 0.154 | <.0001 | 0.960 | <.0001 | | | |
| 15-Nov | 0.470 | 0.583 | 0.340 | 0.222 | 0.950 | 0.932 | 0.987 |
| 15-Dec | 0.584 | 0.320 | 0.404 | 0.097 | 0.994 | 0.989 | 0.973 |
| 15-Jan | 0.596 | 0.622 | 0.506 | 0.327 | 0.956 | 0.929 | 0.971 |
| 1-Feb | 0.770 | 0.442 | 0.941 | 0.351 | 0.960 | 0.932 | 0.968 |
| 15-Feb | 0.493 | 0.674 | 0.806 | 0.739 | 0.866 | 0.933 | 0.668 |
| 1-Mar | 0.339 | 0.489 | 0.659 | 0.284 | 0.166 | <.0001 | <.0001 |
| 15-Mar | 0.451 | 0.616 | 0.455 | 0.298 | 0.890 | <.0001 | <.0001 |
| 1-Apr | 0.573 | 0.502 | 0.645 | 0.290 | 0.093 | <.0001 | 0.000 |
| 15-Apr | 0.408 | 0.601 | 0.255 | 0.192 | 0.086 | <.0001 | 0.001 |
| 1-May | 0.125 | 0.999 | 0.971 | 0.983 | <.0001 | <.0001 | 0.701 |
| 1-Jun | 0.536 | 0.684 | 0.001 | 0.097 | 0.983 | 0.003 | <.0001 |
| 1-Jul | 0.998 | 0.742 | 0.000 | 0.054 | 0.797 | 0.001 | <.0001 |
| 1-Aug | 0.651 | 0.735 | 0.014 | 0.253 | 0.391 | 0.301 | 0.003 |

Summer Fertilization

Summer fertilization with urea (URS) and controlled release Nitroform® (CRS) significantly increased total IEM-N compared to the thin only (TH) treatment (Table 3). Total IEM-N concentrations were elevated from August to November in the CRS treatment, reaching a peak of 14 mg-N m² day⁻¹ (Figure 6). Total IEM-N concentrations peaked quickly in the URS treatment at 12 mg-N m² day⁻¹ in August and then declined rapidly. Differences between URS and TH total IEM-N responses were found to be highly significant with respect to average response over the entire sampling period (Table 3). However, the analysis of total IEM-N response at each sampling date reveals that the URS treatment was significantly different than TH at four consecutive sampling dates immediately following fertilization, from August 1 to September 15, 2004 (Table 4). At

these significant dates, URS total IEM-N concentrations were 8 to 11 times greater than TH. There were also three sampling dates at the end of the study, from June 1 to August 1, 2005 where URS was significantly greater than TH. Differences between CRS and TH total IEM-N responses were also found to be highly significant for the analysis of average response over the entire sampling period (Table 3). The analysis of IEM-N response at each sampling date reveals that the CRS treatment was significantly different than TH at five sampling dates immediately after fertilization (Table 4). These significant dates included 2.5 months from August 15 to November 1, 2004. At these significant dates, CRS total IEM-N concentrations were 12 to 14 times greater than TH. In addition to this period immediately following fertilization, there were three sampling dates of December 15, 2004, June 1 and July 1, 2005, where CRS total IEM-N concentrations were greater than TH, however only significant at the $\alpha < 0.10$ level (Table 4). Main effects contrasts of average total IEM-N response over the entire sampling period revealed URS and CRS were significantly different over the one year sampling period ($P=0.035$), indicating greater average total IEM-N availability CRS than URS (Table 3). Analysis of response at each sampling date reveals significant differences between CRS and URS at four sampling dates. These differences occurred over a 1.5 month period, from September 15 to November 1, where CRS total IEM-N was 3 to 7 times greater than URS. At only one date, August 1, 2004, URS total IEM-N was 2.5 times greater than CRS.

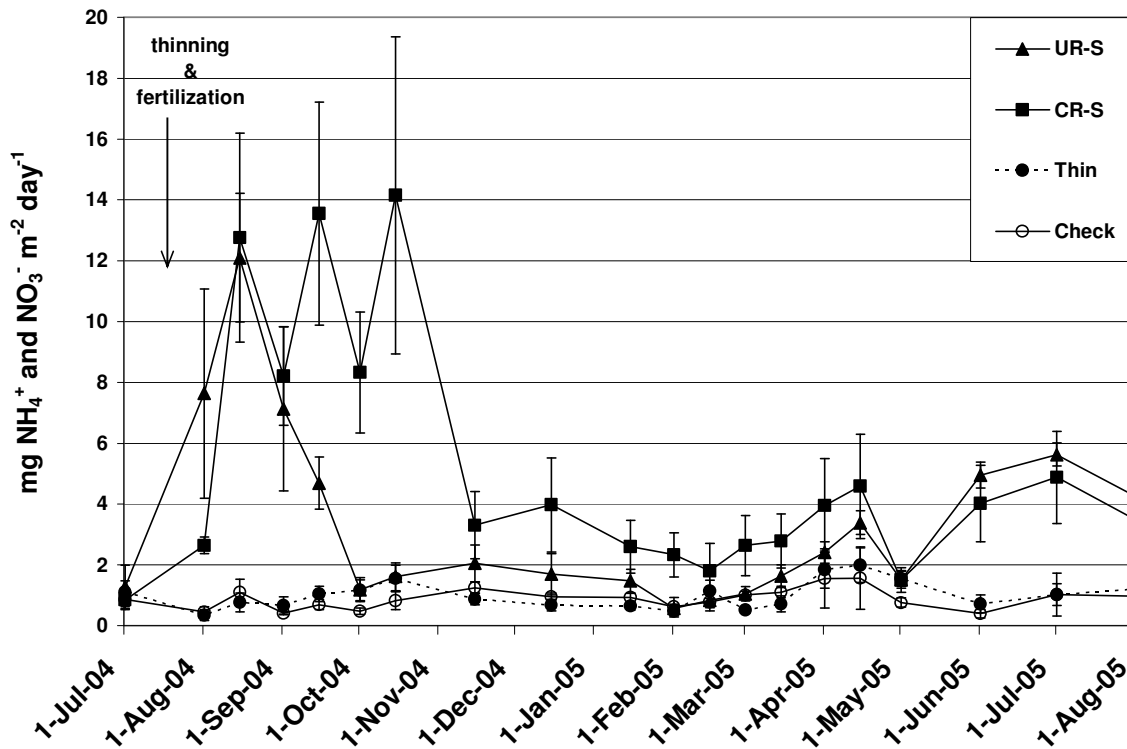


Figure 6. Total ion exchange membrane Nitrogen (total IEM-N) from the surface 15 cm of the mineral soil and O horizon in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

Winter Fertilization

Winter fertilization with both urea (URW) and controlled release Nitroform® (CRW) also significantly increased total IEM-N relative to TH (Figure 7). With respect to average total IEM-N response over the entire sampling period, P-values were <0.01 for both fertilization treatments compared to TH (Table 3). Total IEM-N in URW was found to be significantly greater than TH on seven consecutive sampling dates from March 1 to July 15, 2005 (Table 4). Across these seven dates, URW total IEM-N concentrations ranged from 8 to 17 times greater than TH (Figure 7). There were also significant differences between CRW and TH over seven sampling dates, from March 1 to May 1, 2005 and from June 1 to August 15, 2005. At these sampling dates CRW ranged from 6 to 15 times greater than TH. The May 1 to May 15 sampling period resulted in an

anomalous measurement of CRW total IEM-N which was no different than TH.

Interestingly, main effects contrast for the average total IEM-N response over the entire sampling period revealed no significant differences between URW and CRW treatments ($P > |t| = 0.1881$) (Table 3). However, on two consecutive sampling dates from April 1 to May 1, 2005, URW was 1.6 and 1.5 times greater than CRW respectively, though at the $\alpha < 0.10$ significance level (Table 4).

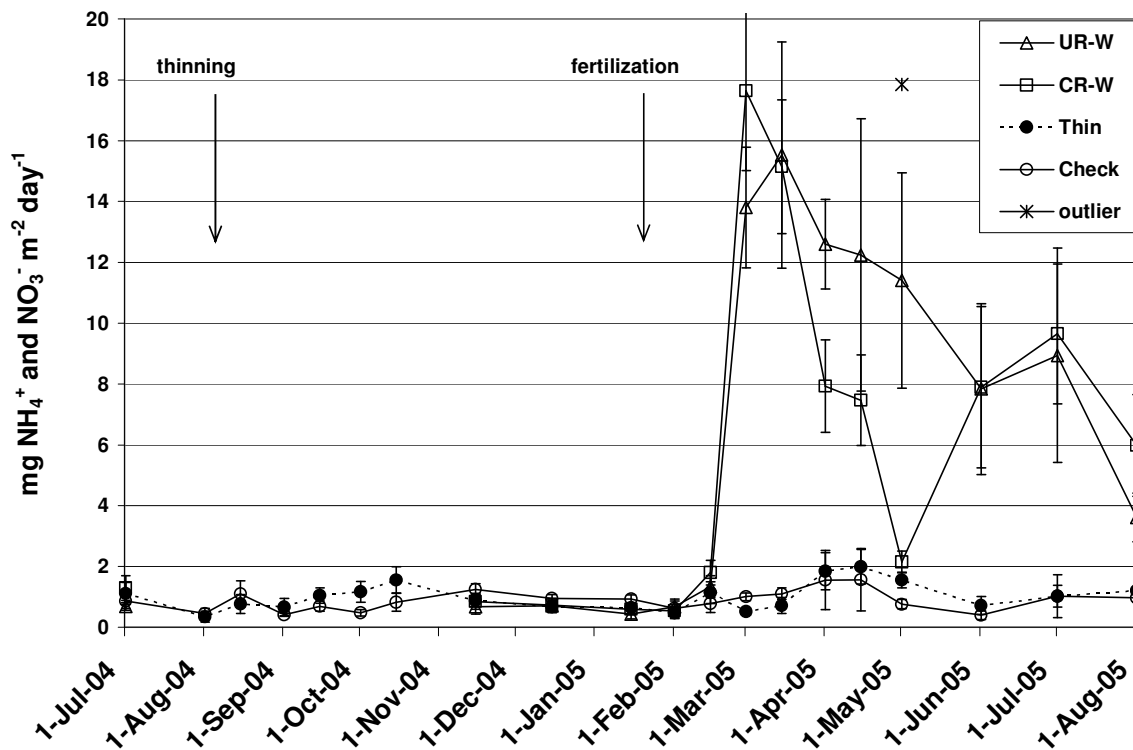


Figure 7. Total ion exchange membrane Nitrogen (total IEM-N) from the surface 15 cm of the mineral soil and O horizon in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

Nitrogen Mineralization

In situ N-mineralization (Nmin) rates in the Check and thin only (TH) treatments were low throughout the study period, ranging from 0.4 to 0.2 mg N day⁻¹ (Figure 8). No significant differences were found in the Nmin response between the thin-only and check

treatments for either the analysis of average response over the entire sampling period or the analysis of response at individual sampling dates (Table 5).

Table 5. Pr>|t| values for treatment contrasts of N mineralization means averaged over a 1-year sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

| Comparison | Estimate | Error | DF | t Value | Pr > t |
|------------|----------|---------|----|---------|---------|
| Check-TH | 0.06798 | 0.08102 | 6 | 0.84 | 0.4336 |
| URS-CRS | 0.1218 | 0.1896 | 6 | 0.64 | 0.5443 |
| URS-TH | -0.1244 | 0.1218 | 6 | -1.02 | 0.3465 |
| CRS-TH | -0.246 | 0.1587 | 6 | -1.55 | 0.1721 |
| URW-CRW | -0.01968 | 0.1403 | 6 | -0.14 | 0.893 |
| URW-TH | 0.1279 | 0.134 | 6 | 0.95 | 0.3765 |
| CRW-TH | 0.1113 | 0.09573 | 6 | 1.16 | 0.2893 |

Table 6. Pr>|t| values for treatment contrasts of N mineralization means at each sampling date for a 1-year sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

| Comparison | Check-TH | URS-CRS | URS-TH | CRS-TH | URW-CRW | URW-TH | CRW-TH |
|------------|----------|---------|--------|--------|---------|--------|--------|
| 1-Jul | 0.767 | 0.241 | 0.306 | 0.634 | 0.525 | 0.676 | 0.713 |
| 1-Aug | 0.658 | 0.858 | 0.591 | 0.935 | | | |
| 15-Aug | 0.818 | 0.586 | 0.662 | 0.540 | | | |
| 1-Sep | 0.063 | 0.733 | 0.501 | 0.108 | | | |
| 15-Sep | 0.095 | 0.396 | 0.716 | 0.028 | | | |
| 1-Oct | 0.809 | 0.262 | 0.548 | 0.450 | | | |
| 15-Oct | 0.624 | 0.277 | 0.931 | 0.528 | | | |
| 1-Nov | 0.735 | 0.926 | 0.983 | 0.710 | | | |
| 15-Nov | 0.564 | 0.928 | 0.885 | 0.584 | | | |
| 15-Dec | 0.879 | 0.807 | 0.824 | 0.998 | 0.548 | 0.963 | 0.454 |
| 1-Jan | 0.872 | 0.929 | 0.961 | 0.884 | 0.551 | 0.940 | 0.378 |
| 15-Jan | 0.428 | 0.914 | 0.493 | 0.714 | 0.727 | 0.975 | 0.616 |
| 1-Feb | 0.001 | 0.994 | 0.072 | <.0001 | 0.372 | 0.421 | 0.789 |
| 15-Feb | 0.202 | 0.693 | 0.085 | 0.007 | 0.324 | 0.595 | 0.478 |
| 1-Mar | 0.114 | 0.908 | 0.139 | 0.004 | 0.617 | 0.378 | 0.729 |
| 15-Mar | 0.044 | 0.734 | 0.046 | 0.000 | 0.473 | 0.457 | 0.917 |
| 1-Apr | 0.847 | 0.077 | 0.176 | 0.390 | 0.388 | 0.319 | 0.996 |
| 15-Apr | 0.799 | 0.116 | 0.709 | 0.996 | <.0001 | 0.000 | 0.037 |
| 1-May | 0.688 | 0.076 | 0.075 | 0.336 | 0.431 | 0.432 | 0.820 |
| 1-Jun | 0.980 | 0.422 | 0.446 | 0.563 | 0.005 | <.0001 | 0.175 |
| 1-Jul | 0.762 | 0.313 | 0.853 | 0.620 | 0.454 | 0.858 | 0.229 |
| 1-Aug | 0.633 | 0.206 | 0.918 | 0.641 | 0.538 | 0.773 | 0.250 |

Summer Fertilization

After summer fertilization, no significant difference was found for the average N_{min} response over the entire sampling period between either urea (URS) and thin only (TH) treatments, or controlled release Nitroform® (CRS) and TH (Table 5). During only one incubation period from March 1 to March 15, 2005, URS was significantly different than TH, where URS exhibited N-immobilization of $-0.5 \text{ mg N day}^{-1}$, while TH demonstrated N-mineralization of $0.5 \text{ mg N day}^{-1}$ (Figure 8 and Table 6). In contrast, CRS was found to be significantly different than TH on five sample dates (Table 6). Immediately after summer fertilization, during the September 15 to October 1, 2004 incubation, mean CRS N_{min} was 9 times greater than TH, indicating much higher N-mineralization (Figure 8). During the incubation period from September 1 to September 15, 2004, mean CRS N_{min} was 8 times greater than TH however not significant ($P=0.108$). Interestingly, during four consecutive incubation periods from January 15 – March 15, 2005, CRS demonstrated significant N-immobilization. During this period, TH N-mineralization ranged from 0.25 to $0.4 \text{ mg N day}^{-1}$, while CRS N-immobilization ranged from -2.4 to $-1.2 \text{ mg N day}^{-1}$ (Figure 8). Main effects contrasts between the URS and CRS treatments revealed no significant differences in either average N_{min} response over the entire sampling period (Table 5) or analysis at each sampling date (Table 6).

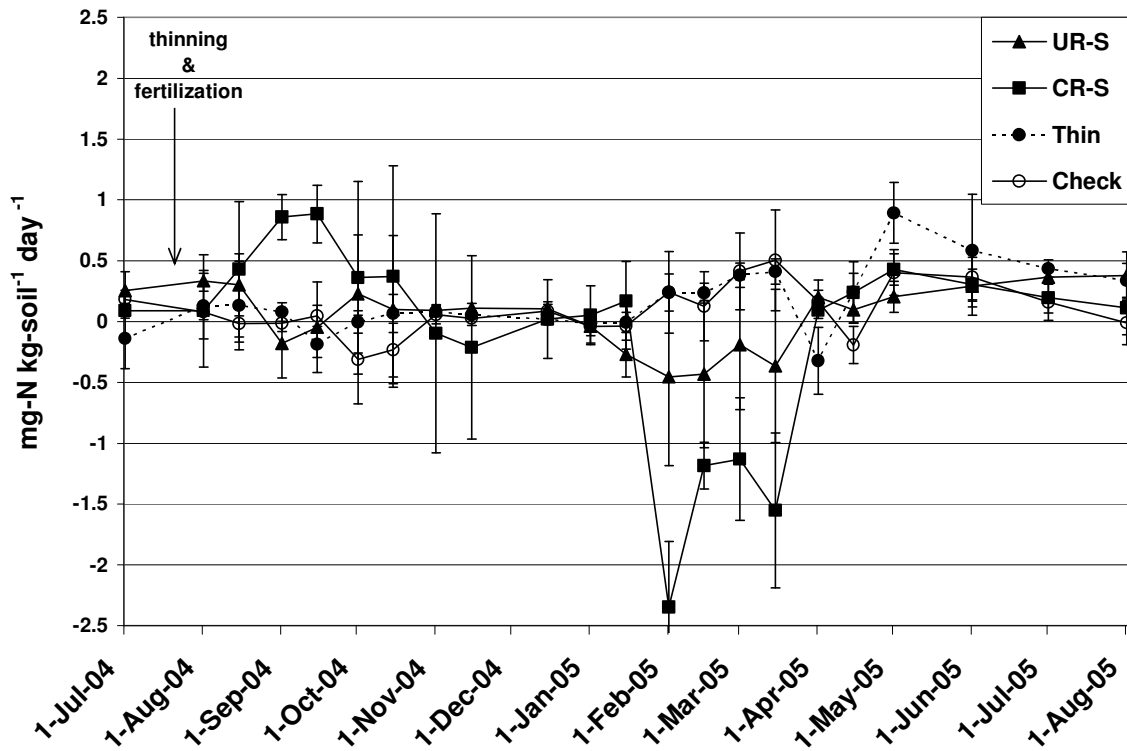


Figure 8. N mineralization (Nmin) from the surface 15 cm of the mineral soil in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha⁻¹.

Winter Fertilization

After winter fertilization, no significant difference was found for the average Nmin response over the entire sampling period either between URW and TH treatments, or CRW and TH (Table 5). There were two individual incubation periods where differences between URW and TH showed significance (Table 6). During the April 1 to April 15, 2005 sampling period, URW Nmin was 8 times greater than TH. In contrast, during the May 15 to June 1, 2005 incubation period, URW demonstrated an N-mineralization rate of -1.5 mg N day⁻¹, indicating N-immobilization while TH continued to mineralize N at rate of 0.6 mg N day⁻¹ (Figure 9). In contrast, there were no individual sampling periods where CRW and TH demonstrated significant differences (Table 6). Main effects contrasts between the two fertilizer treatments showed no significant

difference between the URW and CRW average Nmin response over the entire sampling period (Table 5). During two individual incubation periods, URW and CRW were significantly different (Table 6). From April 15 to May 1, 2005, URW Nmin was 2 mg N day⁻¹ while CRW demonstrated N-immobilization of 0.5 mg N day⁻¹. This scenario changed during the June 1 to June 15, 2005 incubation period with CRW demonstrating N-mineralization of 0.1 mg N day⁻¹ and URW demonstrating N-immobilization of 1.5 mg N day⁻¹ (Figure 9).

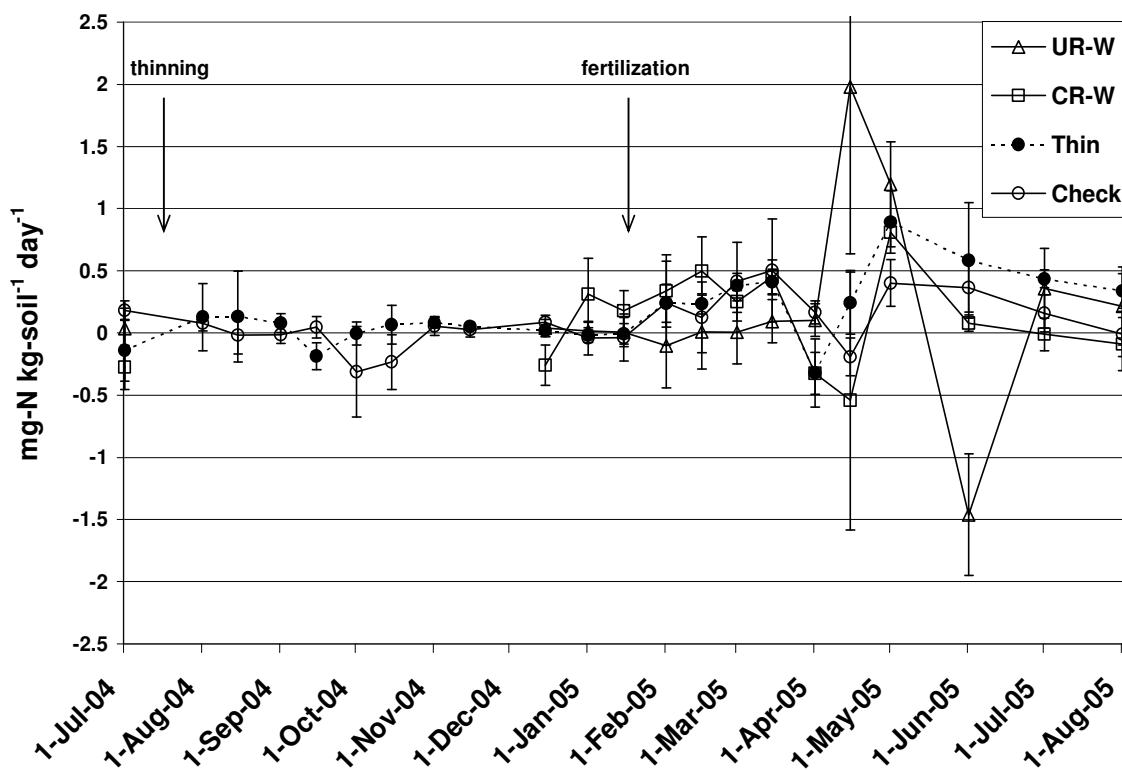


Figure 9. N mineralization (Nmin) from the surface 15 cm of the mineral soil in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha⁻¹.

Nitrogen Volatilization

No significant differences were found in the N-volatilization response between the Check and thin only (TH) treatments for either the analysis of average response over the entire sampling period or the analysis of response at individual sampling dates (Table

7 and 8). The data for these two non fertilized treatments were identical in that after blank samples were analyzed and their values subtracted from the Check and TH concentrations, there was no ammonia trapped in the chambers as a result of the treatments.

Table 7. Pr>|t| values for treatment contrasts of N volatilization means averaged over a 28 day sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 448 kg-N ha⁻¹.

| Contrast | Estimate | Error | DF | t Value | Pr > t |
|----------|----------|---------|----|---------|---------|
| Check-TH | -0.1716 | 0.1188 | 6 | -1.44 | 0.1987 |
| URS-CRS | 204.47 | 16.9027 | 6 | 12.1 | <.0001 |
| URS-TH | 232.46 | 16.6749 | 6 | 13.94 | <.0001 |
| CRS-TH | 27.9896 | 2.7663 | 6 | 10.12 | <.0001 |
| URW-CRW | 30.6133 | 7.25 | 6 | 4.22 | 0.0055 |
| URW-TH | -87.6438 | 6.7563 | 6 | -12.97 | <.0001 |
| CRW-TH | -57.0305 | 2.5894 | 6 | -22.02 | <.0001 |

Table 8. Pr>|t| values for treatment contrasts of N volatilization means at each sampling date over a 28 day sampling period in a mid-rotation loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 448 kg-N ha⁻¹.

| Contrast | 1 | 3 | 5 | 7 | 9 | 19 | 29 |
|----------|--------|--------|--------|--------|--------|--------|--------|
| Check-TH | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| URS-CRS | 0.9231 | <.0001 | <.0001 | <.0001 | 0.5736 | 0.8244 | 0.7897 |
| URS-TH | 0.9195 | <.0001 | <.0001 | <.0001 | 0.4484 | 0.7429 | 0.7209 |
| CRS-TH | 0.9832 | <.0001 | <.0001 | 0.0012 | 0.2273 | 0.5077 | 0.5757 |
| URW-CRW | 0.853 | 0.0097 | 0.0012 | 0.7837 | 0.0008 | 0.3528 | 0.6592 |
| URW-TH | 0.8809 | <.0001 | <.0001 | 0.0011 | <.0001 | 0.0058 | 0.7514 |
| CRW-TH | 0.6822 | <.0001 | <.0001 | 0.0034 | <.0001 | 0.0001 | 0.2136 |

Summer Fertilization

After summer fertilization, the main effects contrasts of the 30-day N volatilization showed significant differences between treatments with URS > CRS > TH (Table 7). Similarly, the main effects contrasts at each sampling date also show significant differences on days 3, 5, and 7 after fertilization with URS > CRS > TH (Table 8). On these days URS volatilized 680, 605, and 308 mg-N respectively, CRS volatilized 95, 40, and 35 mg-N respectively, and TH volatilized no ammonia throughout

the sampling period (Figure 10). At these three dates, URS N-volatilization was 7 times greater than CRS on day 3, 14 times greater than CRS on day 5, and 8 times greater than CRS on day 7. From days 9 through 29, no significant differences in N-volatilization were found between the two fertilizer treatments. In the URS treatment, the data show that 41% of the total nitrogen volatilized was trapped after 3 days, 78% after 5 days, 96% after 7 days, while only 4% of the total nitrogen volatilized was trapped between days 9 and 29 (Figure 10). CRS showed a similar trend with 55% of the total CRS nitrogen volatilized being trapped after 3 days, 79% after 5 days, 98% after 7 days, and 2% between days 9 and 29 (Figure 11). The analysis of cumulative N-volatilization as a percent of applied-N, illustrates that 20% of the URS Nitrogen applied was volatilized after 3 days, 38%, after 5 days, 48% after 7 days, and 51% of total URS nitrogen applied volatilized by 29 (Figure 11). In contrast, cumulative CRS N-volatilization as percent of applied-N showed 3% volatilized after 3 days, 4% after 5 days, 5% after 7 days, and only 6% of total CRS nitrogen was volatilized by day 29 (Figure 11).

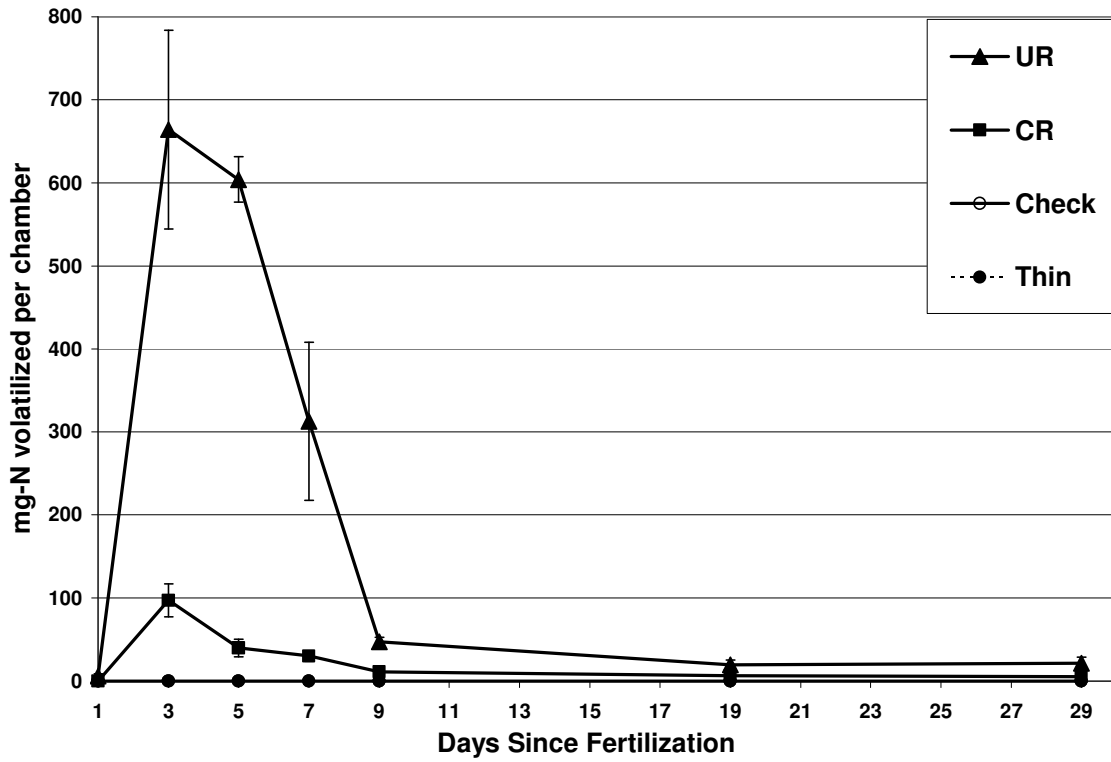


Figure 10. Absolute N volatilization from the mineral soil and forest floor in a mid-rotation loblolly pine plantation in the Virginia Piedmont following fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 448 kg-N ha⁻¹, reported as milligrams of N volatilized within closed-static chamber.

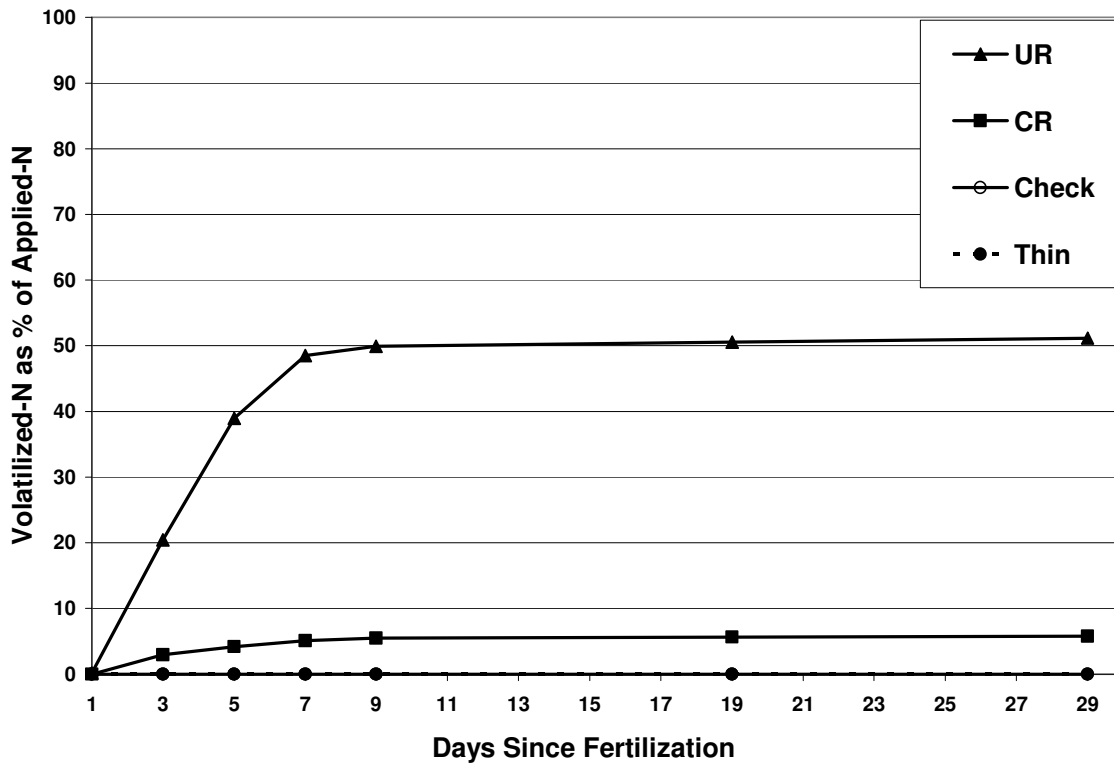


Figure 11. Cumulative N volatilization as a percent of applied N, from the mineral soil and forest floor in a mid-rotation loblolly pine plantation in the Virginia Piedmont following fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 448 kg-N ha⁻¹.

Winter Fertilization

After winter fertilization, the main effects contrasts of the 30-day N volatilization showed significant differences between treatments with URW > CRW > TH (Table 7). Also, the main effects contrasts between these treatments at each sampling date also show highly significant differences on days 3, 5, 7, and 9 with URW > CRW > TH, while on day 19, URW and CRW were both significantly greater than TH but no significant difference was found between the two fertilizer treatments (Table 8). On these days, URS volatilized, 140, 190, 80, 150, and 80 mg-N respectively, CRS volatilized 90, 100, 75, 80, and 85 mg-N respectively, while TH volatilized no ammonia (Figure 12). On these dates, URW N-volatilization was 2.1 times greater than CRW on day 3, 1.9 times greater than CRW on day 5, and 2.3 times greater and CRW on day 9 (Figure 12). These

data show that 25% of the URW total Nitrogen volatilized was trapped after 3 days, 56% after 5 days, 66% after 7 days, 92 % after 9 days, and only 8% after 19 days. Similarly, 20% of the CRW total Nitrogen volatilized was trapped after 3 days, 48% after 5 days, 65% after 7 days, 84% after 9 days, while the remaining 16% was trapped after 19 days (Figure 13). The cumulative Nitrogen volatilized as percent of applied-N shows that 4% of total URW Nitrogen applied was trapped by day 3, 10% by day 5, 12% by day 7, 18% by day 9, and 18% by day 29 (Figure 13). Also, the CRW volatilized 2% of the total Nitrogen applied by day 3, 5% by day 5, 8% by day 7, 9.5% by day 9, and 11% by day 29 (Figure 13).

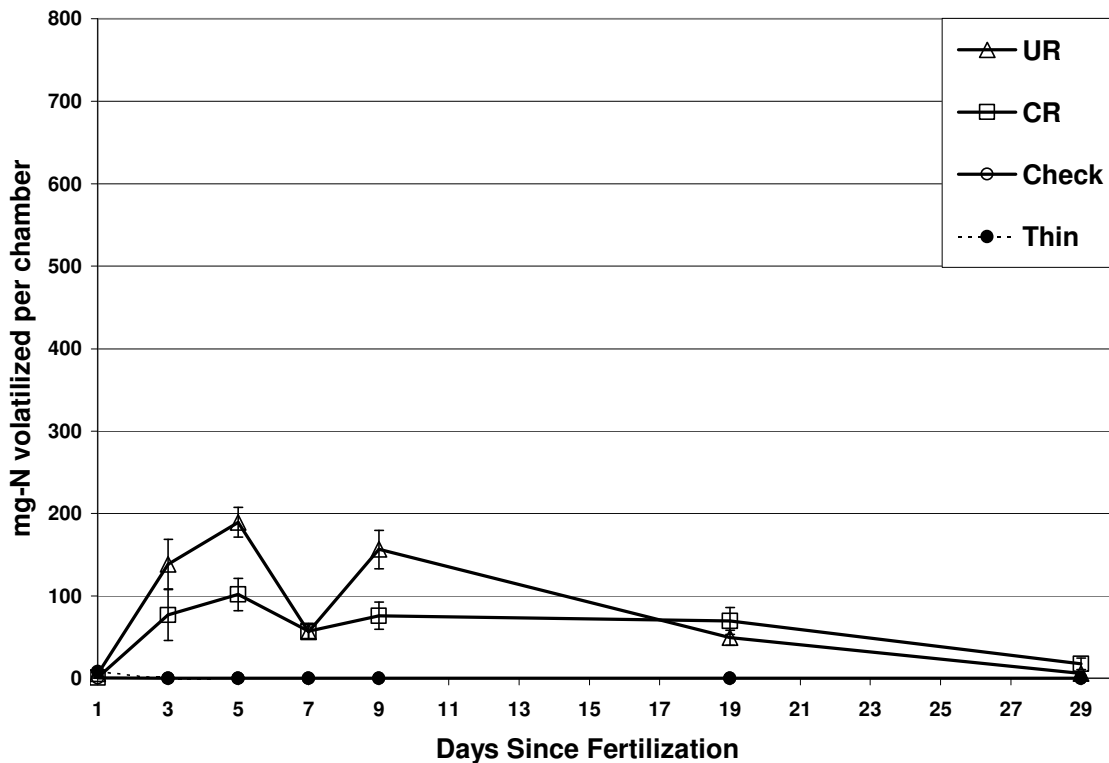


Figure 12. Absolute N volatilization from the mineral soil and forest floor in a mid-rotation loblolly pine plantation in the Virginia Piedmont following fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 448 kg-N ha⁻¹, reported as milligrams of N volatilized within closed-static chamber.

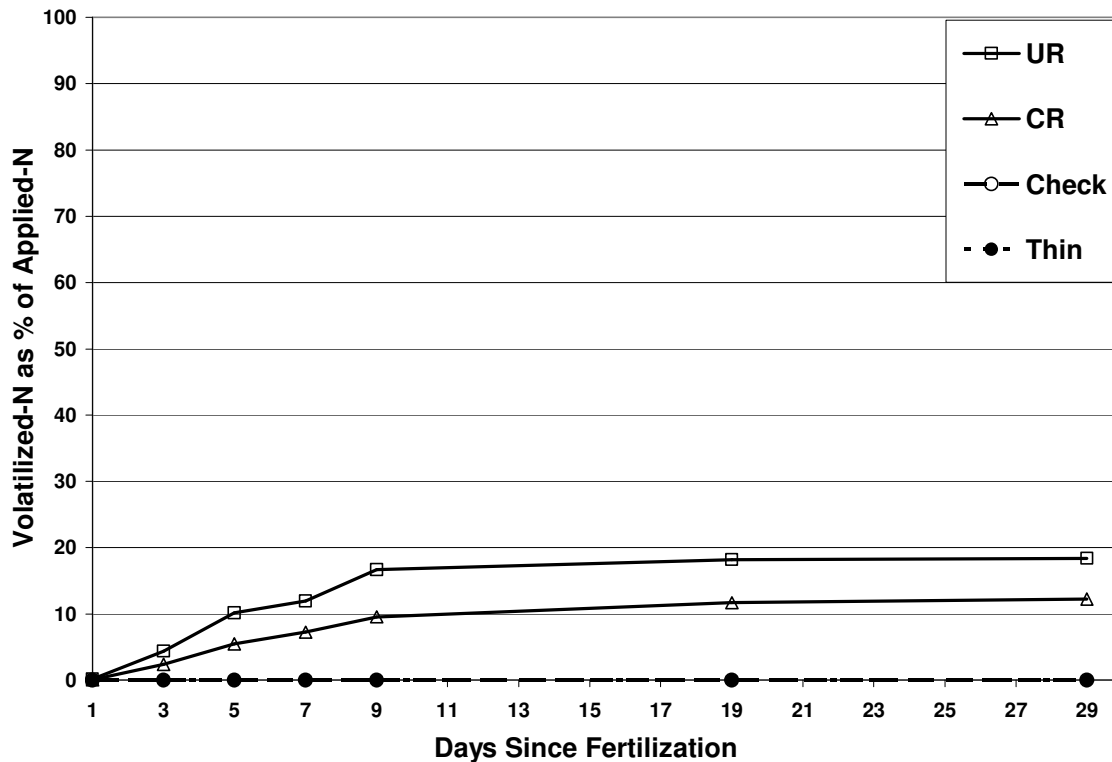


Figure 13. Cumulative N volatilization as a percent of applied N, from the mineral soil and forest floor in a loblolly pine plantation in the Virginia Piedmont following fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 448 kg-N ha⁻¹.

Above Ground Results

Several above ground responses to the six thinning and fertilization treatments were measured, including: leaf area index, foliar macro- and micro-nutrient concentrations, leaf litter biomass, and incremental growth responses as dbh, total height, and length of live crown. Due to the relatively short time frame of this study, the results from these above ground measurements did not show any significant treatment effects, therefore they are presented graphically as treatment mean response profiles with accompanying standard error bars in Appendices D through G. These above ground treatment responses are scheduled to be re-measured for at least two further growing season at which point the data will be analyzed and presented formally in a subsequent publication.

DISCUSSION

Mineral soil N availability indices showed low pre-treatment mineral soil concentrations of total KCl extractable-N, ranging from 4 to 10 mg-N kg-soil⁻¹. These low levels are characteristic of loblolly pine plantation soils, though slightly higher than previous studies located on Coastal Plain and Piedmont soils (Gurlevik et al., 2004; Piatek and Allen, 2001; Polglase et al., 1992; Zerpa, 2005). Pre-treatment total IEM-N was also quite low, ranging from 0.8 to 1.1 mg-N m⁻² day⁻¹, though also higher in comparison to levels found on coastal plain soils (Zerpa, 2005). The higher results found in this study are most likely different from Zerpa (2005), because of differing methodologies in IEM-N sampling such as the summation of O horizon and A horizon IEM-N, and shorter IEM incubation periods than used in Zerpa (2005). The slightly higher initial N availability found in this study may also be attributed to the CEC and AEC associated with the Cecil soil series, or possibly a residual effect of intensive site preparation at planting.

Check and Thin-only (TH) treatments remained near the pre-treatment levels throughout the study. Also for both measured responses of N availability, Check and TH were never significantly different when average responses were contrasted over the entire study period. During only two sampling periods during the spring of 2006, were significant differences found in the total KCl extractable-N and N-mineralization responses in the Check and Thin only plots. There were no trends associated with these sampling periods, and the variation could not be attributed to any significant soil moisture or temperature differences between treatments. Therefore, it is concluded that thinning only (TH) did not increase the absolute inorganic-N quantity in the soil, but serves in reducing competition for soil N and allows for a greater relative inorganic-N availability for residual trees, as discussed in (McGoll and Powers, 1984).

Both total KCl extractable-N and total IEM-N indices demonstrated similar trends following fertilization. Fertilizer treatments contained significantly greater total KCl extractable-N and total IEM-N than Check or TH treatments during the first 3 to 5 months immediately following both summer and winter fertilization treatments, similar to findings of Mudano (1986). Interestingly, both URS and CRS fertilization treatments demonstrated significantly greater total IEM-N than Check or TH on 6 sampling dates from 7 to 12 months after summer fertilization. This duration of increased mineral soil inorganic-N availability was much greater than expected and may be a result of residual fertilizer-N being released from O horizon exchange sites (Kissel et al., 2004) or possibly retention of inorganic-N by exchange sites on kaolinitic clays (McGoll and Powers, 1984).

After summer fertilization, the average CRS total KCl extractable-N and total IEM-N responses were significantly greater than URS, TH, or Check when contrasted over the entire sampling period. This is most likely due to differences in the volatilization characteristics of the two fertilizers. The summer fertilization with urea resulted in 51% of the applied-N being volatilized, similar to Kissel et al. (2004). Whereas controlled release Nitroform® demonstrated significantly lower volatility than urea in the summer months, as was also shown in a laboratory setting by Christianson et al. (1988). Therefore, it is concluded that following the summer fertilization, the lower solubility of controlled release Nitroform®, resulted in less fertilizer-N loss to the atmosphere through volatilization, thereby allowing a greater portion of fertilizer-N to be incorporated into the forest floor and mineral soil than urea.

The fertilizer treatments also differed in the pattern of N release. After the summer fertilization, CRS illustrated the greater difference from TH over a greater period of time, from 1.5 to 3 months more than URS (Figures 4 and 6). Also, CRS

demonstrated two distinct inorganic-N release peaks measured by both total KCl extractable-N and total IEM-N, while URS demonstrated only one brief inorganic-N release peak. This is likely a result of the immediately water soluble portion of Nitroform® releasing inorganic-N directly after fertilization, and the slowly water soluble portion releasing inorganic-N from 2-4 months after fertilization (Goertz, 1993; Jacobs et al., 2003; Praveen-Kumar and Brumme, 1995). Though this pattern of release would more closely match the needs of growing crop trees (Hauck, 1985), it was also out of phase with the active growing season in this study. Therefore, according to the release characteristics measured in this study, a Nitroform® deployment earlier in the growing season may be effective in releasing inorganic-N over a greater portion of the growing season, with the possibility of greater crop tree uptake.

The trends in soil N availability measured after winter fertilization were quite different than those measured after summer fertilization, in that urea fertilization resulted in the greater soil N-availability. Mainly, the total KCl extractable-N response in URW was significantly greater than CRW when averaged over the entire sampling period and at four sampling dates during the 2005 growing season. Also, URW was significantly greater than TH and Check for 3 months longer than was CRW. It is also interesting to note that urea fertilization in the winter resulted in greater total KCl extractable-N and total IEM-N than fertilization with urea or controlled release Nitroform® in the summer. This trend was notable in that URW total KCl extractable-N levels, 3 to 5 months after winter fertilization were 2 times greater than CRS and 4 times greater than URS total KCl extractable-N levels 3 to 5 months after summer fertilization. Also, URW total IEM-N response 2 to 5 months after winter fertilization was from 1.5 to 3 times greater than URS, 2 to 5 months after summer fertilization. The greater N availability after winter fertilization with urea is supported by the volatilization data in this study. Cumulative

volatilization in URW was similar to previous studies (Kissel et al., 2004; Mattos et al., 2003; Nommik, 1973; Volk, 1961; Watkins et al., 1972), being only 18% of applied-N, which was 3 times less than URS cumulative volatilization, and only 1.6 times greater than CRW cumulative volatilization. Clearly, decreased volatility of urea during winter months translated into greater incorporation of urea-N into the mineral soil, thereby increasing mineral soil inorganic-N availability as measured by total KCl extractable-N and total IEM-N. These higher soil inorganic-N levels associated with the winter fertilization may also be a function of lower tree uptake immediately following the winter fertilization.

Pre-treatment net N-mineralization rates were quite low in this study, ranging from -0.1 to 0.25 mg-N day⁻¹. These values are similar to previous studies (Gurlevik et al., 2004; Mattos et al., 2003; Piatek and Allen, 2001; Piatek and Allen, 1999; Raison et al., 1992), and support previous assumptions that only a small percentage of total soil N is mineralized (Rapp, 1979; Vitousek et al., 1983). N mineralization response in Check and TH treatments remained at or near pretreatment levels throughout the duration of the study, with no significant difference between the two treatments when average N mineralization response was contrasted over the entire sampling period. Therefore, it can be concluded that thinning only had no effect on Nitrogen mineralization after one year. This is somewhat contradictory to previous assumptions that thinning increases light and rainfall penetration to the forest floor and in turn soil temperature and moisture which has been shown to increase net N mineralization (Gurlevik et al., 2004; Goncalves and Carlyle, 1994). The lack of N mineralization response to thinning in this study may be explained by the low intensity of the thinning operation which did not significantly increase soil temperature or moisture over the control.

N mineralization response to fertilization was also minimal, with no significant differences between fertilizer treatments at either application date and Check or TH, when average N mineralization response was contrasted over the entire sampling period. This supports the findings of Polglase et al. (1992) who demonstrated the inability of fertilizer to positively affect N mineralization. However, these findings are contradictory to those found in other studies that show significant increases in N mineralization after fertilization (Maimone et al., 1991; and Gurlevik et al., 2004). One possible reason for these contradictory findings is that this study utilized sequential *in situ* mineralization techniques (Raison et al., 1987) with two week incubation periods, while other studies utilized laboratory methods (Maimone et al., 1991) or longer incubation periods up to two months (Gurlevik et al., 2004). Adams and Attiwill (1986) show that changes in incubation length can have significant consequences on *in situ* N mineralization estimates because of the inherent rate of N turnover at a site and the decomposition of fine roots within the sampling core.

Although N mineralization demonstrated no overall significant differences between treatments, there were several individual dates where treatment responses were significantly different. The most striking N mineralization response was shown in the CRS treatment (Figure 8). On the sampling dates from 2 to 4 months following summer fertilization with Nitroform, a significant positive N mineralization was measured in CRS over URS, Check and TH. This initial increase in net N mineralization may have a 2 fold explanation. First, there was a large enough increase in soil inorganic-N from the immediately water soluble portion of Nitroform® (as shown in the total KCl extractable-N and total IEM-N data), which satisfied microbial N demands and in turn allowed for the mineralization of the short chain polymer-urea portion of Nitroform®. This would support the N mineralization scenarios described by Schimmel and Bennett (2004), where

as overall N availability increases, microbial N dependence on N diffusing from N-rich sites is diminished, effectively increasing mineralization. This trend would be beneficial in a plantation setting during the growing season, however it was short-lived in this study, lasting only one month. In the early spring following the summer fertilization, as soil temperatures increased, CRS demonstrated significant net N immobilization for 2 months, similar to that found in previous studies (Raison et al., 1992; Vitousek and Matson, 1985; and Gurlevik et al., 2004). One possible explanation for the N immobilization in CRS is that during spring months, microbial activity begins to increase with rising temperatures (Paul and Clark, 1989) and increases in photosynthate C exuded from pine roots (Taneva et al., 2006), at which point the long-chain polymer urea portion of Nitroform® also serves as a labile C source which further stimulates soil microbes to actively compete for inorganic-N diffusing through the mineral soil (Schimmel and Bennett, 2004), effectively increasing microbial immobilization of N as shown in previous studies (Compton and Boone, 2002; Gallardo and Schlesinger, 1995; and Whalen et al., 2000). Interestingly, during this period of N immobilization in CRS, the total KCl extractable-N and IEM-N remained significantly greater than Check or TH, however at the end of the two month immobilization period, these soil N availability indices had decreased to pre-treatment levels. This demonstrates that the long-chain polymer portion of Nitroform, sufficiently increased the capacity of net N immobilization to a point where the quantity of mineral soil inorganic-N was significantly diminished. The immobilization of N from controlled release ureaform fertilizers such as Nitroform® was hypothesized by Hauck (1968) to possibly reduce the efficiency in meeting the N requirements of crop trees, since the immobilized N must be re-mineralized for crop tree uptake. The measurement of the subsequent re-mineralization of immobilized Nitroform®-N was outside of the time scope of this study. However, a concurrent study

is in place to understand the long-term above-ground effects of both urea and Nitroform® on loblolly pine incremental growth, foliar nutrient status, leaf area increment, and litterfall, which would reflect the intensity of the re-mineralization.

In conclusion, thinning only had no effect on the N availability indices measured in this study. Controlled release Nitroform® displayed significantly lowered volatilization properties at less than 15% of applied-N during both summer and winter applications. Also, release of inorganic-N from controlled release Nitroform® was greater and more prolonged than urea after summer fertilization, which would make it a biologically viable summer season fertilizer. However, the labile Carbon in the long-chain polymer urea portion of this material, equivalent to 400 kg-C ha⁻¹ in this study, stimulated microbial N immobilization during early spring, thereby decreasing the inorganic-N available for crop trees in this mid-rotation scenario. It has been previously shown that large amounts of labile photosynthate C are also exuded by pine roots during this time period (Taneva, et al., 2006). Therefore the additional labile C from controlled release Nitroform®, coupled with labile C inputs from pine roots, appears to have increased the C:N ratio to a point where soil microbes were once again N limited, resulting in N immobilization. In addition, winter application of urea increased mineral soil N-availability more than winter application of Nitroform®. Though urea demonstrated typical volatilization properties at greater than 50% of applied-N during summer application, as shown in previous studies (Kissel et al., 2004), this volatilization was significantly decreased to 18% of applied-N during winter fertilization. This reduction in winter urea volatilization allowed for greater incorporation of urea-N into the mineral soil and thus greater soil total KCl extractable-N and total IEM-N than either summer or winter application of controlled release Nitroform®.

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CHAPTER 1V.

GENERAL CONCLUSIONS

Thinning only treatment responses were not significantly different from Check treatment responses throughout the time frame of this study. Therefore it may be concluded that thinning does not significantly affect absolute levels of total KCl extractable-N, IEM-N, N-mineralization, or N-volatilization. Over the 1 year time frame of this study, data suggest that thinning affects pine plantation nutrient availability only by increasing the relative amount of available-N for residual crop trees, not by an absolute increase in the quantity of inorganic-N in the mineral soil.

Both urea and Nitroform® fertilizer treatments significantly increased total KCl extractable-N and total IEM-N relative to thin-only and check, during both winter and summer treatments. Thin-only and Check treatments remained at or near pre-treatment levels of $1.0 \text{ mg-N m}^{-2} \text{ day}^{-1}$ for total IEM-N and $5 \text{ mg-N kg-soil}^{-1}$ for total KCl extractable-N, throughout the study. Whereas, summer fertilization treatments peaked at $12 \text{ mg-N m}^{-2} \text{ day}^{-1}$ for total IEM-N and $20\text{-}40 \text{ mg-N kg-soil}^{-1}$ for total KCl extractable-N. Summer fertilization with Nitroform demonstrated the most prolonged increase in total KCl extractable-N and total IEM-N, though only for 2 to 4 sampling periods (1 to 2 months) more than summer fertilization with urea. Urea fertilization in winter demonstrated the greatest increase in the mineral soil N-availability indices, ranging from 2 to 3 times greater than any other fertilization treatment at similar time intervals after fertilizer application.

Nitrogen mineralization also demonstrated low pre-treatment levels ranging from -0.1 to $0.25 \text{ mg-N day}^{-1}$, and no significant differences between Check and Thin-only treatments when average response was contrasted over the entire sampling period. Interestingly, fertilizer treatments had only minimal positive effects on Nitrogen

mineralization, generally fluctuating between 0 and 0.5 mg-N day⁻¹. None of the fertilizer treatments were found to be significantly greater than Check or Thin-only when average N mineralization response was contrasted over the entire sampling period. However, summer deployment of Nitroform®, demonstrated 2 dates of significantly greater N mineralization, 2 months after fertilizer application, and 4 dates of significantly greater N immobilization at 7 and 8 months after fertilizer application.

No N-volatilization was measured in Check or TH treatments, while both urea and Nitroform® fertilization significantly increased N-volatilization over Check and TH at both fertilization dates. However, Nitroform® demonstrated much lower volatility with 8% and 11% of applied-N being volatilized during summer and winter applications respectively, which was similar to rates found in a previous study (Christianson, 1988). Urea volatility was significantly greater than Nitroform, Check, and TH at both application dates, volatilizing 51% and 18% of applied-N during summer and winter application respectively, which is similar to past studies (Kissel et al., 2004; Mattos et al., 2003 ; Nommik, 1973; Volk, 1961; Watkins et al., 1972).

Though fertilization with controlled release Nitroform® demonstrated prolonged release characteristics over a 12 month sampling period, this fertilizer material also demonstrated marked N-immobilization which in turn decreased soil inorganic N-availability to near pre-treatment levels. Fertilization with urea during winter months provided for lower volatility, minimal N-immobilization, and greater mineral soil inorganic-N availability. Therefore this study shows that because of the high cost of controlled release Nitroform® fertilizers, being 2 to 3 times greater than urea on a per ton basis, a winter deployment of urea remains the most cost effective method for ameliorating southeastern mid-rotation pine plantation N-deficiencies. However, the slow N release characteristics of controlled release Nitroform® may be beneficial for

mitigating environmental concerns surrounding urea fertilization, such as reducing winter nitrate leaching and nitrate run-off into streams.

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**APPENDIX A:
KCl EXTRACTABLE-N RESULTS ASSOCIATED WITH CHAPTER III**

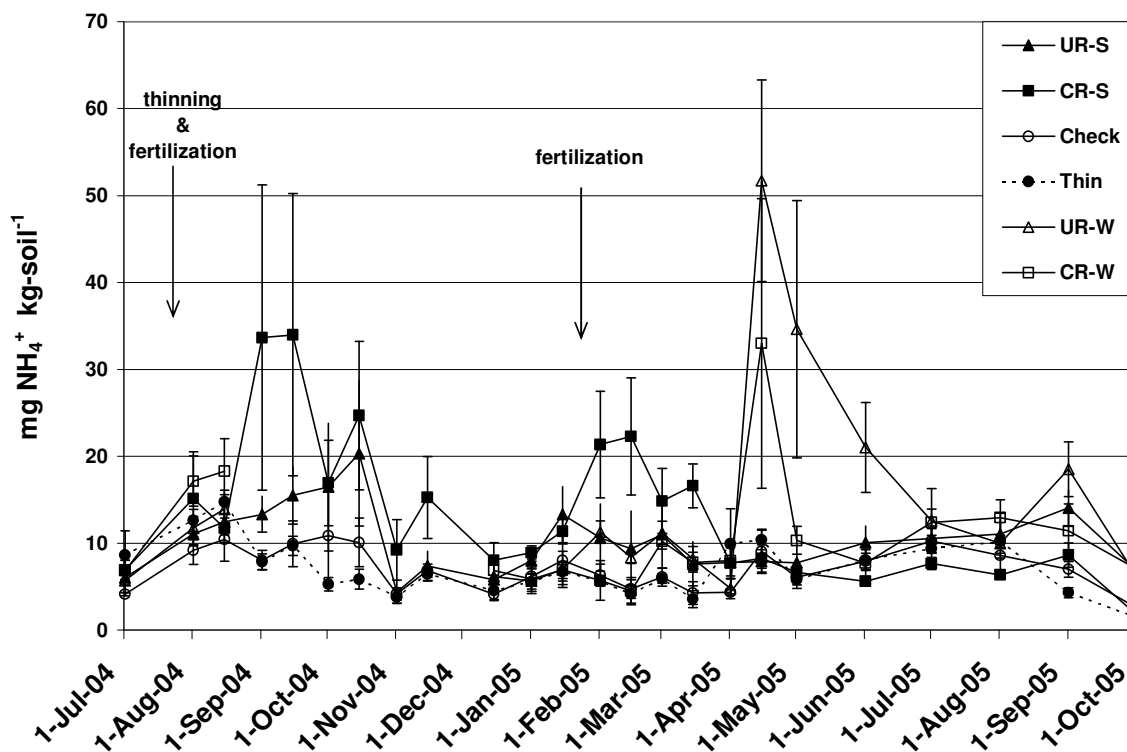


Figure A1. KCl extractable- NH_4^+ from the surface 15 cm of the A horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

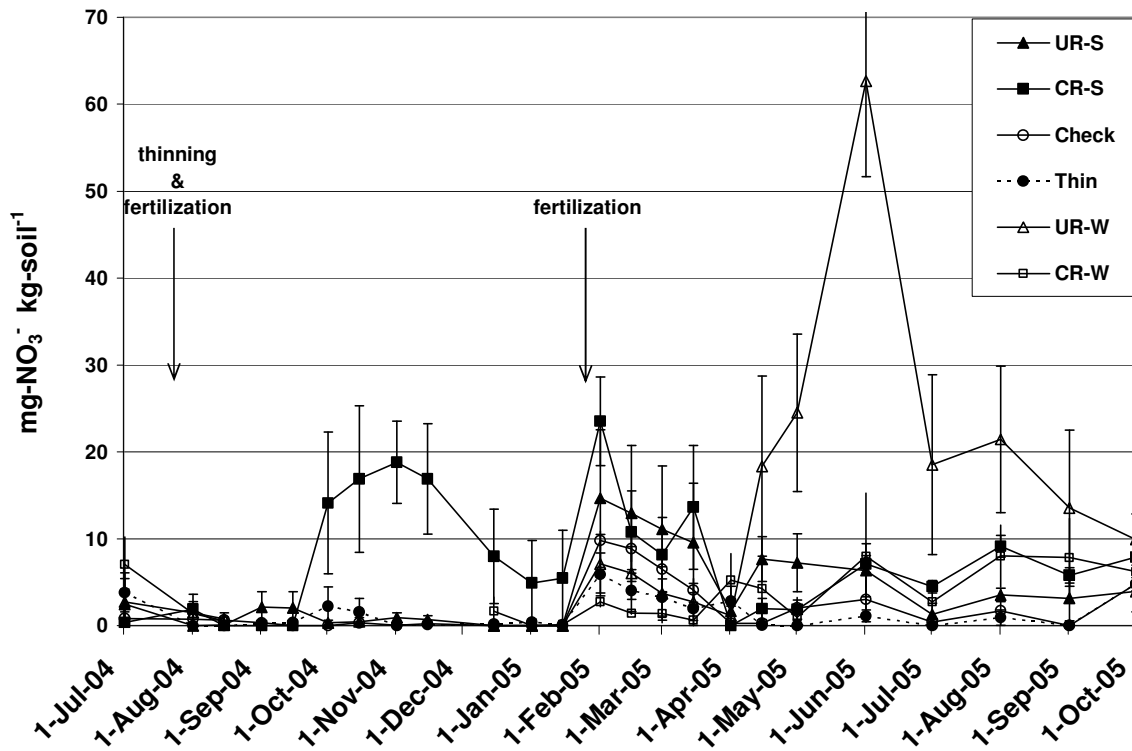


Figure A2. KCl extractable-NO₃⁻ from the surface 15 cm of the A horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied at a rate of 224 kg-N ha⁻¹.

APPENDIX B:
CUMULATIVE NITROGEN MINERALIZATION FROM THE SURFACE 15 CM
OF THE MINERAL SOIL IN A MID-ROTATION LOBLOLLY PINE
PLANTATION IN THE VIRGINIA PIEDMONT FOLLOWING THINNING AND
FERTILIZATION WITH UREA OR CONTROLLED RELEASE NITROFORM®
APPLIED AT A RATE OF 224 KG-N HA⁻¹

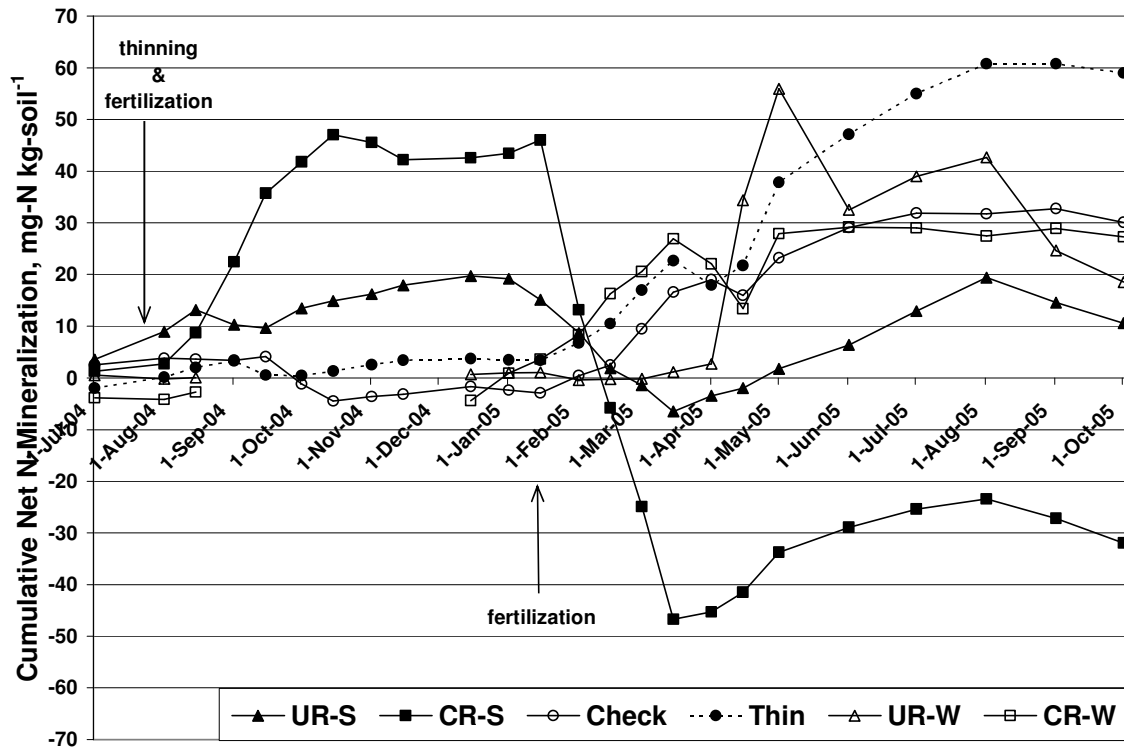


Figure B1. Cumulative Net N mineralization in the upper 15 cm of the A horizon over a 16 month sampling period including 24, 14-day *in situ* incubation periods, in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

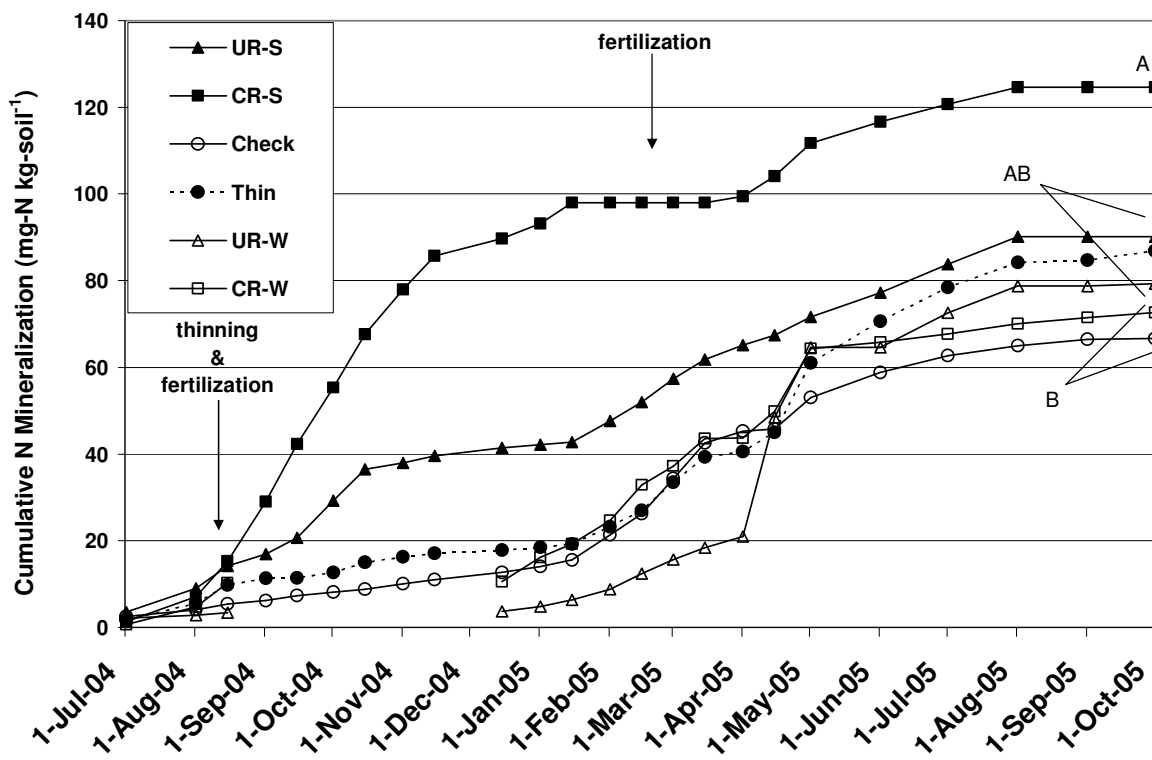


Figure B2. Cumulative N mineralization in the upper 15 cm of the A horizon over a 16 month sampling period including 24, 14-day *in situ* incubation periods, in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

**APPENDIX C:
ION EXCHANGE MEMBRANE RESULTS ASSOCIATED WITH CHAPTER III**

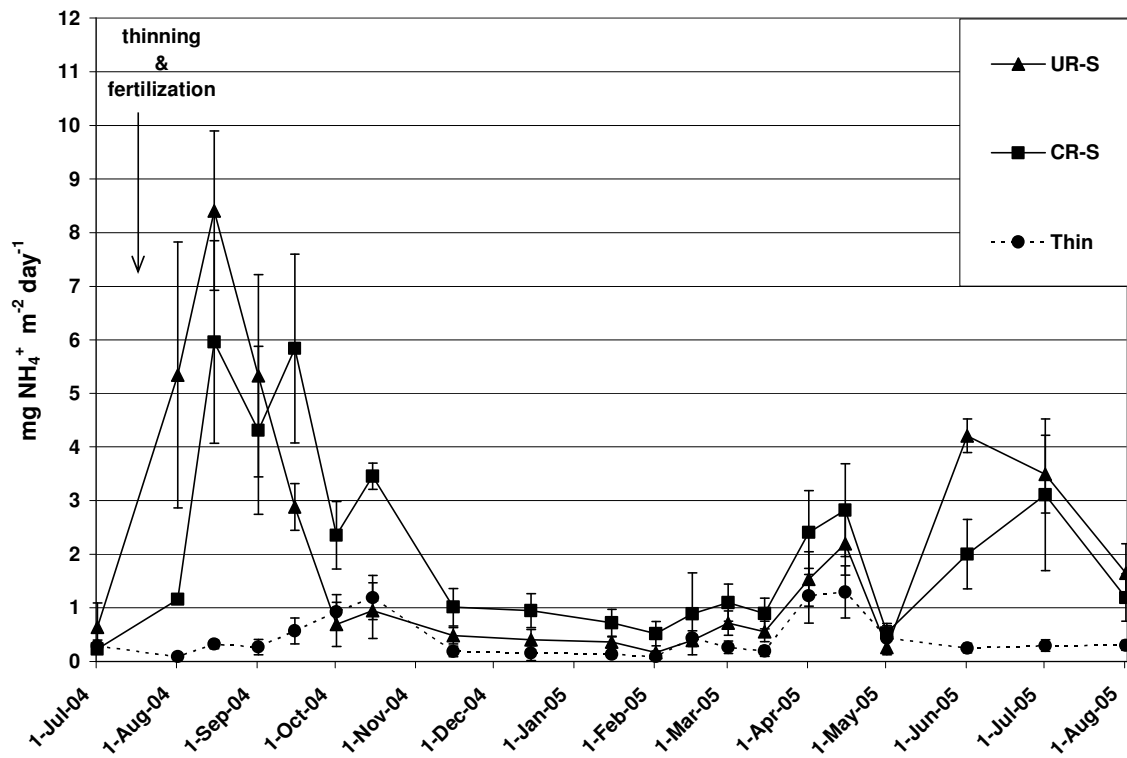


Figure C1. Cation ion exchange membrane- NH_4^+ from the O horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha^{-1} , and reported in units of mg-N m^{-2} of ion exchange membrane surface.

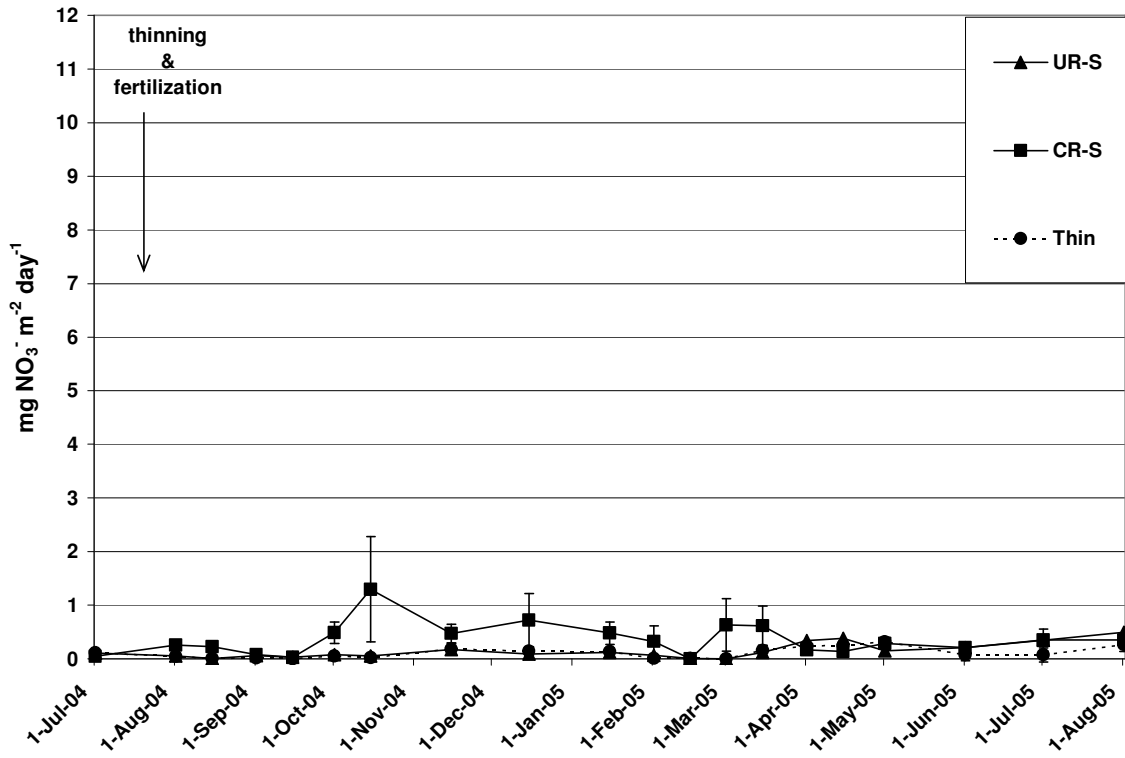


Figure C2. Anion exchange membrane-NO₃⁻ from the O horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

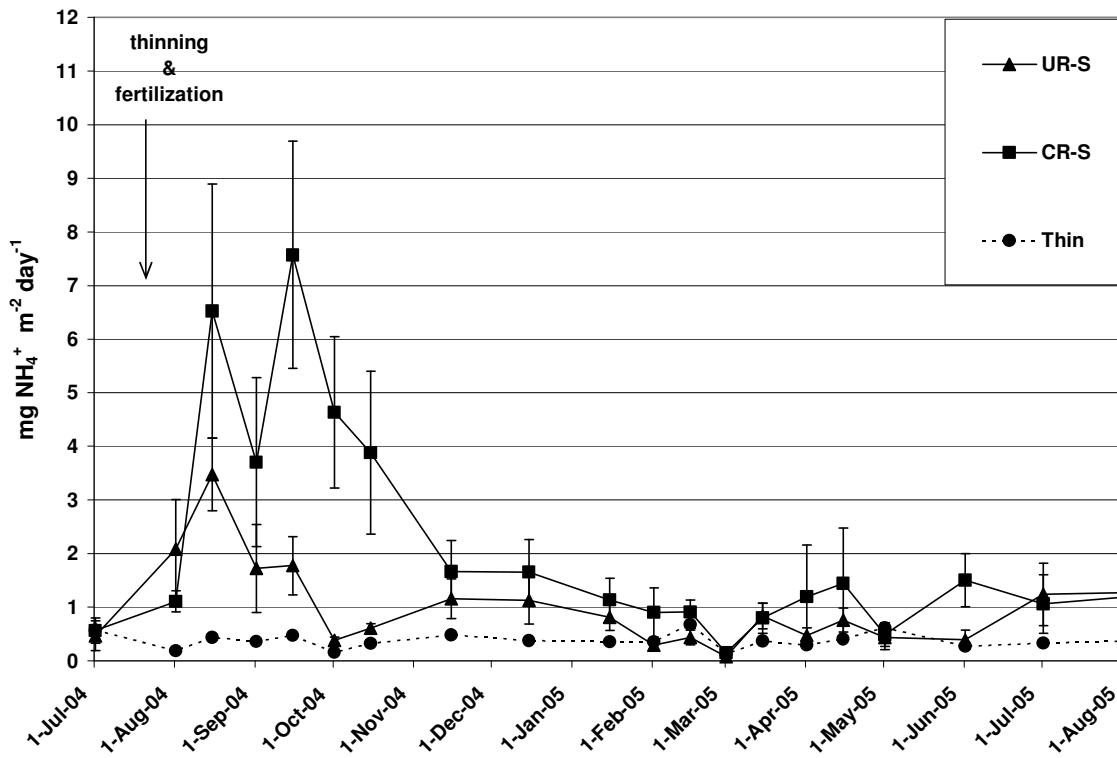


Figure C3. Cation exchange membrane- NH_4^+ from the upper 15cm of the A horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha^{-1} , and reported in units of mg-N m^{-2} of ion exchange membrane surface.

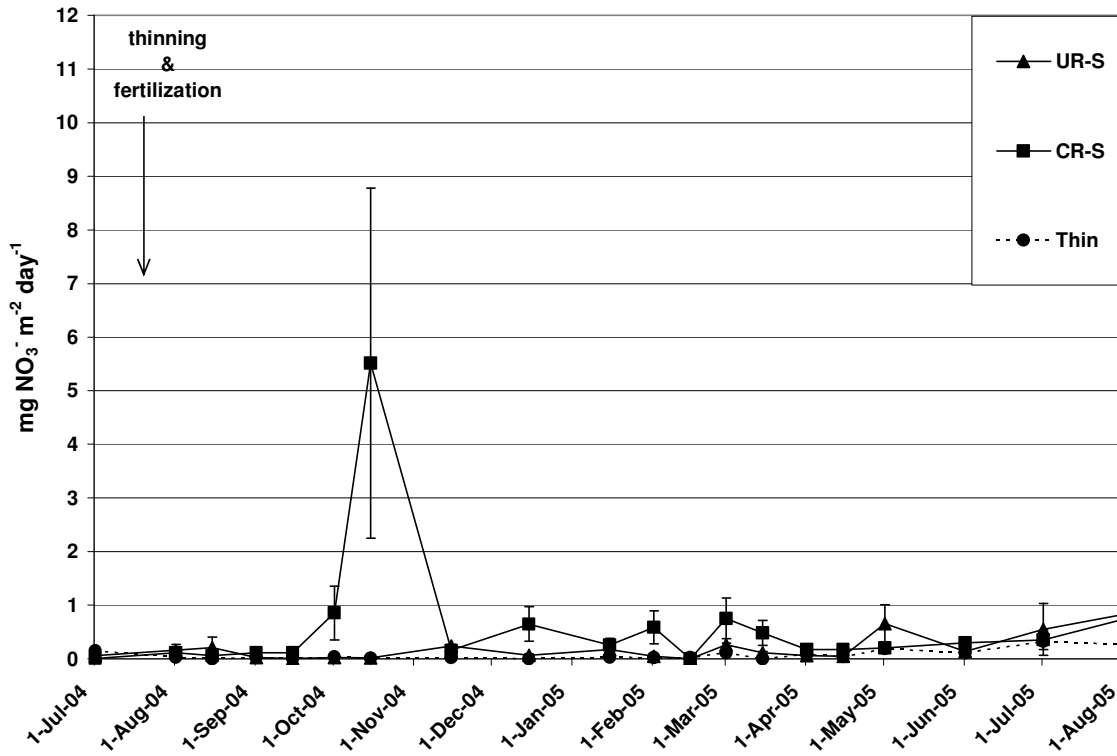


Figure C4. Anion exchange membrane-NO₃⁻ from the upper 15cm of the A horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the summer at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

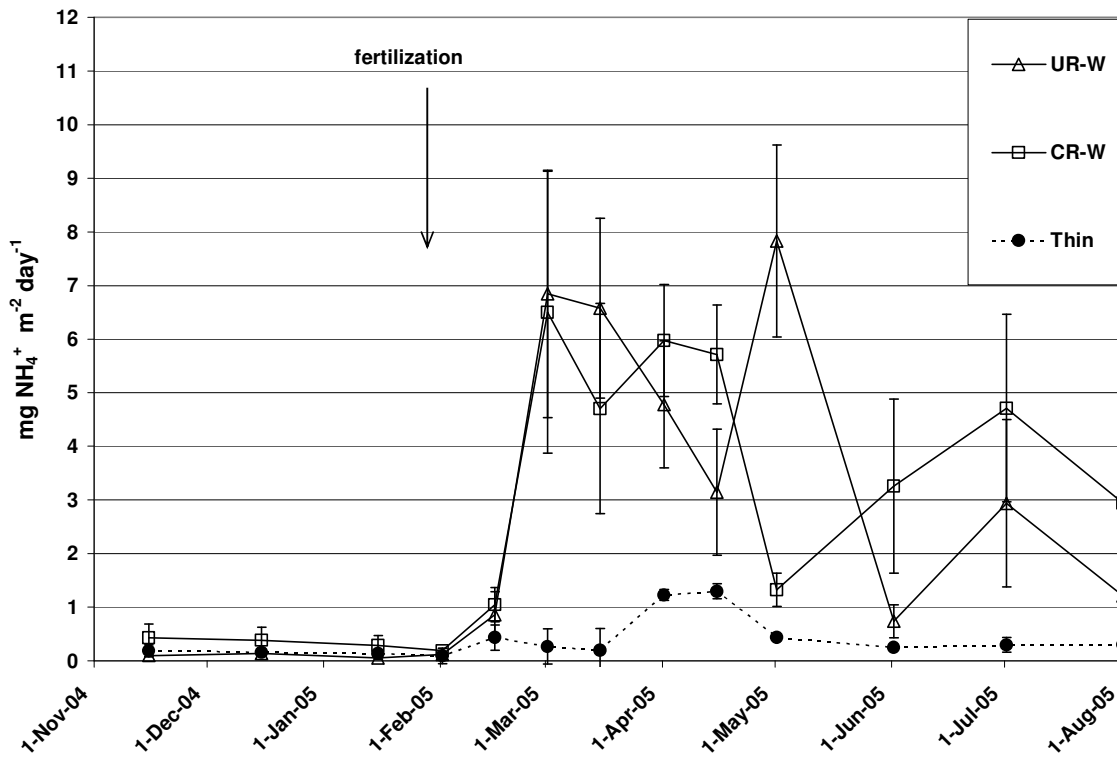


Figure C5. Cation exchange membrane- NH_4^+ from the O horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha^{-1} , and reported in units of mg-N m^{-2} of ion exchange membrane surface.

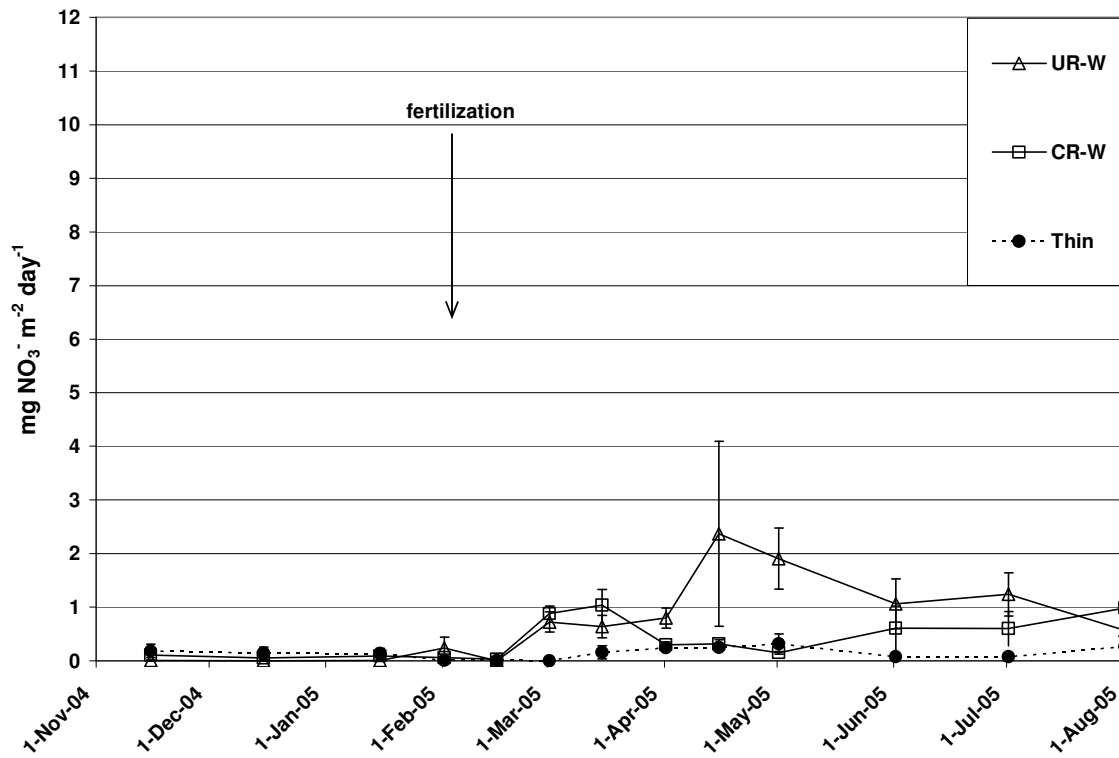


Figure C6. Anion exchange membrane-NO₃⁻ from the O horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

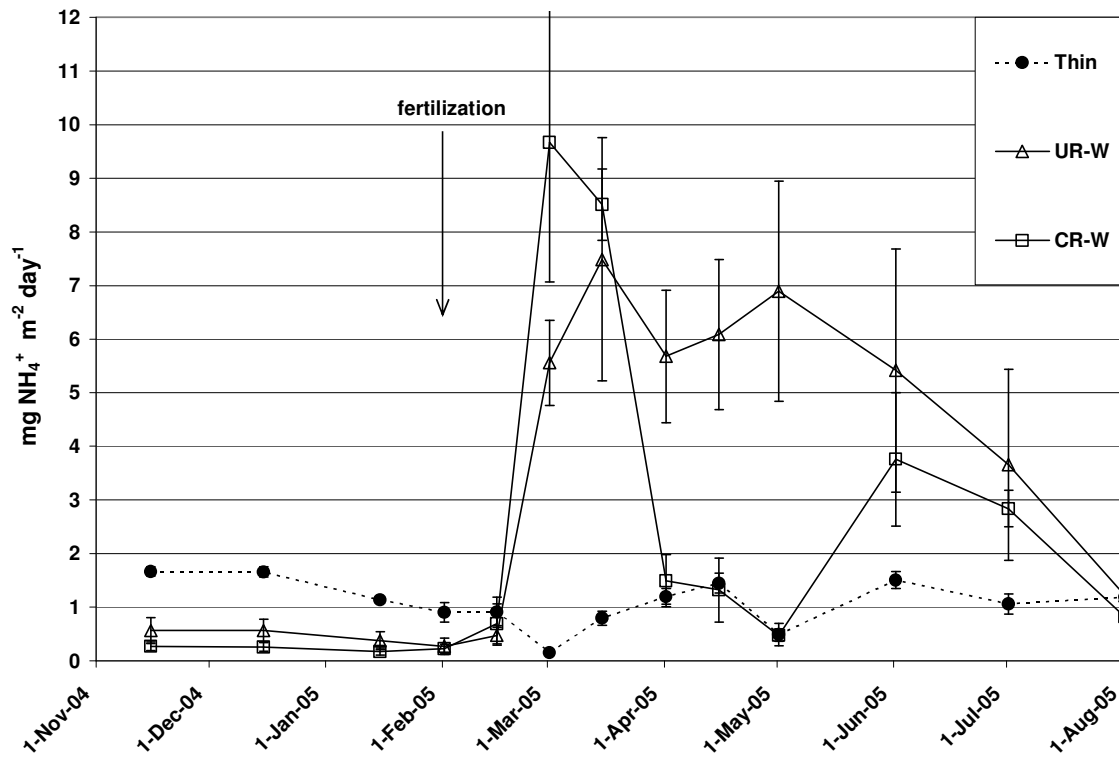


Figure C7. Cation exchange membrane-NH₄⁺ from the upper 15cm of the A horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

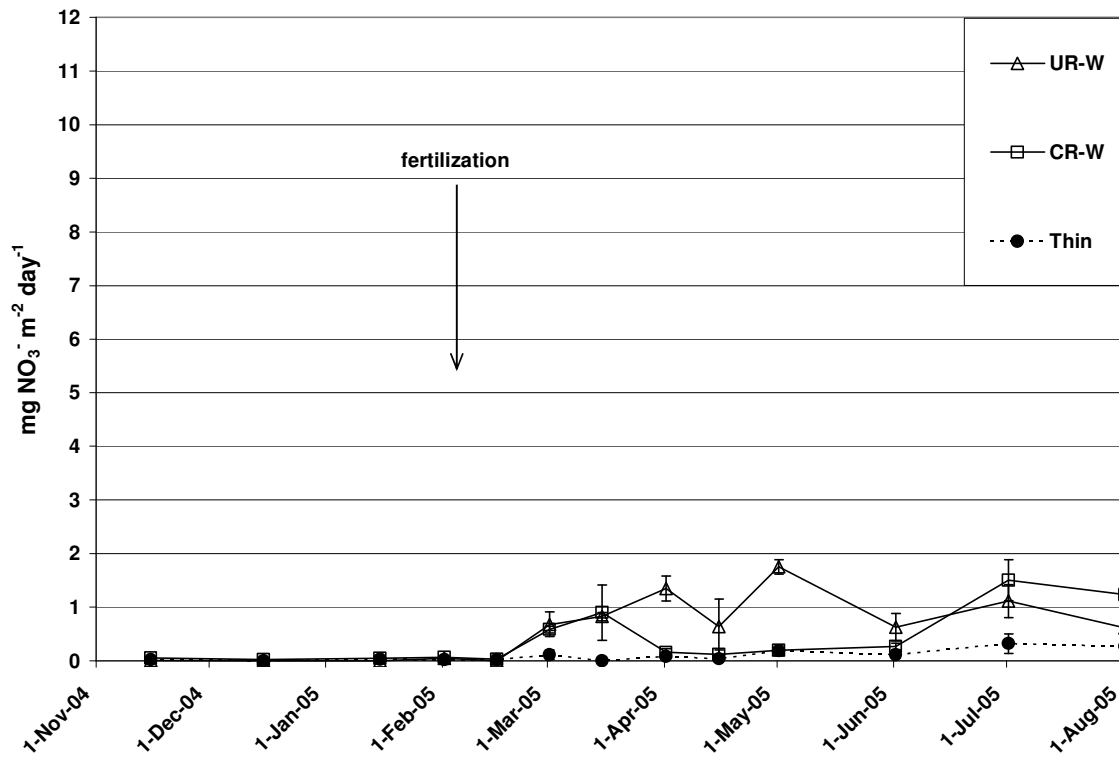


Figure C8. Anion exchange membrane-NO₃⁻ from the upper 15cm of the A horizon in a loblolly pine plantation in the Virginia Piedmont following thinning and fertilization with urea or controlled release Nitroform® applied during the winter at a rate of 224 kg-N ha⁻¹, and reported in units of mg-N m⁻² of ion exchange membrane surface.

**APPENDIX D:
PLOT LEVEL TREE GROWTH RESULTS FROM A MID-ROTATION
LOBLOLLY PINE PLANTATION IN THE VIRGINIA PIEDMONT
FOLLOWING THINNING AND FERTILIZATION WITH UREA OR
CONTROLLED RELEASE NITROFORM® APPLIED AT A RATE OF 224 KG-N
HA⁻¹**

Table D1. Plot level mean tree growth response measured as the difference between January, 2004 initial plot measurement, and January 2006 plot re-measurement including dbh (DBH), total height (HT), height to live crown (HTC), length of live crown (LC), and basal area (BA), taken from a 22-year-old loblolly pine plantation in the Piedmont of Virginia after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

| Block | Treatment | Plot | DBH (cm) | HT (m) | HTC (m) | LC (m) | BA (m ² ha ⁻¹) |
|-------|-----------|------|----------|--------|---------|--------|---------------------------------------|
| 1 | 1 | 5 | 1.03 | 1.68 | 1.18 | 0.49 | 2.41 |
| 2 | 1 | 2 | 0.83 | 1.93 | 1.65 | 0.28 | 1.73 |
| 3 | 1 | 3 | 1.23 | 2.40 | 1.54 | 0.86 | 2.88 |
| 4 | 1 | 6 | 1.00 | 2.02 | 0.88 | 1.14 | 2.35 |
| 1 | 2 | 10 | 1.56 | 1.79 | 1.76 | 0.03 | 3.32 |
| 2 | 2 | 9 | 1.34 | 1.84 | 1.54 | 0.30 | 2.95 |
| 3 | 2 | 13 | 1.05 | 1.59 | 1.40 | 0.19 | 2.55 |
| 4 | 2 | 12 | 1.05 | 2.01 | 1.74 | 0.27 | 2.50 |
| 1 | 3 | 8 | 1.02 | 2.04 | 2.28 | -0.24 | 2.40 |
| 2 | 3 | 4 | 0.94 | 1.97 | 1.97 | 0.00 | 2.39 |
| 3 | 3 | 7 | 0.89 | 1.86 | 1.43 | 0.43 | 2.10 |
| 4 | 3 | 18 | 1.16 | 1.81 | 1.25 | 0.57 | 2.61 |
| 1 | 4 | 11 | 0.84 | 1.23 | 1.16 | 0.07 | 3.27 |
| 2 | 4 | 20 | 0.51 | 1.03 | 1.69 | -0.65 | 3.24 |
| 3 | 4 | 16 | 0.82 | 0.84 | 0.86 | -0.02 | 3.85 |
| 4 | 4 | 15 | 1.25 | 1.36 | 1.51 | -0.15 | 4.59 |
| 1 | 5 | 23 | 2.02 | 1.35 | 0.82 | 0.54 | 3.69 |
| 2 | 5 | 24 | 1.26 | 0.97 | 0.82 | 0.15 | 2.52 |
| 3 | 5 | 17 | 1.34 | 1.81 | 1.34 | 0.46 | 2.91 |
| 4 | 5 | 19 | 0.98 | 1.93 | 2.13 | -0.19 | 2.22 |
| 1 | 6 | 22 | 1.13 | 1.16 | -0.93 | 2.08 | 2.51 |
| 2 | 6 | 1 | 0.77 | 2.24 | 1.76 | 0.48 | 1.86 |
| 3 | 6 | 14 | 0.90 | 1.77 | 2.02 | -0.25 | 1.85 |
| 4 | 6 | 21 | 1.10 | 2.37 | 1.59 | 0.80 | 2.46 |

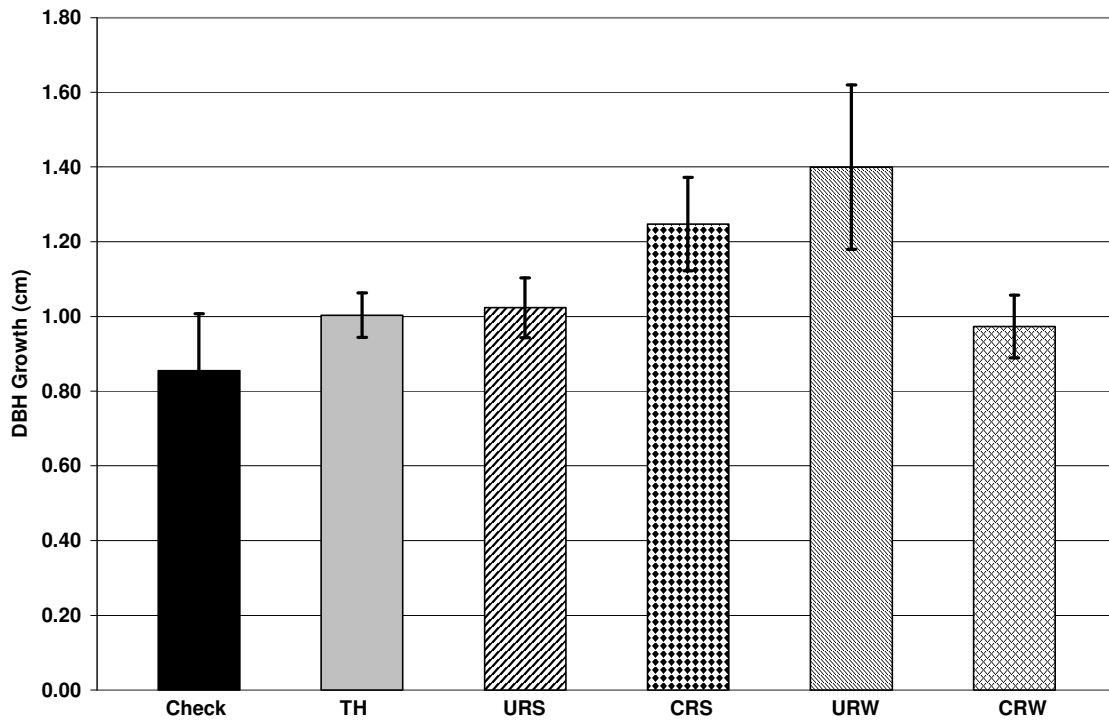


Figure D1. Mean dbh growth by treatment measured as the difference between initial plot measurement in January, 2004 and plot re-measurement in January, 2006 in a 22-year-old loblolly pine plantation in the Piedmont of Virginia following thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Error bars represent +/- 1 standard error.

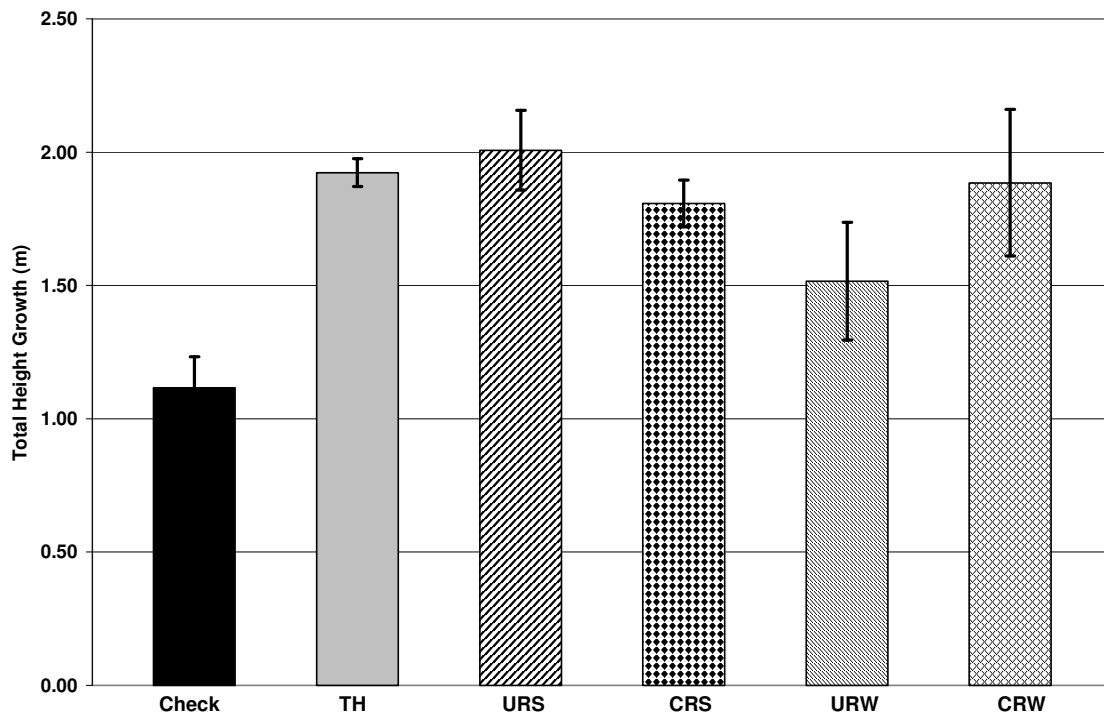


Figure D2. Mean total height growth by treatment measured as the difference between initial plot measurement in January, 2004 and plot re-measurement in January, 2006 in a 22-year-old loblolly pine plantation in the Piedmont of Virginia following thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹ Error bars represent +/- 1 standard error.

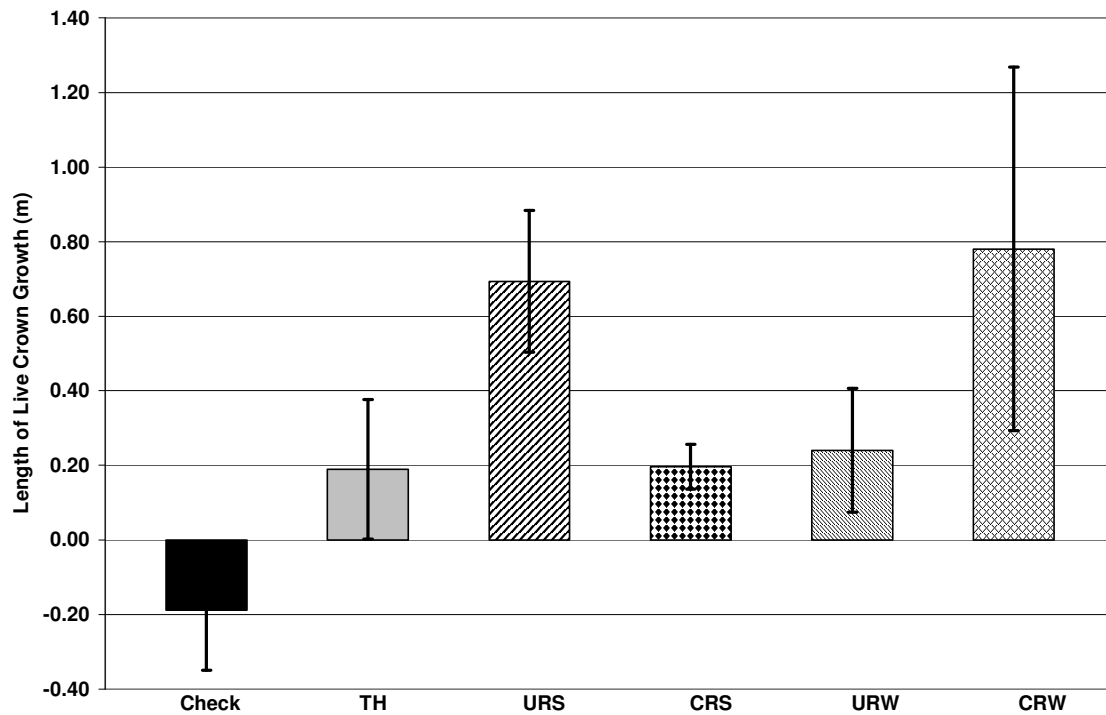


Figure D3. Mean length of live crown growth by treatment measured as the difference between initial plot measurement in January, 2004 and plot re-measurement in January, 2006 in a 22-year-old loblolly pine plantation in the Piedmont of Virginia following thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Error bars represent +/- 1 standard error.

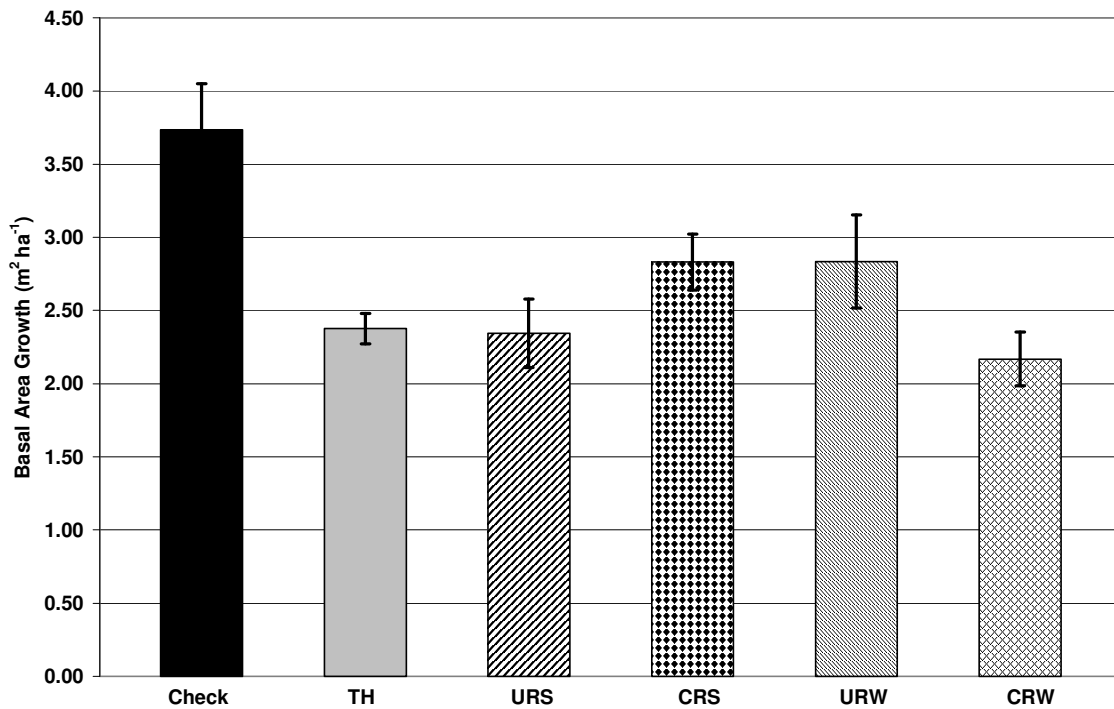


Figure D4. Mean basal area growth by treatment measured as the difference between initial plot measurement in January, 2004 and plot re-measurement in January, 2006 in a 22-year-old loblolly pine plantation in the Piedmont of Virginia following thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Error bars represent +/- 1 standard error.

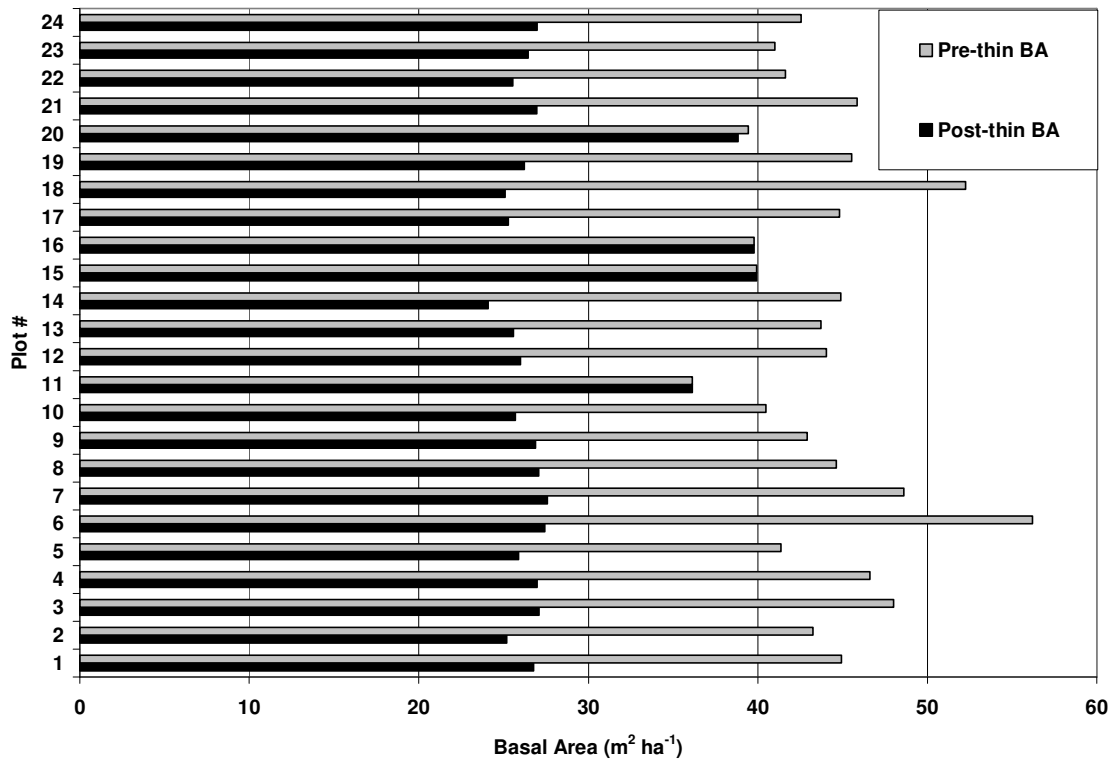


Figure D5. Plot level mean basal area values measured before thinning (pre-thin BA) and after thinning (post-thin BA) in a 22-year-old loblolly pine plantation in the Piedmont of Virginia.

APPENDIX E.
FOLIAR WEIGHT AND NUTRIENT CONCENTRATION RESULTS FROM A
MID-ROTATION LOBLOLLY PINE PLANTATION IN THE VIRGINIA
PIEDMONT FOLLOWING THINNING AND FERTILIZATION WITH UREA
OR CONTROLLED RELEASE NITROFORM® APPLIED AT A RATE OF 224
KG-N HA⁻¹

Table E1. Summary of pre-treatment foliar weight and macro-nutrient concentrations (%) by plot from a 22-year-old loblolly pine plantation in the Piedmont of Virginia.

| Block | Trt. | Plot | Weight | C | N | P | K | Ca | Mg |
|-------|------|------|---------------------------|----------|-------|-------|-------|-------|-------|
| | | | <i>mg / 100 fascicles</i> | <i>%</i> | | | | | |
| 1 | 1 | 5 | 19.72 | 55.650 | 1.526 | 0.130 | 0.437 | 0.266 | 0.087 |
| 2 | 1 | 2 | 16.04 | 56.170 | 1.394 | 0.108 | 0.386 | 0.255 | 0.093 |
| 3 | 1 | 3 | 17.06 | 56.640 | 1.435 | 0.114 | 0.416 | 0.253 | 0.081 |
| 4 | 1 | 6 | 18.50 | 57.000 | 1.589 | 0.124 | 0.452 | 0.250 | 0.088 |
| 1 | 2 | 10 | 18.61 | 58.030 | 1.739 | 0.139 | 0.438 | 0.251 | 0.099 |
| 2 | 2 | 9 | 18.12 | 57.434 | 1.532 | 0.127 | 0.456 | 0.242 | 0.107 |
| 3 | 2 | 13 | 19.13 | 57.720 | 1.672 | 0.133 | 0.445 | 0.260 | 0.095 |
| 4 | 2 | 12 | 20.14 | 57.210 | 1.553 | 0.114 | 0.403 | 0.210 | 0.095 |
| 1 | 3 | 8 | 19.30 | 56.130 | 1.535 | 0.130 | 0.468 | 0.280 | 0.100 |
| 2 | 3 | 4 | 17.78 | 58.450 | 1.500 | 0.127 | 0.429 | 0.242 | 0.083 |
| 3 | 3 | 7 | 17.26 | 58.180 | 1.589 | 0.123 | 0.454 | 0.208 | 0.077 |
| 4 | 3 | 18 | 13.41 | 56.650 | 1.483 | 0.119 | 0.383 | 0.199 | 0.093 |
| 1 | 4 | 11 | 15.91 | 56.230 | 1.569 | 0.136 | 0.444 | 0.251 | 0.104 |
| 2 | 4 | 20 | 17.57 | 56.960 | 1.547 | 0.118 | 0.426 | 0.223 | 0.087 |
| 3 | 4 | 16 | 15.27 | 56.450 | 1.350 | 0.117 | 0.388 | 0.231 | 0.087 |
| 4 | 4 | 15 | 17.12 | 57.080 | 1.554 | 0.211 | 0.397 | 0.252 | 0.107 |
| 1 | 5 | 23 | 15.27 | 56.710 | 1.607 | 0.130 | 0.374 | 0.271 | 0.105 |
| 2 | 5 | 24 | 18.12 | 57.600 | 1.544 | 0.131 | 0.397 | 0.190 | 0.086 |
| 3 | 5 | 17 | 17.32 | 57.620 | 1.496 | 0.131 | 0.410 | 0.276 | 0.100 |
| 4 | 5 | 19 | 15.94 | 56.900 | 1.538 | 0.147 | 0.454 | 0.236 | 0.108 |
| 1 | 6 | 22 | 16.46 | 56.150 | 1.580 | 0.131 | 0.455 | 0.204 | 0.104 |
| 2 | 6 | 1 | 17.10 | 57.060 | 1.491 | 0.114 | 0.440 | 0.229 | 0.097 |
| 3 | 6 | 14 | 18.22 | 57.020 | 1.596 | 0.121 | 0.412 | 0.170 | 0.088 |
| 4 | 6 | 21 | 18.41 | 56.780 | 1.584 | 0.122 | 0.444 | 0.248 | 0.091 |

Table E2. Summary of pre-treatment foliar macro-nutrient content (mg / 100 fascicles) by plot from a 22-year-old loblolly pine plantation in the Piedmont of Virginia.

| Block | Trt. | Plot | N | P | K | Ca | Mg |
|-------|------|------|---------------------------|--------|--------|--------|--------|
| | | | <i>mg / 100 fascicles</i> | | | | |
| 1 | 1 | 5 | 300.927 | 25.565 | 86.186 | 52.364 | 17.111 |
| 2 | 1 | 2 | 223.598 | 17.291 | 61.903 | 40.876 | 14.988 |
| 3 | 1 | 3 | 244.811 | 19.523 | 71.038 | 43.116 | 13.887 |
| 4 | 1 | 6 | 293.965 | 22.969 | 83.700 | 46.280 | 16.370 |
| 1 | 2 | 10 | 323.628 | 25.933 | 81.443 | 46.683 | 18.452 |
| 2 | 2 | 9 | 277.598 | 22.949 | 82.604 | 43.812 | 19.479 |
| 3 | 2 | 13 | 319.854 | 25.414 | 85.042 | 49.759 | 18.168 |
| 4 | 2 | 12 | 312.774 | 23.010 | 81.182 | 42.381 | 19.074 |
| 1 | 3 | 8 | 296.255 | 25.140 | 90.339 | 53.994 | 19.335 |
| 2 | 3 | 4 | 266.700 | 22.639 | 76.363 | 42.949 | 14.695 |
| 3 | 3 | 7 | 274.261 | 21.293 | 78.405 | 35.972 | 13.349 |
| 4 | 3 | 18 | 198.870 | 16.003 | 51.356 | 26.727 | 12.501 |
| 1 | 4 | 11 | 249.628 | 21.679 | 70.667 | 40.003 | 16.475 |
| 2 | 4 | 20 | 271.808 | 20.771 | 74.926 | 39.239 | 15.351 |
| 3 | 4 | 16 | 206.145 | 17.846 | 59.196 | 35.309 | 13.228 |
| 4 | 4 | 15 | 266.045 | 36.140 | 67.984 | 43.144 | 18.332 |
| 1 | 5 | 23 | 245.389 | 19.784 | 57.160 | 41.354 | 16.056 |
| 2 | 5 | 24 | 279.773 | 23.759 | 71.956 | 34.352 | 15.516 |
| 3 | 5 | 17 | 259.107 | 22.699 | 71.062 | 47.724 | 17.403 |
| 4 | 5 | 19 | 245.157 | 23.435 | 72.289 | 37.650 | 17.177 |
| 1 | 6 | 22 | 260.068 | 21.643 | 74.852 | 33.615 | 17.082 |
| 2 | 6 | 1 | 254.961 | 19.521 | 75.228 | 39.171 | 16.518 |
| 3 | 6 | 14 | 290.791 | 22.137 | 74.977 | 30.970 | 16.114 |
| 4 | 6 | 21 | 291.614 | 22.510 | 81.801 | 45.690 | 16.803 |

Table E3. Summary of pre-treatment foliar micro-nutrient concentrations (ppm) by plot from a 22-year-old loblolly pine plantation in the Piedmont of Virginia.

| Block | Trt | Plot | Cu | Fe | B | Mn | Mo | S | Zn |
|-------|-----|------|------------|------|------|-------|-----|-------|-------|
| | | | <i>ppm</i> | | | | | | |
| 2 | 6 | 1 | 4.4 | 58.1 | 13.9 | 637.1 | 0.5 | 589 | 37.9 |
| 2 | 1 | 2 | 4.2 | 57.4 | 15.1 | 589.6 | 0.5 | 562.2 | 50.3 |
| 3 | 1 | 3 | 3.5 | 53.6 | 12.2 | 542.1 | 0.5 | 576.3 | 41.3 |
| 2 | 3 | 4 | 3.5 | 56.6 | 13.7 | 619.6 | 0.5 | 762.1 | 42.6 |
| 1 | 1 | 5 | 4.4 | 73.2 | 13.2 | 638.1 | 0.7 | 578.6 | 55.1 |
| 4 | 1 | 6 | 4.3 | 58.9 | 16.5 | 931.2 | 0.5 | 585.2 | 47.2 |
| 3 | 3 | 7 | 4.7 | 69.8 | 13.9 | 687.4 | 0.5 | 557.2 | 56.5 |
| 1 | 3 | 8 | 5.2 | 59.3 | 12.5 | 536 | 0.5 | 623.7 | 44.8 |
| 2 | 2 | 9 | 3.4 | 70 | 13.5 | 922.7 | 0.5 | 628.3 | 55.4 |
| 1 | 2 | 10 | 3.6 | 66.4 | 14 | 836 | 0.5 | 579.1 | 47.7 |
| 1 | 4 | 11 | 3.1 | 59.8 | 17.4 | 744.1 | 0.5 | 637.4 | 43.6 |
| 4 | 2 | 12 | 2.8 | 63.7 | 14 | 689.3 | 0.5 | 600.4 | 37.3 |
| 3 | 2 | 13 | 3.4 | 67.6 | 18.1 | 613.4 | 0.5 | 650.3 | 57.1 |
| 3 | 6 | 14 | 3.8 | 80 | 20.1 | 495 | 0.5 | 479.6 | 50.2 |
| 4 | 4 | 15 | 4 | 61.2 | 14 | 510.6 | 0.5 | 466.9 | 56.2 |
| 3 | 4 | 16 | 4.2 | 57.2 | 12.4 | 685 | 0.5 | 558.6 | 58.1 |
| 3 | 5 | 17 | 2.9 | 56.7 | 14.7 | 651.5 | 0.7 | 577.2 | 49.2 |
| 4 | 3 | 18 | 2.9 | 63.8 | 18 | 601.7 | 0.5 | 494.5 | 43.6 |
| 4 | 5 | 19 | 3.6 | 67.6 | 16.6 | 372.8 | 0.6 | 552.7 | 47.9 |
| 2 | 4 | 20 | 2.8 | 57.1 | 12.7 | 444 | 0.5 | 499 | 46 |
| 4 | 6 | 21 | 2.8 | 57 | 18.2 | 629.6 | 0.5 | 504.3 | 45.6 |
| 1 | 6 | 22 | 2.6 | 75.5 | 17.1 | 441.5 | 0.5 | 598.8 | 100.3 |
| 1 | 5 | 23 | 3.6 | 61.2 | 17.2 | 551.3 | 0.5 | 539.9 | 40.6 |
| 2 | 5 | 24 | 5 | 65.9 | 12.6 | 545.3 | 0.5 | 499.8 | 41.4 |

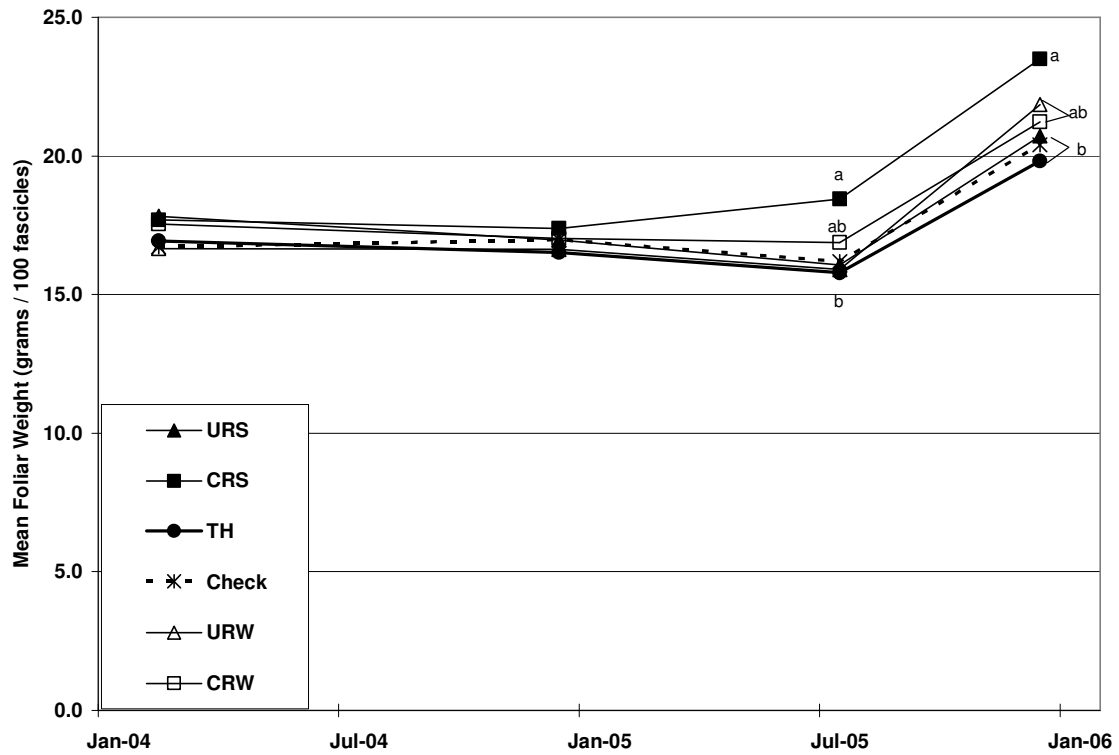


Figure E1. Mean foliar weight (g / 100 fascicles) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

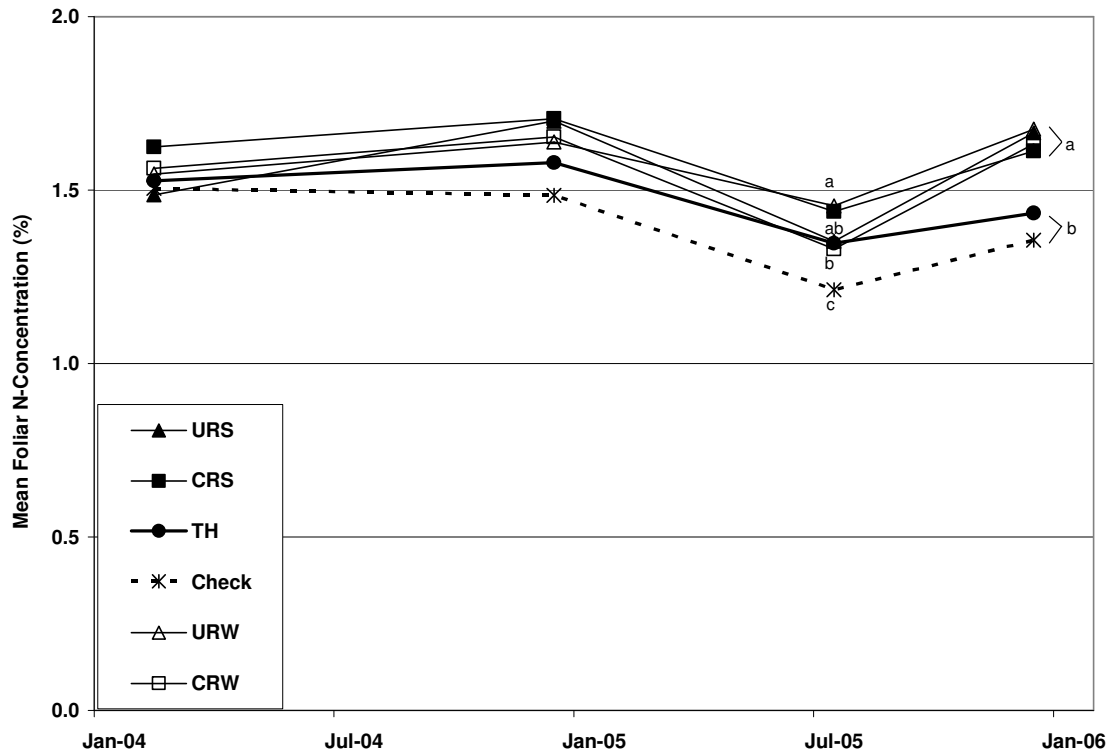


Figure E2. Mean foliar N concentration (%) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

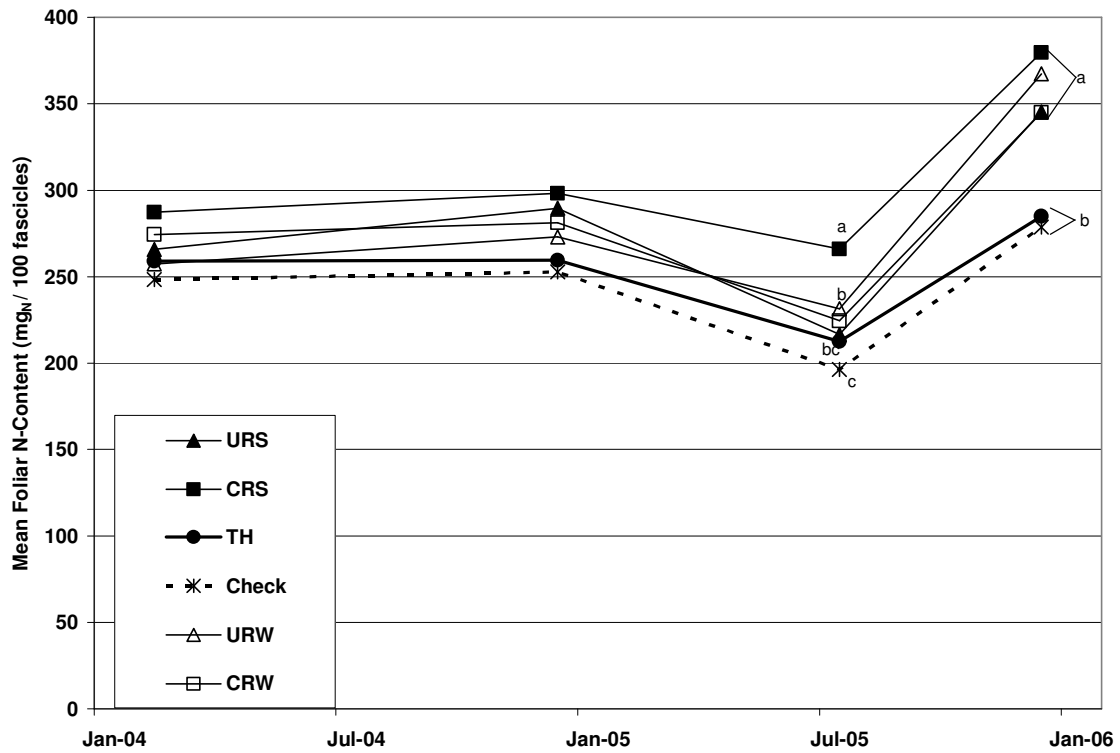


Figure E3. Mean foliar N content (mg / 100 fascicles) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

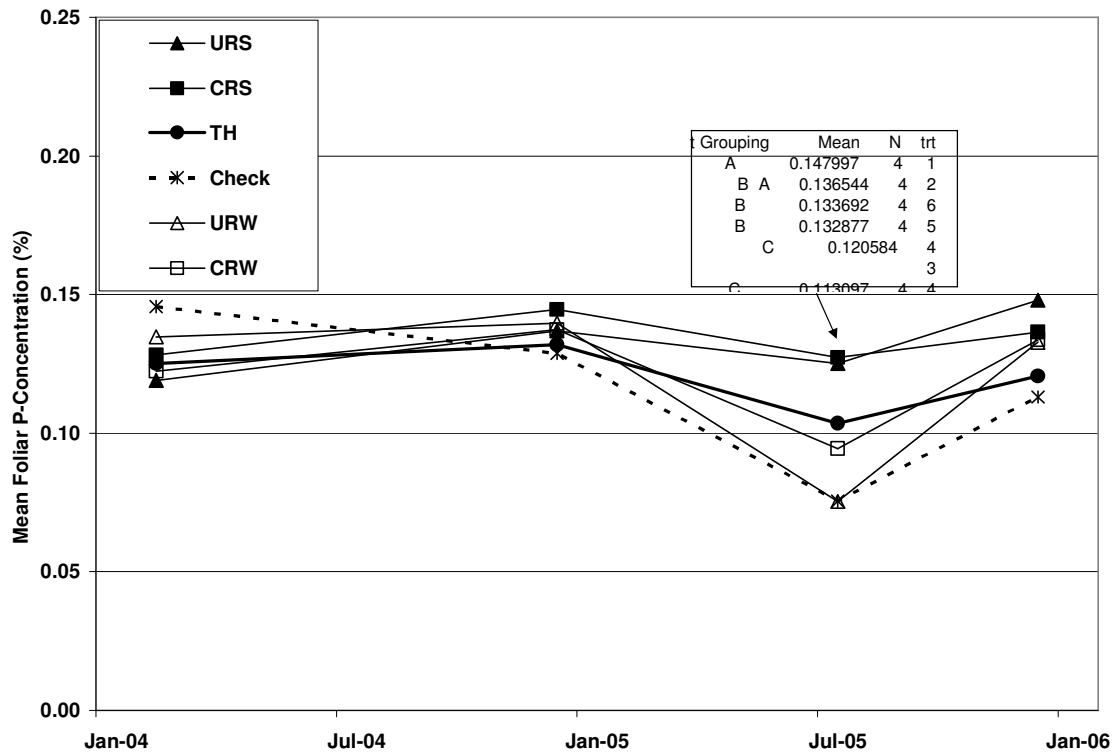


Figure E4. Mean foliar P concentration (%) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

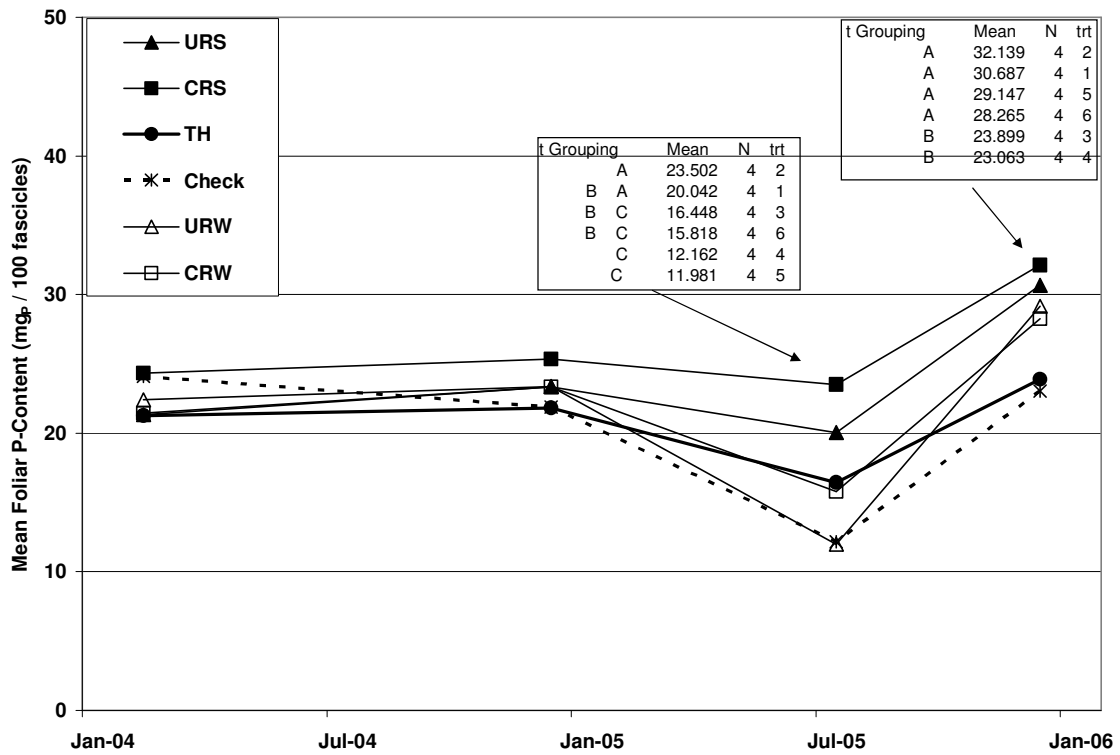


Figure E5. Mean foliar P content (mg / 100 fascicles) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

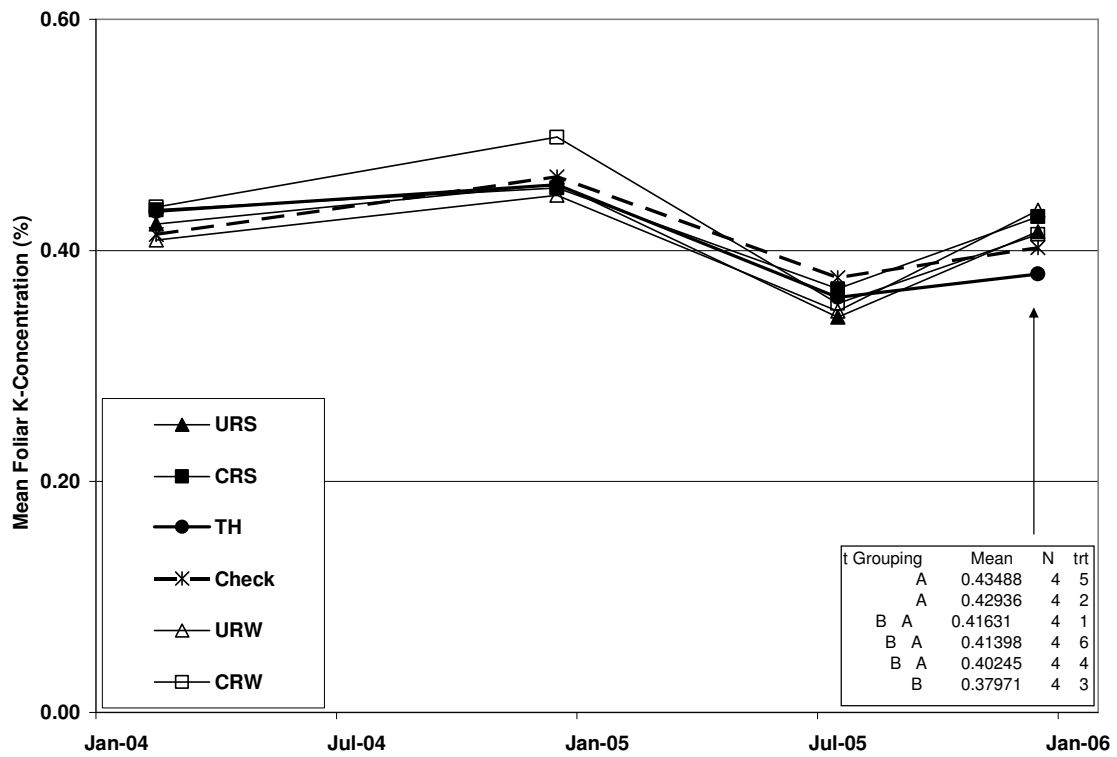


Figure E6. Mean foliar K concentration (%) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

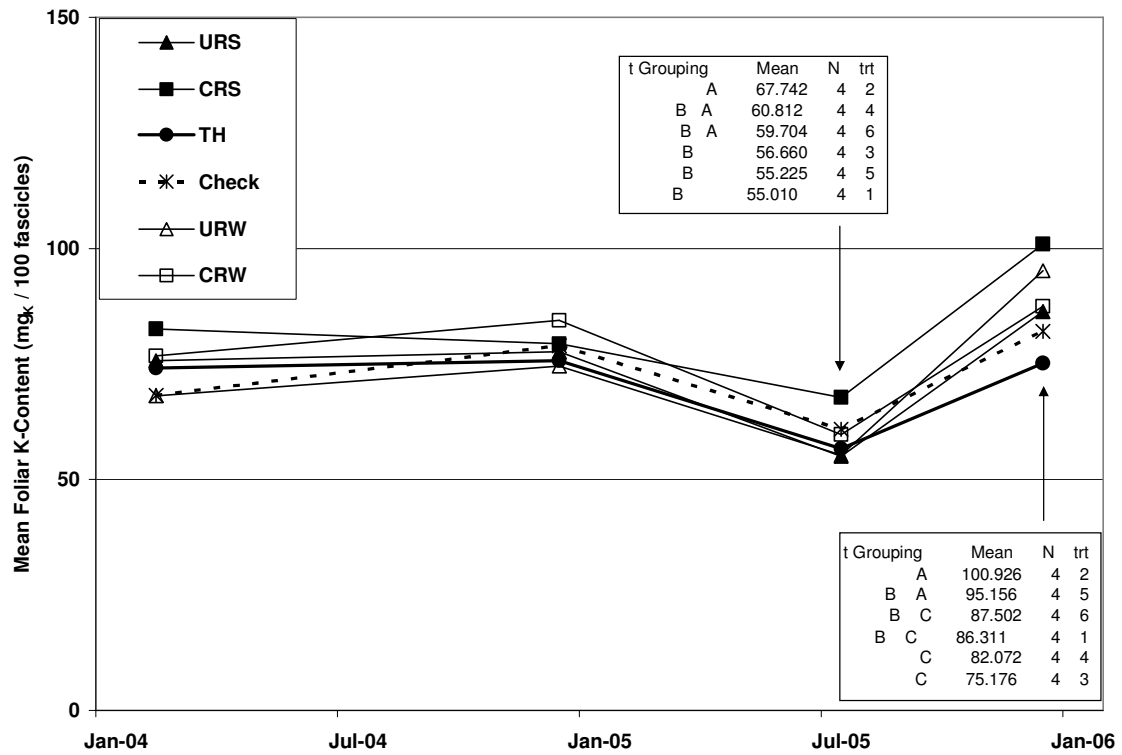


Figure E7. Mean foliar K content (mg / 100 fascicles) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

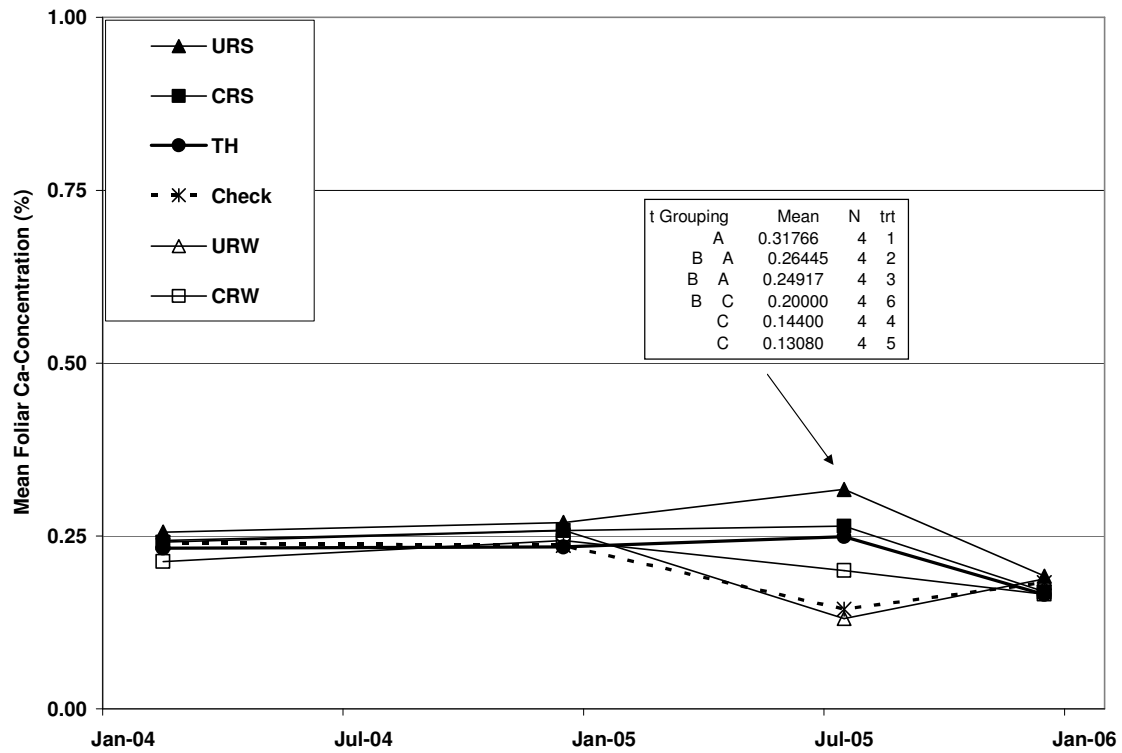


Figure E8. Mean foliar Ca concentration (%) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

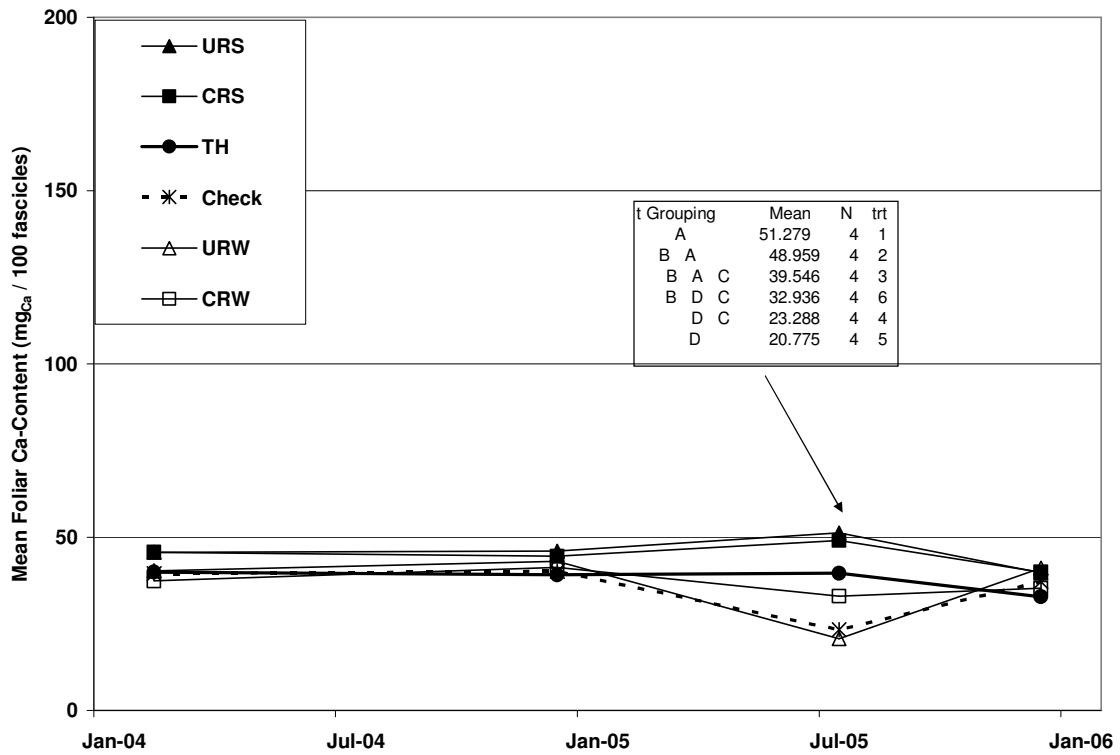


Figure E9. Mean foliar Ca content (mg / 100 fascicles) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

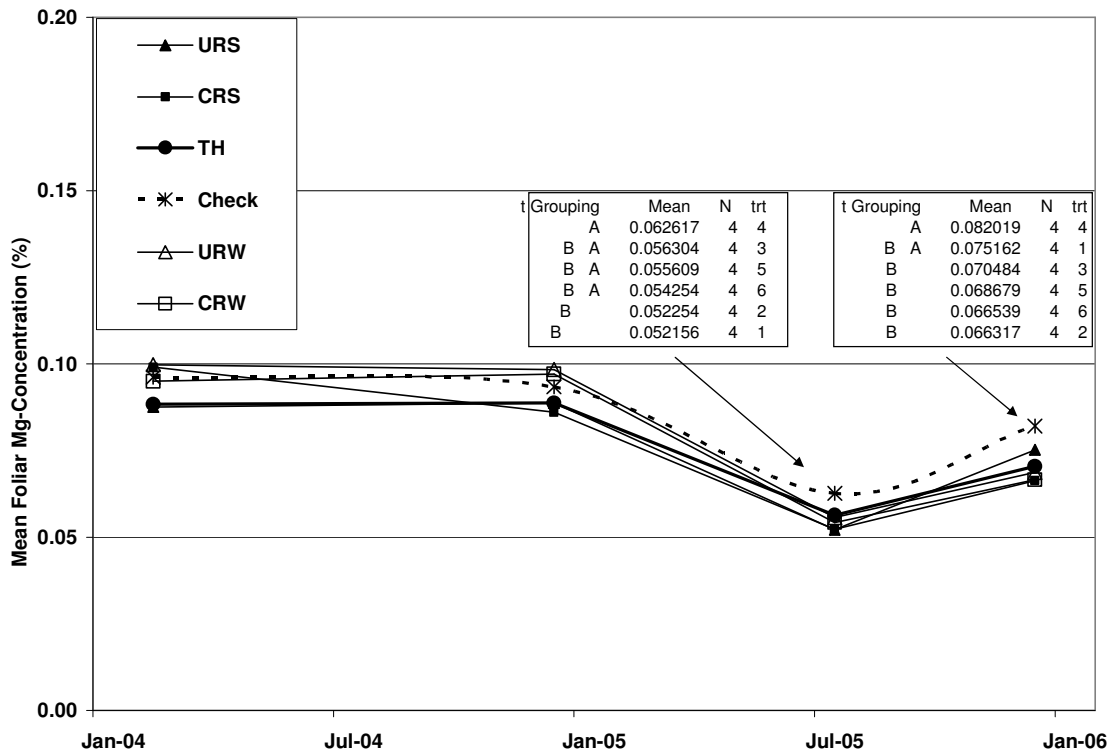


Figure E10. Mean foliar Mg concentration (%) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

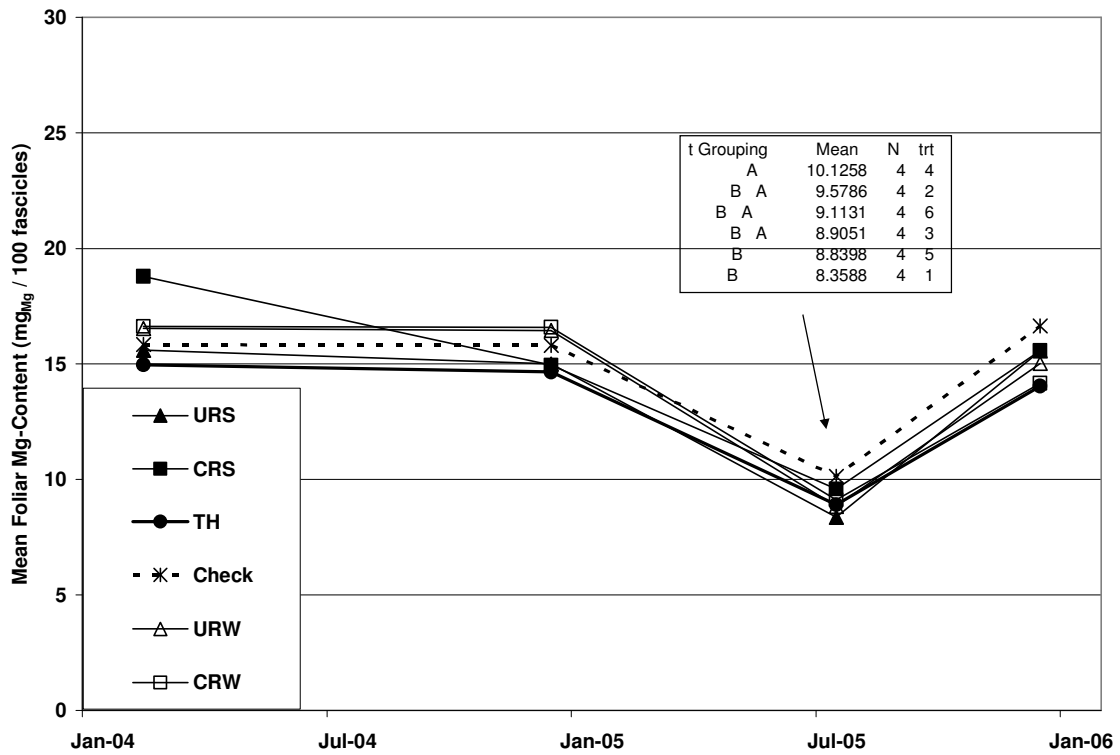


Figure E11. Mean foliar Mg content (mg / 100 fascicles) results taken from four sampling periods in 22-year-old loblolly pine stand in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹. Means with different letters indicate significant differences at the $\alpha < 0.05$ level.

APPENDIX F.
LITTERFALL WEIGHT AND NUTRIENT CONCENTRATION RESULTS
FROM A MID-ROTATION LOBLOLLY PINE PLANTATION IN THE
VIRGINIA PIEDMONT FOLLOWING THINNING AND FERTILIZATION
WITH UREA OR CONTROLLED RELEASE NITROFORM® APPLIED AT A
RATE OF 224 KG-N HA⁻¹

Table F1. Litterfall weight (g / 100 fascicles) and macro-nutrient concentration (%) by treatment results after two collection periods in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

| Trt. | Weight (g / 100 fascicles) | | N (%) | | P (%) | | K (%) | | Ca (%) | | Mg (%) | |
|-------|-------------------------------|-------|-------|-------|-------|-------|-------|-------|--------|-------|--------|-------|
| | 2004 | 2005 | 2004 | 2005 | 2004 | 2005 | 2004 | 2005 | 2004 | 2005 | 2004 | 2005 |
| Check | 14.39 | 13.48 | 0.451 | 0.413 | 0.030 | 0.029 | 0.069 | 0.064 | 0.410 | 0.410 | 0.067 | 0.062 |
| TH | 14.34 | 12.72 | 0.518 | 0.518 | 0.040 | 0.040 | 0.077 | 0.058 | 0.392 | 0.373 | 0.062 | 0.052 |
| URS | 14.10 | 12.82 | 0.612 | 0.581 | 0.049 | 0.052 | 0.092 | 0.055 | 0.388 | 0.395 | 0.060 | 0.052 |
| CRS | 14.57 | 12.43 | 0.871 | 0.534 | 0.065 | 0.047 | 0.138 | 0.065 | 0.370 | 0.367 | 0.064 | 0.049 |
| URW | 14.43 | 13.64 | 0.475 | 0.561 | 0.035 | 0.044 | 0.056 | 0.076 | 0.400 | 0.358 | 0.066 | 0.054 |
| CRW | 14.48 | 13.52 | 0.573 | 0.568 | 0.041 | 0.051 | 0.095 | 0.061 | 0.366 | 0.344 | 0.070 | 0.057 |

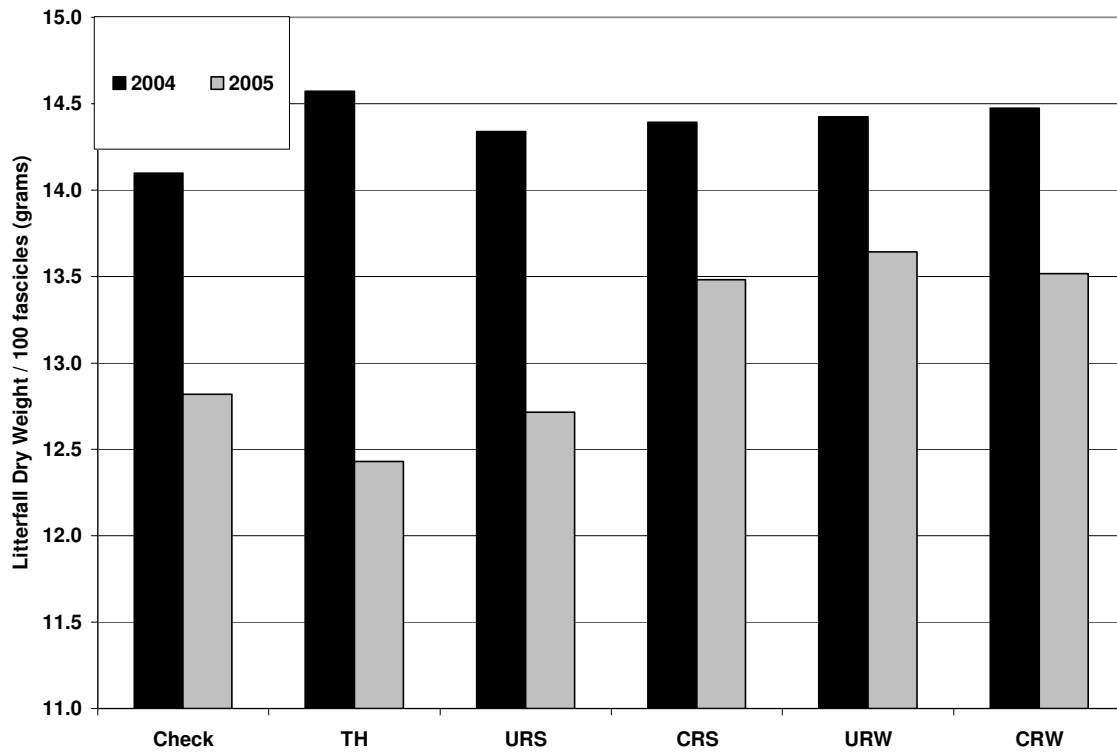


Figure F1. Litterfall dry weight (g / 100 fascicles) by treatment after two collection periods in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

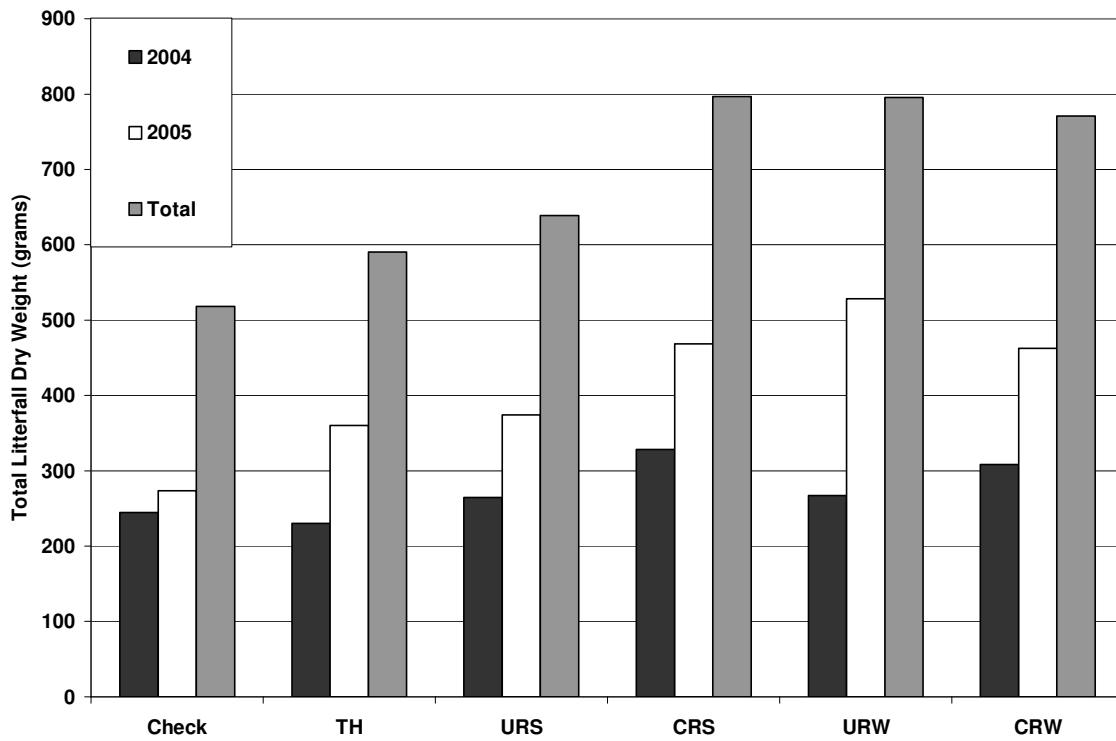


Figure F2. Total collected litterfall dry weight (grams) by treatment after two collection periods in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

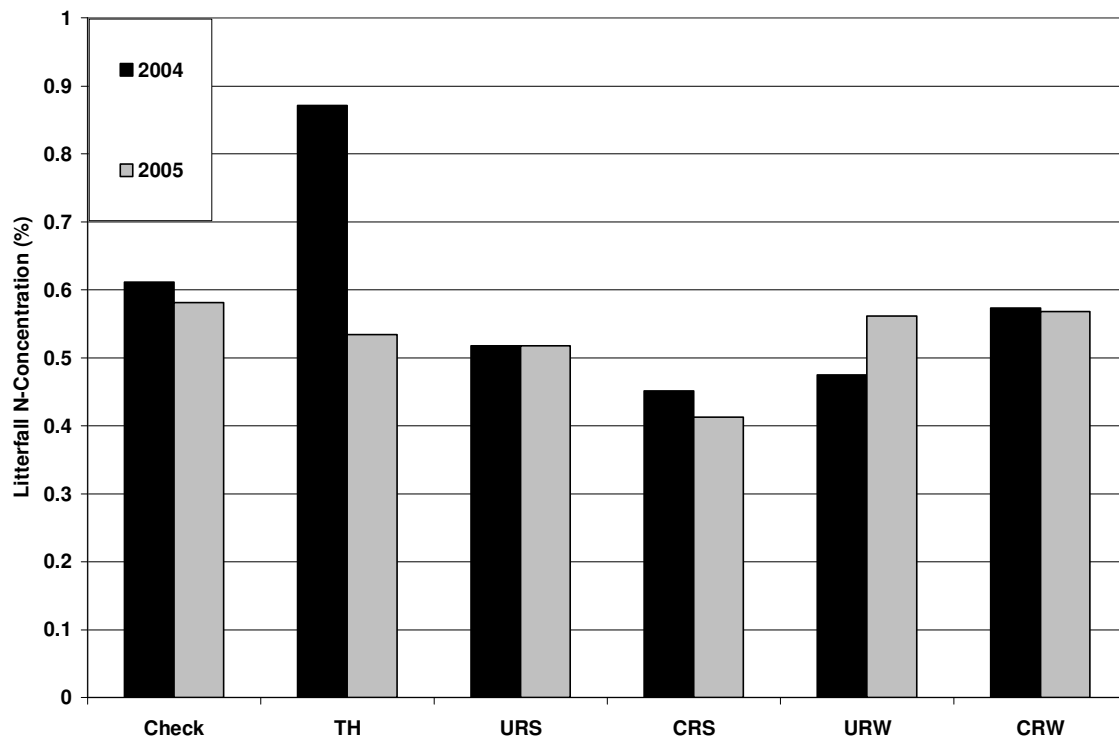


Figure F3. Litterfall N concentration (%) by treatment after two collection periods in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

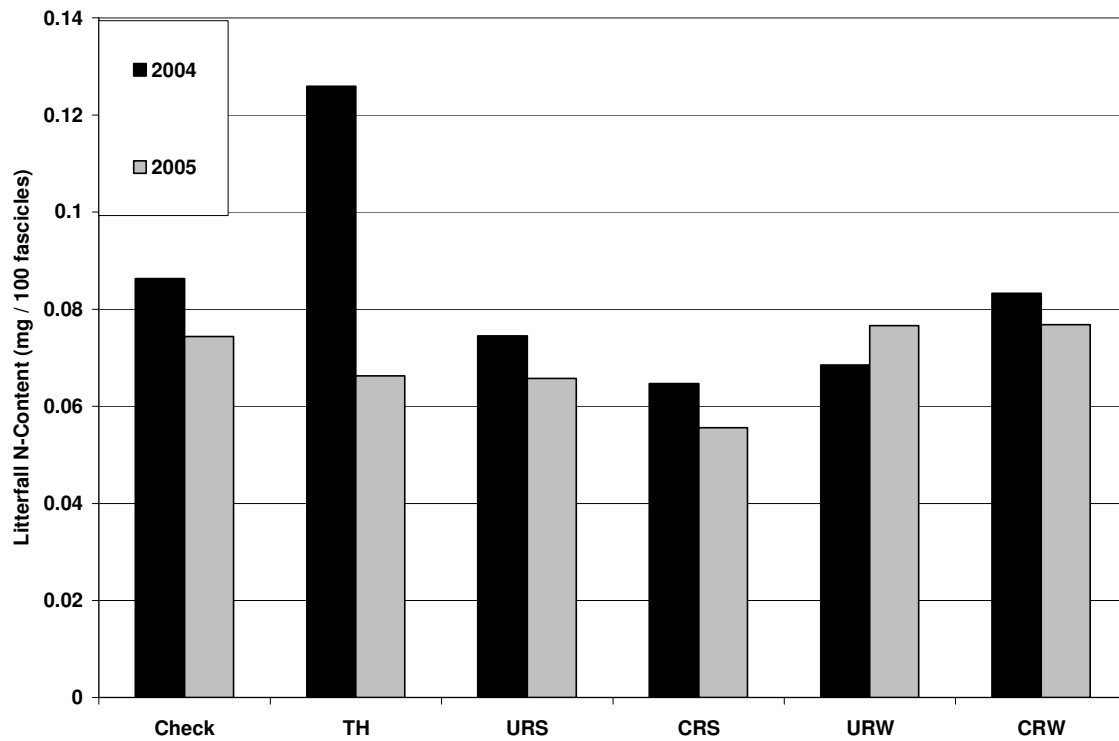


Figure F4. Litterfall N content (mg / 100 fascicles) by treatment after two collection periods in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

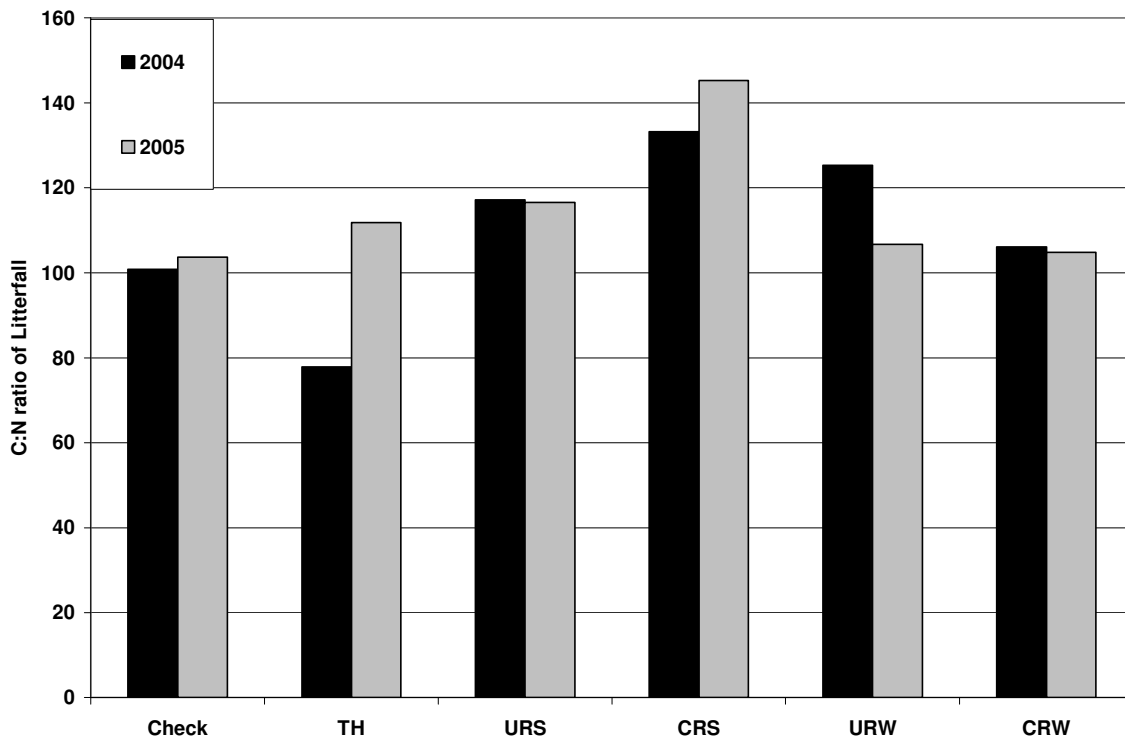


Figure F5. Litterfall C:N ratio by treatment after two collection periods in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea or controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

APPENDIX G.

LEAF AREA INDEX RESULTS FROM A MID-ROTATION LOBLOLLY PINE PLANTATION IN THE VIRGINIA PIEDMONT FOLLOWING THINNING AND FERTILIZATION WITH UREA OR CONTROLLED RELEASE NITROFORM® APPLIED DURING THE SUMMER AT A RATE OF 224 KG-N HA⁻¹

Table G1. Mean Leaf Area Increment (LAI) by treatment as measured by litterfall collections after two growing seasons in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea and controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

| Treatment | 2004 Litterfall LAI estimate | 2005 Litterfall LAI estimate | Total Litterfall LAI estimate |
|------------------|-------------------------------------|-------------------------------------|--------------------------------------|
| Check | 1.43 | 1.57 | 2.99 |
| TH | 1.34 | 2.06 | 3.41 |
| URS | 1.54 | 2.14 | 3.69 |
| CRS | 1.91 | 2.68 | 4.60 |
| URW | 1.56 | 3.03 | 4.58 |
| CRW | 1.80 | 2.65 | 4.45 |

Table G2. Mean Leaf Area Increment (LAI) by treatment as measured by Hemiview® and hemispherical canopy photography after two growing seasons in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea and controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

| Treatment | Date of Photo Acquisition | | | | | |
|------------------|----------------------------------|-----------------|------------------|------------------|------------------|-----------------|
| | <i>3/23/2004</i> | <i>7/1/2004</i> | <i>8/18/2004</i> | <i>1/13/2005</i> | <i>9/21/2005</i> | <i>1/4/2006</i> |
| | LAI | | | | | |
| Check | 2.09 | 2.11 | 2.05 | 2.00 | 2.19 | 1.72 |
| TH | 1.87 | 2.18 | 1.80 | 1.94 | 1.93 | 1.87 |
| URS | 1.89 | 2.11 | 1.91 | 1.88 | 1.98 | 1.87 |
| CRS | 2.08 | 2.06 | 1.79 | 1.71 | 1.91 | 1.78 |
| URW | 1.99 | 2.17 | 1.77 | 1.65 | 2.03 | 1.84 |
| CRW | 1.96 | 2.13 | 1.71 | 1.75 | 1.93 | 1.83 |

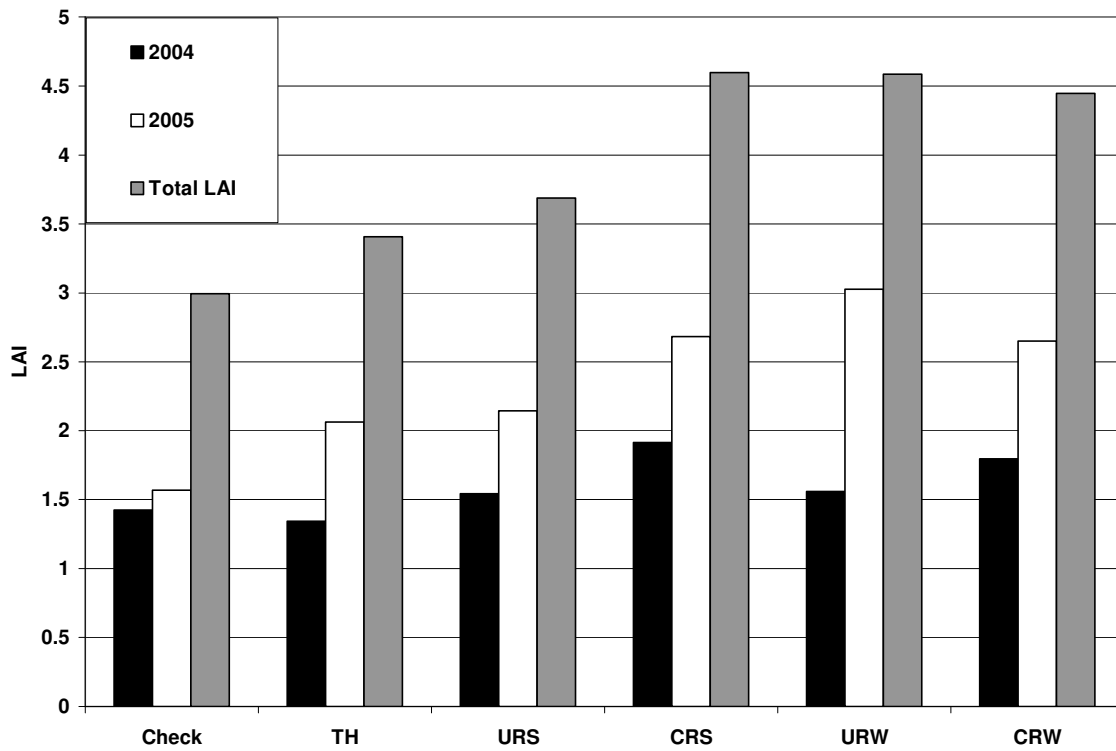


Figure G1. Mean Leaf Area Increment (LAI) by treatment as measured by Hemiview collections after two growing seasons in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea and controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

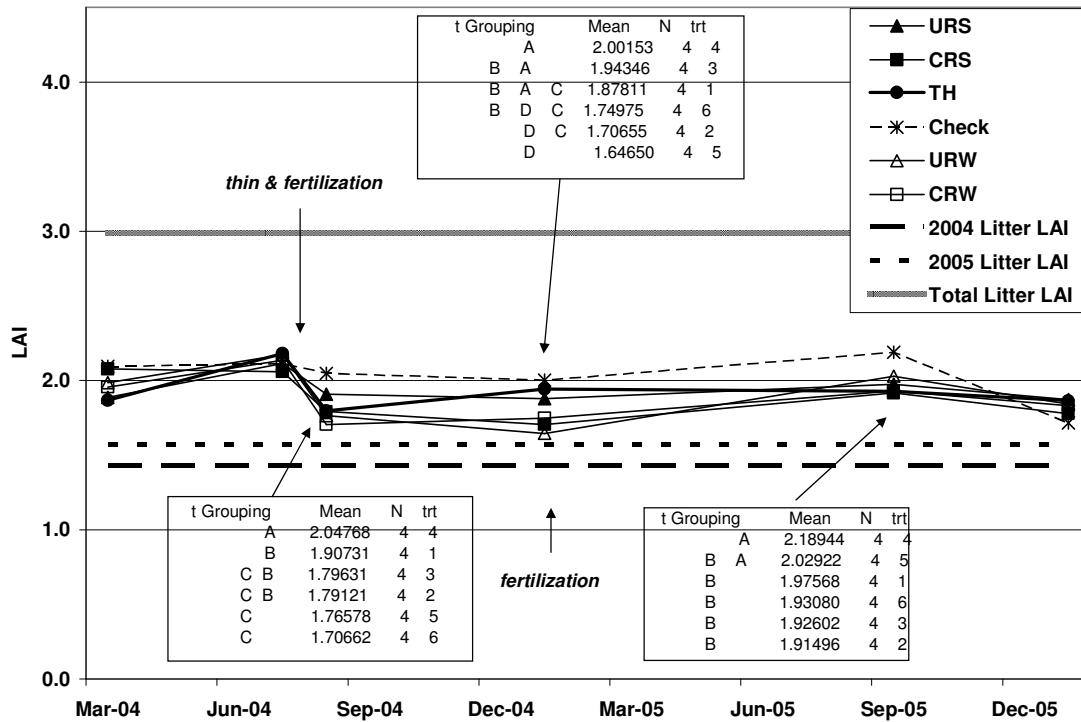


Figure G2. Comparison of mean Leaf Area Increment (LAI) by treatment as measured by Hemiview® hemispherical canopy photography and mean LAI of all treatment plots as measured by litterfall collections after two growing seasons in a loblolly pine plantation in the Virginia Piedmont after thinning and fertilization with urea and controlled release Nitroform® at the rate of 224 kg-N ha⁻¹.

VITA

James Robertson Elliot was born in Rocky Mount, North Carolina. He attained a Bachelor's of Science degree in Biology, with a minor in Chemistry from the University of North Carolina at Chapel Hill in May of 1999. After graduating, he worked for a variety of forest management and research institutions encompassing a wide range of ecosystems including the Pacific northwest temperate rainforest for Oregon State University and Mt. Hood National Forest, Sequoia National Park in the Sierra Nevada for Duke University, the Canadian boreal forest and Amazon rainforest for the University of California at Irvine, and the Rocky Mountain montane forests for the Arapahoe-Roosevelt National Forest. He has been happily married to his wife Andi since 2005 and is blessed with a supportive family, especially, mother and father, Ginger and Jay Elliot, two older sisters, Laura Elliot and Kristi Strickland, mother-in-law and father-in-law, Cindy and Van Dotson Sr., and brother-in-law Van Dotson Jr..