Nutrient Retention and Cycling in Southeastern U.S. Loblolly Pine (*Pinus taeda* L.) and Sweetgum (*Liquidambar styraciflua* L.) Plantations

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Abstract

Forest plantations in the southeastern U.S. are fertilized to increase growth on infertile, sandy soils. Nitrogen (N) and phosphorus (P) are the most common growth limiting nutrients. A key question that arises following fertilization of these soils is whether the applied fertilizer benefits only the current trees in the stand or also improves long-term site quality. The objectives of this study were to compare accumulation of N and P in the forest floor and mineral soil among unfertilized and fertilized plantations, determine soluble and residual N and P fractions and soluble carbohydrate and phenol fractions in foliage and litter, determine whether higher N in the forest floor from fertilization resulted in increased release of N from the forest floor and increased mineral soil N availability, and determine loblolly pine forest floor decomposition rate and release of nutrients in a simulated disturbance environment. Research was conducted at a 25-year old loblolly pine (*Pinus taeda* L.) plantation in NC (SETRES) and 13-year old loblolly pine and sweetgum (*Liquidambar styraciflua* L.) plantations in GA (Mt. Pleasant). Fertilization resulted in increases in mineral soil N that were likely to be temporary and not sustained following cessation of fertilization N applications. This was likely due to an inability of acidic, sandy mineral soils to retain NH_4-N and NO_3-N. The forest floor accumulated N due to slow release of N during decomposition. Fertilization with N results in only temporary increases in mineral soil N availability that occur during

fertilizer application and from forest floor decomposition. Future changes in N availability are primarily determined by decomposition of the forest floor following a disturbance that accelerates decomposition. In contrast to N, fertilization of loblolly pine and sweetgum with P results in a long-term increase in site P availability. Fertilization with P has lasting effects by increasing mineral soil P in stable forms that can be made available for plant uptake over time suggesting increased supply of P to trees in the next rotation. Retention of P in the mineral soil was likely due to the tendency of acidic, sandy mineral soils to accumulate P in Al- and Fe-phosphates.

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Chapter 1
Introduction

1.1. Justification

Fertilization of forest plantations is widely practiced in the southeastern U.S. to improve growth on infertile soils. Most soils supporting forest plantations are both nitrogen (N) and phosphorus (P) limited (Fox et al., 2007a). Additions of 225 kg N ha^{-1} and 28 kg P ha^{-1} have been shown to result in large and consistent volume increases of approximately 25% for loblolly pine (*Pinus taeda* L.) that last for 6 to 10 years following fertilization (Fox et al., 2007b). Physiological changes in loblolly pine following fertilization include a temporary increase in photosynthetic capacity resulting in increased leaf area and foliar nutrients (Gough et al., 2004). Albaugh et al. (1998) found that these increases were accompanied by productivity gains and a reduction in rotation length. Fertilization of sweetgum (*Liquidambar styraciflua* L.) on nutrient limited soils has been found to result in similar foliar and productivity responses (Scott et al., 2004; Cobb et al., 2008).

While fertilization with N and P is successful in increasing tree growth on infertile, sandy soils typical of the southeastern U.S., a key question that arises from fertilization of these soils is whether the applied fertilizer benefits only the current trees in the stand or also improves long-term site quality. Miller (1981) suggests that N fertilization of pine on a sandy soil will only benefit the trees and will not increase mineral soil N. Instead, N accumulates only in the trees and the forest floor resulting in a transitory increase in tree growth. Unlike N, fertilization with P has been shown to increase mineral soil P often resulting in a decades-long increase in tree growth (Prichett and Comerford, 1982; Fox et al. 2011).

The forest floor N sink is suggested to result from loblolly pine litter accumulating N as it decomposes (Piatek and Allen, 2001; Sanchez, 2001). While Piatek and Allen (2001) and Sanchez (2001) suggest loblolly pine litter also accumulates P as it decomposes, Polglase et al. (1992a) found that this litter readily releases P through leaching while essentially no N leaches. As much as 15% of total litter P leached in 5 days as inorganic-P. In another study assessing loblolly pine litter P, Polglase et al. (1992b) found that fertilization increases litter P in a soluble fraction. Yuan (1960) suggests acidic, sandy soils retain approximately 80% of added P as Al- and Fe-phosphates. Retention of P was found to increase linearly with exchangeable Al and Fe and was highest in Ultisols (Ballard and Fiskell, 1974). However, P sorption of approximately 87 ppm did occur in sandy textured Entisols in the 0-20 cm depth.

Results of these studies suggest accumulations of N primarily in the forest floor and accumulations of P in the mineral soil that are related to the release of N and P from litter and mineral soil properties. These studies also suggest that long-term site quality will be improved by increasing mineral soil P but not N. Considering these findings, research that investigates these responses on additional sites is warranted. Two forest plantation sites in the southeastern U.S. with similar soils that received similar total N and P applications were evaluated. One site is a 25-year old loblolly pine plantation while the other is a 13-year old loblolly pine and sweetgum plantation. Our overall objective was to determine whether fertilization improves long-term site quality by increasing long-term mineral soil N and P availability. Subsequent objectives were developed to further investigate specific N and P cycling responses of the sites to fertilization.

1.2. Major Objectives

The major objectives of this research are to:

1. Compare accumulation of nitrogen and phosphorus in the forest floor and mineral soil among unfertilized and fertilized plantations.
2. Determine soluble and residual nitrogen and phosphorus fractions and soluble carbohydrate and phenol fractions in foliage and litter.
3. Determine whether higher nitrogen in the forest floor from fertilization resulted in increased release of nitrogen from the forest floor and increased mineral soil nitrogen availability.
4. Determine loblolly pine forest floor decomposition and release of nutrients in a simulated disturbance environment.
5. Compare nitrogen and phosphorus accumulation and cycling among loblolly pine and sweetgum.

1.3. Hypotheses Tested

Research objectives were met by testing the following null hypotheses:

1. H_o: Fertilization does not increase mineral soil nitrogen and phosphorus.
2. H_o: Fertilization does not alter residual and soluble nitrogen and phosphorus fractions in foliage and litter.

3. H_o: Higher nitrogen in the forest floor does not result in increased release of nitrogen from the forest floor and elevated mineral soil nitrogen availability.

4. H_o: Increased endogenous nitrogen availability from fertilization does not increase the rate of decomposition and nutrient release from the loblolly pine forest floor.

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Chapter 2
Nitrogen and phosphorus retention in soils of fertilized loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) plantations

Abstract

Forest plantations in the southeastern U.S. are fertilized to improve growth on infertile, sandy soils. A key question that arises following fertilization of these soils is whether the applied fertilizer benefits only the current trees in the stand or also improves long-term site quality. Long-term site quality could improve if fertilization increased mineral soil N and P availability. However, if the impact of fertilization is only an increase in forest floor N and P, an improvement in long-term site quality is less likely since the forest floor rapidly decomposes following disturbance. Forest floor and mineral soil N and P pools were measured at a 23-year old loblolly pine (*Pinus taeda* L.) plantation in NC (SETRES) and 12-year old loblolly pine and sweetgum (*Liquidambar styraciflua* L.) plantations in GA (Mt. Pleasant) growing on sandy soils. Total N and P applications at SETRES from 1992 to 2008 were 1378 kg ha^{-1} and 168 kg ha^{-1}, respectively. Total N and P applications at Mt. Pleasant from 1997 to 2006 were 1128 kg ha^{-1} and 169 kg ha^{-1}, respectively. Fertilization increased mineral soil N at SETRES by 199 kg ha^{-1}. Mineral soil N was not increased by fertilization at the Mt. Pleasant site where fertilizer application ended 3 years prior to sampling. Results indicated that the increase in mineral soil N at SETRES was in a labile pool, was temporary, and would not likely be sustained upon cessation of N fertilization. Fertilization increased forest floor N at both sites. Loblolly pine plantations were found to retain only 50% of added N while sweetgum retained 100% of added N. In contrast to N, fertilization increased loblolly pine mineral soil P at SETRES by 56 kg ha^{-1}. Mineral soil P was elevated at Mt. Pleasant

from past fertilizer applications. Results suggest fertilization increases a mineral soil P pool with the potential to improve long-term site quality by supplying higher quantities of P to the next rotation. Relative to P, N fertilization results in short-term rather than long-term increases in N availability.

2.1. Introduction

Fertilization of forest plantations is widely practiced in the southeastern U.S. to improve growth on infertile soils (Albaugh et al., 2007). Most of these soils are both nitrogen (N) and phosphorus (P) limited. Fertilization with 225 kg N ha^{-1} and 28 kg P ha^{-1} have been shown to result in large and consistent growth increases of approximately 25% for loblolly pine (*Pinus taeda* L.) that last for 6 to 10 years following fertilization (Fox et al., 2007). Physiological changes in loblolly pine following fertilization include increased foliar nutrient concentrations and a temporary increase in photosynthetic capacity that produces additional photosynthate used to increase leaf area (Gough et al., 2004) producing a growth response in fertilized trees (Albaugh et al., 1998). Similar foliar and productivity responses have been observed for sweetgum (*Liquidambar styraciflua* L.) following fertilization on nutrient limited soils (Scott et al., 2004; Cobb et al., 2008).

While fertilization with N and P increases tree growth on infertile, sandy soils typical of the southeastern U.S., a key question that arises following fertilization of trees on these soils is whether the applied fertilizer benefits only the current trees in the stand or also improves long-term site quality. Long-term site quality could improve if fertilization increased mineral soil N and P and long-term N and P availability.

Miller (1981) suggests that N fertilization only benefits the trees because there is generally little impact of N fertilization on the mineral soil. Rather, according to the hypothesis of Miller (1981) the forest floor rather than the mineral soil accumulates N following fertilization. Piatek and Allen (2001) and Sanchez (2001) suggest the forest floor is a sink for N following fertilization due to accumulation of N during decomposition. Due to forest floor accumulation, N availability gradually declines through time and a deficiency develops. The N accumulated in the forest floor is released as the forest floor decomposes following disturbance resulting in the Assart effect (Kimmins, 1997). However, the Assart effect usually occurs following harvest when tree nutrient demand is small. Nitrogen availability declines following the Assart flush of nutrients and nutrient deficiencies then begin to develop in the stand (Fox et al., 2007).

Fertilization also increases forest floor P (Harding and Jokela, 1994), however, much of the P in the forest floor is readily leached (Polglase et al., 1992). As much as 15% of total litter P was leached in 5 days as inorganic-P while no N was leached (Polglase et al., 1992). Unlike N, P fertilization is proposed to also benefit the site by increasing mineral soil P leading to a long-term increase in P availability (Prichett and Comerford, 1982; Fox et al., 2011). A single application of P has been found to increase tree growth throughout the rotation and even in subsequent rotations across a range of soils and tree species (Ballard, 1978; Gentle et al., 1986; Comerford et al., 2002; Crous et al., 2007; Everett and Palm-Leis, 2009).

Our objective was to compare accumulation of N and P in the forest floor and mineral soil among unfertilized and fertilized plantations. Based on findings in the literature, we hypothesized that fertilization would primarily result in N accumulations in the forest floor with little N accumulation in the mineral soil. In contrast to N, fertilization would increase P in both the forest floor and mineral soil. To test the hypothesis, forest floor and mineral soil N and P concentration and content were determined at two sites in the southeastern U.S. with similar total N and P applications and mineral soil properties.

2.2. Materials and Methods

2.2.1. Site Descriptions

SETRES

The Southeast Tree Research and Education Site (SETRES) was established in 1985 in order to investigate the effects of irrigation and fertilization on loblolly pine growing on an infertile, excessively-drained sandy soil. The site was located in the Sandhills of Scotland County, NC 17 km north of Laurinburg, NC (34° 54′N, 79° 28′W). Mean annual precipitation was 120 cm and the mean minimum and maximum temperatures were 10.4 and 23.6 $^{\circ}$C, respectively. Prior to planting of loblolly pine in 1985, the site was a native longleaf pine (*Pinus palustris* Mill.) forest. The stand was 23-years old when sampling began for this study.

The soil was mapped as the Wakulla series; an infertile, excessively drained, sandy, siliceous, thermic Psammentic Hapludult. While the soil was mapped as a Hapludult, the clay lenses giving this designation were weakly expressed. The A-horizon extends to 10-15 cm with a weak Bt horizon at ~60 cm followed by multiple C horizons. Select mineral soil properties can be found in Table 2.1.

The experimental design at SETRES was a 2^2 factorial randomized complete block with 4 blocks (n=4, N=16) with fertilization and irrigation treatments. In 1992, 50 x 50 m treatment plots containing interior 30 x 30 m measurement plots were established. Prior to initial treatment application, plots were thinned and competing vegetation was controlled. Treatments included fertilization (no addition and optimal foliar nutrition based on target foliar nutrient concentration) and irrigation (no addition and addition of 2.5 cm water week^{-1} March to November). Treatment levels included a control (Ct), irrigation (I), fertilization (F), and fertilization x irrigation (FI). Fertilization was conducted by applying a balance of macro- and micro-nutrients to provide optimal foliar nutrition and irrigation was conducted to maintain a soil water content >40% field capacity. Fertilization began in March 1992 and irrigation began in April 1993 and continues to the present. Fertilization was conducted each spring with a broadcast surface application of various fertilizer sources including granular urea, boron-coated urea, ammonium sulfate, diammonium phosphate, triple super phosphate, potassium chloride, dolomitic lime, Epsom salts, calcium sulfate, and borax (Albaugh et al., 2008). Total N and P applications from 1992 to 2008 were 1378 kg ha^{-1} and 168 kg ha^{-1}, respectively (Table 2.2). Nutrient additions significantly increased tree growth with volume increased by 100% after 13 yrs. of fertilization (Albaugh et al., 1998; Albaugh et al., 2004).

Mt. Pleasant, GA

A similar study designed to evaluate the effects of irrigation and fertilization on a number of pine and hardwood species was established in Mt. Pleasant, GA on an infertile, excessively-drained sandy soil. The site was located in the southeastern Georgia coastal plain in Wayne Co. (31°23′N, 81°43′W). Mean annual precipitation was 127 cm and the mean minimum and maximum temperatures were 12.0 and 26.0 °C, respectively. Prior to establishment of the study in 1997, the site was a slash pine (*Pinus elliottii* Engelm.) plantation. After harvesting of the previous pine stand, the site was cleared, disked, limed, and planted with separate plots containing loblolly pine, slash pine, sweetgum, and sycamore (*Platanus occidentalis* L.). This paper includes data from only the loblolly pine and sweetgum plots. The stands were 12-years old when sampling began for this study.

The soil was mapped as the Klej series; a mesic, coated Aquic Quartzipsamment. Due to site preparation, the Ap horizon extended to approximately 25 cm. Below the Ap horizon were a series of C horizons. A poorly developed spodic horizon occurred in some locations. Select mineral soil properties can be found in Table 2.1.

The study was a randomized complete block design with 3 blocks (n=3, N=18). In 1997, 35 x 25.5 m treatment plots containing interior 22.2 x 13.5 m measurement plots were established. Treatment levels included a control (Ct), irrigation (I), and 3 levels of fertilization combined with irrigation (FI). Only the high fertilization rate was evaluated in this study where annual nutrient additions averaged 113 N, 17 P, and 62 K kg ha^{-1} yr^{-1} (Cobb et al., 2008). Fertilization and irrigation treatments began in 1997. Irrigation was conducted by applying 3.05 cm water week^{-1} for 28 weeks year^{-1} (April to November) from 1997 to 2003. Fertilization from 1997 to 2003 was conducted by applying nutrients in solution in the irrigation water throughout the 28 weeks of irrigation each year. Nitrogen was supplied as ammonium nitrate (NH_4NO_3). From 2004 to 2006, surface applications of N, P, and micronutrients were conducted in the spring. Nitrogen and P were applied as diammonium phosphate (DAP) and urea. Total N and P applications from 1997 to 2006 were 1128 kg ha^{-1} and 169 kg ha^{-1} (Table 2.3), respectively, which were comparable to total fertilizer N and P applied at SETRES. Nutrient additions increased aboveground biomass (Cobb et al., 2008). After 9 yrs. of fertilization, loblolly pine volume increased by 30% and sweetgum volume increased by 300%.

2.2.2. Sampling

The forest floor and mineral soil were sampled in April 2008 (SETRES) and March 2009 (Mt. Pleasant). At 18 (SETRES) and 15 (Mt. Pleasant) random locations within each plot, a 2 cm diameter punch tube was used to sample to a depth of 60 cm. Each of the subsamples was divided into 5 depth classes (0-3.75 cm, 3.75-7.5 cm, 7.5-15 cm, 15-30 cm, and 30-60 cm). At 5 (SETRES) and 3 (Mt. Pleasant) random locations within each plot, a bucket auger was used to collect subsamples at 60-90 cm, 90-120 cm, and 120-150 cm. Subsamples were composited to produce 1 sample for each depth class per plot. Soils were air-dried at room temperature for one week and sieved (2 mm).

Bulk density determined in a previous study at SETRES (Lee, 2002) was used to report nutrients in kg ha^{-1}. Bulk density measurements were conducted at Mt. Pleasant in January 2010. Five subsample cores were collected in each plot for the depth classes described above to a depth of 60 cm (n=15 per treatment/species combination). One core was collected in each plot for classes contained within the 60-120 cm depth (n=3 per treatment/species combination). Bulk density of the 90-120 cm depth class was used to approximate the 120-150 cm depth class.

At 5 (SETRES) and 3 (Mt. Pleasant) random locations within each plot samples of the Oi, Oe, and Oa (when present) forest floor horizons were collected using a 0.07 m^2 sampler (SETRES) and a 0.25 m^2 sampler (Mt. Pleasant). Collection of forest floor material from a specific area allowed extrapolation of nutrient contents to kg ha^{-1}. The subsamples were composited to produce 1 sample for each forest floor horizon per plot. Samples were oven-dried at 60 ^{0}C for approximately 1 week, weighed to determine ovendry mass, and ground using a Thomas-Wiley Model 4 mill (Thomas Scientific, USA). Mass of Oa material was corrected with loss on ignition.

2.2.3. Nitrogen Budget

A simple N budget comparing means was developed in order examine site retention of N added through fertilization. Tree biomass N content at SETRES was taken from Albaugh et al. (2008) at age 21 and extrapolated to age 23. Tree biomass components at Mt. Pleasant were calculated with the methods of Cobb et al. (2008) (stem and branch) and Coyle et al. (2008) (roots). Foliar nutrient concentration at Mt. Pleasant was determined in the sampling for Chapter 3 and foliage mass was estimated from litterfall mass with the method of Allen et al. (2005). Higher N in tree, forest floor, and mineral soil components was assumed to be derived from N fertilizer inputs.

2.2.4. Chemical Analysis

Mineral soil and forest floor samples were analyzed for total-C and -N by dry combustion with a Vario MAX CNS analyzer (Elementar, Hanau, Germany). Mineral soil and forest floor phosphorus were determined by Mehlich 3 extraction and dry-ashing at 500 ^{0}C followed by dissolution with 6N HCl, respectively. Total-P was determined on a subset of the surface mineral soil samples (0-15 cm) from both sites by digestion in sulfuric acid with a copper sulfate catalyst. Phosphorus in solution was analyzed with inductively-coupled plasma atomic emission spectrophotometry (ICP-AES) on a Varian Vista MPX **(**Varian, Palo Alto, CA, USA). Mineral soil total-N concentrations fell below the method detection limits of 0.05 g kg^{-1} in the subsoil at both sites. Values below the detection limits were replaced with zero (Yess, 1993).

2.2.5. Statistical Analysis

Forest floor and mineral soil variables were tested for treatment, species, forest floor horizon, mineral soil depth, and interaction effects with mixed model ANOVA, with forest floor horizon or mineral soil depth as a repeated measure (Littell et al., 1998) (PROC MIXED, SAS Institute Inc., Cary, NC, USA). Interactions of the main effects of treatment or species with horizon or depth indicated statistically significant main effects at a specific horizon or depth. Tests for these effects were conducted with the ‘slice’ option in the least squared means within PROC MIXED (Shuster et al., 2001) and references to differences in specific horizons or depths in the results indicate these effects were found.

2.3. Results

2.3.1. Forest Floor

SETRES

Fertilization increased total forest floor mass by 16.7 Mg ha^{-1} (mean difference of unfertilized and fertilized treatments) with this increase attributable to increases in mass of the Oe horizon (Table 2.4, 2.5).

Fertilization increased forest floor N concentration in all horizons (Table 2.4, Table 2.5). Fertilization increased Oe, Oa, and total forest floor N content (Table 2.4, 2.5). Oe horizon N content was increased by 160.0 kg ha^{-1} (mean difference of unfertilized and fertilized treatments). Oa horizon N content was increased by 100.6 kg ha^{-1}. Total forest floor N content was increased by 300 kg ha^{-1}.

An overall fertilization effect was found for forest floor P concentration indicating a general increase from fertilization not specific to any horizon (Table 2.4, 2.5).

Fertilization increased P content in the Oe and Oa horizons and total forest floor P content (Table 2.4, 2.5). Oe horizon P content was increased by 7.5 kg ha^{-1} (mean difference of unfertilized and fertilized treatments). Oa horizon P content was increased by 9.8 kg ha^{-1}. Total forest floor P content was increased by 19.8 kg ha^{-1}.

Mt. Pleasant

Forest floor Oe horizon mass and total forest floor mass was higher in loblolly pine than in sweetgum with a difference of 5.2 and 7.2 Mg ha^{-1} (mean difference of all loblolly pine and sweetgum treatments), respectively (Table 2.6, 2.7). An overall treatment effect was observed indicating loblolly pine forest floor mass decreased on the order of Ct > FI > I (Table 2.6, 2.7). However, loblolly pine FI treatment Oa horizon mass appeared higher than both unfertilized treatments (Table 2.6). Unlike loblolly pine, sweetgum forest floor mass decreased on the order of FI > Ct > I as indicated by a statistically significant Trt. x Species effect (Table 2.7). Irrigation appeared to have decreased forest floor mass in both species indicated by lower mass in the I treatments compared to the Ct treatments.

An overall treatment effect was observed for forest floor total-N concentration indicating differences not specific to any horizon (Table 2.6, 2.7). Total-N concentration generally decreased on the order of FI > Ct > I for both species. However, total-N concentration in the loblolly pine Oa horizon was similar among Ct and FI treatments (Table 2.6). Oe horizon and total forest floor N content varied by treatment and species (Table 2.6, 2.7). In loblolly pine, Oa horizon N content of the FI treatment was higher than both unfertilized treatments (Table 2.6). In the Oe horizon, FI treatment N content was higher than in the I treatment but not the Ct treatment (Table 2.6). Total forest floor

N content of FI treatments was higher than both unfertilized treatments (Table 2.6). Oe horizon and total forest floor N content was higher in loblolly than in sweetgum with a difference of 37.3 and 82.4 kg ha^{-1} (mean difference of all loblolly pine and sweetgum treatments), respectively (Table 2.6). Loblolly pine total forest floor N content was 136 (Ct), 89 (I), and 233 (FI) kg ha^{-1}. Sweetgum total forest floor N content was 54 (Ct), 28 (I), and 128 (FI) kg ha^{-1}. Results indicated that irrigation decreased forest floor N content below the control.

Oe and Oa horizon P concentrations varied by treatment (Table 2.7). In the Oe horizon of loblolly pine and sweetgum, P concentration was lowest in the I treatment (Table 2.6). In the loblolly pine Oa horizon, P concentration was highest in the Ct treatment and similar among I and FI treatments indicating irrigation decreased P concentration (Table 2.6). Both treatment and species effects were observed for forest floor P content (Table 2.7). Oe horizon and total forest floor P content was higher in loblolly pine than in sweetgum with a difference of 2.8 and 5.7 kg ha^{-1} (mean difference of all loblolly pine and sweetgum treatments), respectively (Table 2.6). In the Oe horizon, FI treatment P content was higher than the I treatment but did not differ from the Ct treatment (Table 2.6). In the loblolly pine Oa horizon, FI treatment P content was higher than both unfertilized treatments. For both loblolly pine and sweetgum, total forest floor P content of the FI treatment was higher than the I treatment but did not differ from the Ct treatment (Table 2.6). Loblolly pine total forest floor P content was 11.7 (Ct), 7.3 (I), and 14.4 (FI) kg ha^{-1}, respectively (Table 2.6). Sweetgum total forest floor P content was 4.3 (Ct), 2.3 (I), and 9.6 (FI) kg ha^{-1}, respectively (Table 2.6).

2.3.2. Mineral Soil

SETRES

Fertilization had no effect on mineral soil N concentration (Table 2.8, 2.10). Nitrogen concentration decreased with depth and began to fall below the method detection limit in the 30-60 cm depth class. However, fertilization increased N content of the mineral soil. Fertilization increased N content in the 30-60 depth class resulting in an increase throughout the sampled profile from 0-150 cm (Table 2.8, 2.10). Fertilization increased N content throughout the sampled profile from 0-150 cm by 199 kg ha^{-1} (mean difference of unfertilized and fertilized treatments). Comparison of N content throughout the sampled profile from 0-150 cm in the Ct treatment and the F treatment indicated fertilization increased N content by 312 kg ha^{-1} (Table 2.8). Comparison of N content in the irrigated treatments indicated N content was 86 kg ha^{-1} higher in the FI treatment compared to the I treatment (Table 2.8). A Frt. x Irr. interaction effect was found for mineral soil total-N concentration (Table 2.9). Total-N concentration in the FI treatment appeared lower than in the F treatment in the 4 depth classes ranging from 0-30 cm and appeared higher in the 2 depth classes ranging from 30-90 cm suggesting downward movement of N (Table 2.8). This suggests that the increase in mineral soil N from fertilization was in a labile N pool that could be mobilized by irrigation. The mineral soil CN ratio was not altered by fertilization and irrigation and decreased with depth (Table 2.8, 2.10).

Fertilization increased mineral soil Mehlich 3-P concentration in the 4 depth classes ranging from 0-30 cm (Table 2.9, 2.10). Mineral soil P content was increased by fertilization in the 3.75-7.5 cm and 7.5-15 cm depth classes (Table 2.9, 2.10).

Fertilization increased P content throughout the sampled profile from 0-150 cm by 57 kg ha^{-1} (mean difference of unfertilized and fertilized treatments) (Table 2.9). Comparison of total-P and Mehlich 3-P indicated that fertilization increased the Mehlich 3 extractable P fraction. Mehlich 3-P concentration as a percent of total-P concentration was 4.6% for the Ct and I treatments and 24.0% and 28.5% for the F and FI treatments in the 0-15 cm depth, respectively (Table 2.11).

Mt. Pleasant

Fertilization did not increase mineral soil N concentration or content (Table 2.12, 2.14). Nitrogen concentration and content decreased with depth. In the 90-120 depth, N concentrations were only detected in the Ct treatments and were below instrument detection limits. In the 90-120 cm depth, N contents were only detected in the Ct treatments. Nitrogen concentration and content decreased with depth and began to fall below the method detection limit in the 60-90 cm depth class. Nitrogen content throughout the sampled profile from 0-150 cm averaged 1.9 Mg ha^{-1}. A treatment effect on the mineral soil CN ratio was nearly statistically significant ($p = 0.0589$) (Table 2.14). Data indicated the loblolly Ct treatment had the highest CN ratio (Table 2.12).

At Mt. Pleasant, mineral soil P concentrations were higher than those of fertilized treatments at SETRES in all treatment/species combinations with the exception of the loblolly I treatment. At Mt. Pleasant, the whole site has likely undergone one or more past P fertilizer applications for this to have occurred. Mineral soil P concentration did not vary by treatment or species and decreased with depth (Table 2.13, 2.14). A Trt. x Species interaction was observed for P content and indicated that P content was lowest in the loblolly pine I treatment while in sweetgum P content was lowest in the Ct treatment

(Table 2.13, 2.14). For loblolly pine, irrigation appeared to have increased the mobility of P resulting in leaching. These differences were most apparent in the surface depths. Increased P mobility was also indicated in comparisons of total-P and Mehlich 3-P. In the loblolly I treatment, Mehlich 3-P concentration as a percent of total-P concentration was consistently lower at 13.8% in the 0-15 cm depth than the other treatments which averaged 27.4% (Table 2.15). Mehlich 3-P concentration as a percent of total-P concentration was highest in loblolly pine and sweetgum FI treatments indicating that fertilization increased the Mehlich 3 extractable P fraction.

2.3.3. Nitrogen Budget

The N budget reflected differences in means and not statistical differences. At SETRES, fertilization increased tree, forest floor, and mineral soil N content by 275, 300, and 199 kg ha^{-1}, respectively (Table 2.16). This indicated 56% retention of fertilizer N at SETRES. Accumulations were more or less equal among components. At Mt. Pleasant, loblolly pine tree, forest floor, and mineral soil N contents were 68, 120, and 294 kg ha^{-1}, respectively, higher in the FI treatment (Table 2.16). This indicated 43% retention of fertilizer N in loblolly pine at Mt. Pleasant. Also at Mt. Pleasant, sweetgum tree, forest floor, and mineral soil N contents were 380, 87, and 805 kg ha^{-1}, respectively, higher in the FI treatment (Table 2.16). This indicated 113% retention of fertilizer N in sweetgum at Mt. Pleasant. Comparison of N retention among loblolly pine at SETRES and Mt. Pleasant indicated that the fertilization regime at SETRES, additions of approximately 56 kg ha^{-1} as granular urea N over a longer period of time, led to higher N retention compared to Mt. Pleasant where additions of 114 kg ha^{-1} as soluble NH_4-NO_3 occurred over a shorter period of time. Sweetgum was much more effective at capturing fertilizer N than loblolly pine.

2.4. Discussion

Fertilization at SETRES led to an increase in mineral soil N. This was not in agreement with the hypothesis of Miller (1981) who stated that N fertilization only benefits the trees and not the site because there is generally little impact of N fertilization on the mineral soil. The soils examined in this study had similar properties that influence retention of mineral-N as the soils in the study by Miller (1981). However, the fertilization regime differed among our study and the one by Miller (1981). At SETRES, the 1378 kg ha^{-1} of N added was applied incrementally from 1992 to 2008. In the study by Miller (1981), between 252 and 1512 kg N ha^{-1} was applied over 3 years. Comparison of fertilization regimes suggests a more gradual application of N can lead to retention of N in sandy mineral soils. However, results from SETRES suggested that the increase in mineral soil N was not in a stable, long-term fraction due to increased N mobility in the FI treatment. At Mt. Pleasant, fertilization ended 3 years prior to sampling and mineral soil total-N was not higher in fertilized plots. The lack of an increase in mineral soil N at Mt. Pleasant indicates the fertilization effect on mineral soil N at SETRES may disappear when N applications end suggesting short-term rather than long-term effects.

The hypothesis by Miller (1981) also stated that site improvement through increased mineral soil N is not likely unless the amount of nutrient applied is large in relation to the site capital. At SETRES, site N capital was approximately 1500 kg ha^{-1} in the unfertilized plots (Table 2.16). The 1378 kg ha^{-1} of N added was 92% of site N capital. In the study by Miller (1981), the highest amount of N fertilization was 140% of the site N capital in unfertilized stands. The amount of N added in relation to site N capital in the study by Miller (1981) was larger than at SETRES suggesting that the gradual application of N at SETRES rather than the amount added in relation to site capital contributed to the retention of N in the mineral soil.

Increases in mineral soil N from fertilization have been reported at sites where the amount of N applied was much less than the site N capital relative to our study sites (Binkley and Reid, 1985; Will et al., 2006). The soil in a study by Binkley and Reid (1985) had andic properties and high organic matter content. The soils in a study by Will et al. (2006), referred to in this discussion, were Ultisols with mineral soil C concentration similar to our sites. Nitrogen fertilization of Douglas-fir [*Pseudotsuga menziesii* (Mirb.) Franco] at Wind River was found to result in a long-term increase in mineral soil N availability (Binkley and Reid, 1985). Fertilization with a broadcast application of 470 kg ha^{-1} of NH_4NO_3 sustained a growth response at Wind River for 15 years (Miller and Tarrant, 1983). The 470 kg ha^{-1} of N added was only 13% of the site N capital. Mineral soil N availability in the 0-15 cm depth (56 mg kg^{-1}) 15 years after the NH_4NO_3 application was still double that of unfertilized plots (25 mg kg^{-1}). The soil at Wind River had higher organic matter than our sites (Tarrant and Miller, 1963) and was a gravelly loam Andic Haplumbrept (Binkely and Reid, 1985) indicating more active mineralogy. In a study by Will et al. (2006), addition of 900 kg N ha^{-1} as DAP and NH_4NO_3 was 40% of the site N capital in unfertilized plots. Extractable NH_4-N and NO_3-N in the 0-10 cm mineral soil depth was increased from 7.1 to 21.7 µg g^{-1} and total-N increased from 0.55 g kg^{-1} to 0.71 g kg^{-1}. Carbon concentration was not increased by fertilization suggesting the increase in mineral soil N was attributable to retention of mineral-N. Although not statistically significant, we observed that mineral soil total N in the 0-3.75 cm depth at SETRES was 0.50 g kg^{-1} in the Ct treatment and 0.67 g kg^{-1} in the F treatment, a change similar to that observed by Will et al. (2006). Comparison of our results with those of Binkley and Reid (1985) and Will et al. (2006) suggest a greater potential for fertilization to increase mineral soil N at sites with more active mineralogy such as clays and those with andic properties compared to the low activity mineralogy of soils dominated by quartz.

The smaller capacity for fertilization to increase mineral soil N on sandy soils and short-lived effects were likely due to the properties of the mineral soil. The soils examined in this study were acidic leading to minimal CEC. Low pH also leads to conditions unfavorable for fixation of NH_3 by organic matter (Johansson, 1998). The quartz mineralogy also leads to minimal capacity for NH_4^+ retention through chemisorption compared to the higher retention capacity of fine textured soils (Mamo et al., 1993). Since the soils were coarse textured and well-drained, conversion of NH_4-N to NO_3-N would likely result in losses by leaching. In a ^{15}N tracer study, Preston and Mead (1994) suggested fertilizer N that was mineralized became subject to loss through leaching. The increase in mineral soil N through the retention of mineral-N in the studies by Binkley and Reid (1985) and Will et al. (2006) was likely influenced by higher activity mineralogy leading to higher retention of NH_4^+ through both fixation and cation exchange (Mamo et al., 1993). Similar to our sites, fixation of NH_3 by organic matter in the studies by Binkley and Reid (1985) and Will et al. (2006) was likely not as important due to acidic pH typical of forest soils. However, this mechanism was probably more important at the Wind River site due to the high organic matter content of the mineral soil (Tarrant and Miller, 1963).

In a K^+ tracer study, Stone and Kszystyniak (1977) reported that when red pine (*Pinus resinosa* Ait.) growing on a sandy outwash soil was fertilized with K^+, approximately 70% of added K^+ was retained in the forest floor and 0-15 cm mineral soil depth 23 years after fertilization. In addition, 23 years after K^+ fertilization, 40% of foliar K^+ was derived from the fertilizer. One would anticipate NH_4^+ to behave similarly to K^+ in terms of mineral soil mobility resulting in similar retention and cycling processes. However, unlike K^+, NH_4^+ can be converted to the highly mobile and leachable NO_3^-. Therefore, N retention and cycling of fertilized pine growing on a sandy soil is not likely to proceed similarly to K^+ when nitrification occurs.

Fertilization was found to result in a small increase in mineral soil N at SETRES. Mineral soil N retention was 15% of added N. Total retention of added N in the trees, forest floor, and mineral soil was determined to be only 56%. A similar N retention percentage (43%) was found for loblolly pine at Mt. Pleasant while sweetgum appeared to have accumulated N above the amount added by exhibiting a retention of 113%. Possible mechanisms for N losses at SETRES include urea volatilization and NO_3 leaching. At Mt. Pleasant, since soluble NH_4-NO_3 was applied in 7 of the 9 years of fertilization, NO_3^- leaching likely contributed the most to N fertilizer losses. From 10-15% of added N has been found to be lost to urea volatilization (Zerpa and Fox, 2011). At these rates, urea volatilization would account for 137 to 206 kg ha^{-1} of the 1378 kg ha^{-1} of added N. This would leave approximately 30% or 413 kg ha^{-1} to be assumed to be lost to NO_3^- leaching. In loblolly pine at Mt. Pleasant, we assume 643 kg N ha^{-1} was lost to leaching. Remarkably, sweetgum appeared to accumulate N above the amount added with the additional amount assumed to be captured atmospheric N deposition. Although statistically significant differences were not determined, the simple N budget indicated that sweetgum accumulated much more N in the trees and mineral soil relative to loblolly pine. Our results coincided with the findings of George et al. (2003) who reported sweetgum N uptake was approximately double that of loblolly pine.

Results suggest that while N fertilization at SETRES did result in an increase in mineral soil N, the increase was likely in a labile fraction subject to increased mobility with irrigation. The lack of higher mineral soil N at Mt. Pleasant where fertilization ended 3 years prior to sampling suggests the mineral soil fertilization effect at SETRES would not be sustained if N additions ceased. The forest floor was found to accumulate more N relative to the mineral soil and exhibited a sustained fertilization response at Mt. Pleasant. As a result, the effects of N fertilization on loblolly pine and sweetgum plantations are likely transient due to the instability of the forest floor. The forest floor N pool is simply not a stable enough pool to supply N over the long-term from one rotation to the next. Upon harvest, the forest floor rapidly decomposes and releases N that has accumulated during the life of the stand (Kimmins, 1997). At SETRES, which was more representative of a rotation length than the 12-year old stands at Mt. Pleasant, the forest floor had accumulated as much as 565 kg N ha^{-1}. Since next rotation seedling nutrient demand is not great enough to capture the N released from the decomposing forest floor (Fox et al., 2007), much of the N sequestered in the forest floor may be lost following harvest due to the properties of the sandy soils in this study resulting in minimal mineral-N retention.

Results indicated that irrigation appeared to have exerted a strong influence on the forest floor and mineral soil at Mt. Pleasant. Differences in forest floor responses to irrigation among sites suggests that forest floor decomposition of non-irrigated plots was limited by available moisture at Mt. Pleasant but was not limited at SETRES. This could be explained by precipitation at SETRES that supplied adequate moisture to facilitate similar forest floor decomposition rates among irrigated and non-irrigated plots. Results suggest Mt. Pleasant experienced a precipitation deficit of long enough duration to create distinctly different moisture regimes among irrigated and non-irrigated plots. Results provided evidence that irrigation not only accelerated forest floor decomposition at Mt. Pleasant, but also led to greater leaching of P from the forest floor. Leaching of P has been reported to be a key mechanism in the release of P from loblolly pine litter (Polglase et al., 1992) and appeared to have occurred in sweetgum as well.

Fertilization resulted in increased mineral soil P at SETRES consistent with the findings of Harding and Jokela (1994) in a study of P fertilization of slash pine growing on a sandy soil. Mt. Pleasant mineral soil P was not increased by fertilizer P additions began at the initiation of the study in 1997. However, Mehlich 3-P in the Mt. Pleasant control plots was higher than in the fertilized plots at SETRES indicating the site had likely already undergone one or more past P fertilizer applications. Prior to establishment of the study, the Mt. Pleasant site was a short-rotation pulpwood plantation. In contrast, SETRES was a native longleaf pine forest that had not been fertilized. Past P applications at Mt. Pleasant were likely typical operational fertilization rates that range from 28 to 56 kg ha^{-1} (Albaugh et al., 2007). Our results indicated that the recent P fertilizer additions that have taken place since 1997 did not increase mineral soil P. However, high mineral soil P concentrations relative to the SETRES control indicate the mineral soil has accumulated P from past additions providing further support for fertilization resulting in increased mineral soil P in sandy soils. Our results indicated a trend for irrigation to decrease mineral soil P. Therefore, it is possible that further increases in mineral soil P would have been observed at Mt. Pleasant in a fertilization only treatment.

The accumulation of P in the mineral soil was likely due to properties of the mineral soil promoting P retention. In a study by Yuan (1960), a mineral soil P fractionation was conducted on sandy soils with a pH range of 5.4 to 5.7 and a CEC range of 2.8 to 8.0 $cmol_{+}\ kg^{-1}$. The properties were similar to those of the soils sampled in this study. Yuan (1960) found that the form of added P retained was dominated by Al- and Fe-phosphates compared to water soluble and Ca-phosphate forms. Over 80% of the added P was retained by the Al- and Fe-phosphate forms. Smith (1965) also suggests Al and Fe are largely responsible for absorbing P in acid forest soils. Ballard and Fiskell (1974) reported that ammonium oxalate extractable Al and Fe provided the best indices for retention of P in acid forest soils. Phosphorus retention in the 0 to 20 cm depth was high in sandy Entisols and finer textured Inceptisols and Ultisols due to extractable Al and Fe ranging from 250 to 1250 ppm and 130 to 1560 ppm, respectively. We used an extractant with similar properties (Mehlich 3) and found levels of extractable Al, Fe, and P approximate to those found by Ballard and Fiskell for sandy Entisols (1974). Phosphorus retention in the 0 to 20 cm depth was low only in Spodosols due to this depth comprising highly leached A and E horizons that exhibited very low extractable Al and Fe.

Al- and Fe-phosphates are stable forms of mineral soil P. However, these forms can be made available for plant uptake by dissolution and ligand-exchange reactions with organic acids (Fox et al., 1990; Chen, 2008). At SETRES and Mt. Pleasant, mineral soil P concentrations were highest in the surface horizons where organic matter was also higher. Aluminum and Fe were also found in appreciable quantities suggesting the retention of P in a mixture of Al- and Fe-oxides and organic matter as reported by Ballard and Fiskell (1974).

Results suggest that irrigation increased the mobility of mineral soil P. However, this effect was much more pronounced at Mt. Pleasant where forest floor decomposition was increased by irrigation. Mineral soil P in the surface depths was lowest in the I treatment. Furthermore, in spite of the addition of 169 kg P ha^{-1}, P in the FI treatment was generally comparable to the Ct treatment. This effect was greater in loblolly pine compared to sweetgum. In a study comparing forest floor leachate among forest floor types, eastern hemlock (Tsuga Canadensis L. [Carr.]) leachate was more acidic at pH 3.9 than hardwood leachate at pH 4.1 to 4.9 due to higher organic acid concentrations (De Walle et al., 1985). Similar differences were suggested by the results to occur among loblolly pine and sweetgum. We propose that at Mt. Pleasant, irrigation increased the decomposition of the forest floor and the quantity of forest floor leachate entering the mineral soil resulting in increased release of Al- and Fe-phosphate bound P through dissolution and ligand exchange. This effect was more pronounced in loblolly pine than in sweetgum due to the higher acidity of loblolly pine forest floor leachate. This was supported by mineral soil P data indicating that in irrigated loblolly, Mehlich 3-P as a percent of total-P was reduced from 27.4% to 13.8%. The accumulation of P in the mineral soil observed in this study suggests P fertilization resulted in a long-term increase in site P availability which was anticipated to result in increased supply of P to the next rotation. This finding was consistent with the effects of P fertilization on long-term site P availability proposed by Prichett and Comerford (1982) and Fox et al. (2011).

2.5. Conclusions

Results suggested that fertilization of loblolly pine and sweetgum with N will most likely not result in a long-term increase in mineral soil N availability and increased supply of N to trees throughout the next rotation is unlikely. While fertilization was found to increase mineral soil N, the increase was small and likely occurred in labile fractions not stable over the long-term. Relative to the mineral soil, the largest and most consistent increases were in the labile forest floor N pool. Future changes in N availability from fertilization will be largely determined by decomposition of the forest floor. Since the forest floor decomposes rapidly upon harvest and N demand of the newly planted next rotation is low, the N that has accumulated in the forest floor may be lost due to a lack of capture by vegetation. In contrast to N, fertilization of loblolly pine and sweetgum with P results in a long-term increase in mineral soil P availability. Fertilization increases the stable mineral soil P pool that can be made available for plant uptake over time suggesting increased supply of P to trees throughout the next rotation.

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2.7. Tables

Table 2.1. Select properties of the 0-15 cm and 15-30 cm mineral soil depths at SETRES and Mt. Pleasant.

Property	SETRES	Mt. Pleasant
	0-15 cm	
C (g kg^{-1})	13.0	11.7
pH	4.4	5.3
CEC ($cmol_+$ kg^{-1})	4.0	3.9
Mehlich 3-Al (mg kg^{-1})	no data	383.2
Mehlich 3-Fe (mg kg^{-1})	135.3	93.4
	15-30 cm	
C (g kg^{-1})	3.3	5.6
pH	4.9	5.4
CEC ($cmol_+$ kg^{-1})	0.89	2.2
Mehlich 3-Al (mg kg^{-1})	no data	612.3
Mehlich 3-Fe (mg kg^{-1})	59.8	90.2

Table 2.2. Nutrient additions at SETRES from 1992 to 2008. Granular fertilizer was applied to the surface on each application date.

Application Date	N	P	K	Ca	Mg	S	B
	kg ha^{-1}						
Mar-92	224	56	112	0	0	0	0
Apr-92	0	0	0	134	56	0	0
Jun-92	0	0	0	0	0	0	2
Mar-93	0	28	0	0	0	0	0
Apr-93	26	22	21	0	0	0	0
Jun-93	0	0	92	0	56	120	0
Aug-93	56	0	0	0	0	0	0
Mar-94	112	0	0	0	0	0	0
Mar-95	56	28	56	24	34	74	0
May-95	0	0	0	0	0	0	1
Mar-96	112	11	56	10	0	15	0
Apr-96	0	0	0	0	0	0	1
Apr-97	135	0	0	0	0	0	0
Mar-98	56	6	0	0	0	0	0
Mar-99	69	0	0	0	0	0	0
Mar-00	56	6	0	0	0	0	0
Apr-01	56	6	0	0	0	0	0
Apr-02	56	6	0	0	0	0	0
May-02	0	0	56	0	0	0	0
Apr-03	56	0	0	0	0	0	0
Mar-04	56	0	0	0	0	64	0
Mar-05	84	0	0	0	0	0	1
Mar-06	56	0	0	0	0	64	0
Apr-07	56	0	0	0	0	0	0
Apr-08	56	0	0	0	0	0	0
Total	1378	168	393	168	146	337	6

Table 2.3. Nutrient additions at Mt. Pleasant from 1997 to 2006. From 1997 to 2003, soluble fertilizer was applied over a 28 week period in the irrigation water. From 2004 to 2006, a single surface application of granular fertilizer was conducted in the spring.

Year	N	P	K	Cu	Mn	Mo	S	B	Fe
	kg ha^{-1}								
1997	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
1998	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
1999	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
2000	170	26	94	0.0	0.0	0.0	0.0	0.0	0.0
2001	51	8	28	0.0	0.0	0.0	0.0	0.0	0.0
2002	116	17	64	0.0	0.0	0.0	0.0	0.0	0.0
2003	116	17	64	0.0	0.0	0.0	0.0	0.0	0.0
2004	112	17	62	0.6	1.4	0.0	1.4	0.6	3.4
2005	112	17	62	0.7	1.6	0.5	0.0	0.7	3.8
2006	112	17	62	0.7	1.6	0.5	0.0	0.7	3.8
Total	1128	169	621	2.0	4.6	1.0	1.4	2.0	11.0

Table 2.4. Mean forest floor mass and total-N and -P concentration and content at SETRES (n=4). Coefficients of variation are in parentheses. Means in a row followed by different letters are significantly different (Fertilization capital, Irrigation lowercase). † Indicates overall fertilization effect not specific to any horizon.

Horizon	Control			Irrigation			Fertilization			F x I		
						Forest Floor Mass Mg ha^{-1}						
Oi	4.8	(29.1)		5.8	(18.6)		10.2	(22.8)		8.7	(28.1)	
Oe	11.9	(15.8)	B	11.1	(38.4)	B	20.4	(24.8)	A	21.3	(44.5)	A
Oa	8.6	(6.9)		8.4	(57.4)		9.9	(41.6)		13.5	(31.8)	
Total	25.3	(10.7)	B	25.3	(28.5)	B	40.5	(24.0)	A	43.6	(22.3)	A
						total-N g kg^{-1}						
Oi	4.79	(10.7)	B	4.62	(7.0)	B	6.99	(8.1)	A	6.47	(6.0)	A
Oe	8.07	(6.4)	B	7.73	(5.8)	B	12.10	(4.2)	A	11.97	(8.8)	A
Oa	13.42	(11.4)	B	16.47	(8.8)	B	19.69	(7.4)	A	18.75	(6.7)	A
						total-N kg ha^{-1}						
Oi	24	(40.3)		27	(19.4)		72	(29.4)		57	(34.0)	
Oe	96	(13.0)	B	86	(37.2)	B	248	(26.7)	A	254	(41.5)	A
Oa	115	(11.8)	B	137	(54.0)	B	199	(46.9)	A	254	(34.2)	A
Total	235	(13.1)	B	249	(30.9)	B	519	(30.2)	A	565	(20.3)	A
						† total-P mg kg^{-1}						
Oi	253	(11.7)		236	(8.1)		403	(8.6)		405	(6.0)	
Oe	386	(4.8)		362	(12.1)		559	(14.6)		584	(11.5)	
Oa	949	(18.3)		1252	(37.0)		2168	(46.3)		1254	(26.1)	
						total-P kg ha^{-1}						
Oi	1.2	(40.1)		1.4	(21.1)		4.1	(29.5)		3.5	(30.3)	
Oe	4.6	(16.8)	B	3.9	(31.3)	B	11.4	(27.1)	A	12.1	(33.8)	A
Oa	8.1	(13.3)	B	9.1	(29.4)	B	20.1	(44.8)	A	16.6	(36.0)	A
Total	14.0	(11.8)	B	14.4	(24.5)	B	35.6	(27.2)	A	32.3	(7.7)	A

Table 2.5. Repeated measures main and interaction effect p-values for SETRES forest floor mass and total-N and -P concentration and content. Statistically significant p-values are shown in bold.

Effect	p-value	
	Forest Floor Mass	
	Mg ha^{-1}	
Fertilization	**<0.0001**	
Irrigation	0.5433	
Horizon	**<0.0001**	
Fert. x Irr.	0.5323	
Fert. x Horizon	**0.0005**	
Irr. x Horizon	0.9217	
	total-N	
	g kg^{-1}	kg ha^{-1}
Fertilization	**<0.0001**	**<0.0001**
Irrigation	0.6531	0.4889
Horizon	**<0.0001**	**<0.0001**
Fert. x Irr.	0.0763	0.7081
Fert. x Horizon	**0.0014**	**<0.0001**
Irr. x Horizon	**0.0499**	0.7475
	total-P	
	mg kg^{-1}	kg ha^{-1}
Fertilization	**0.0172**	**<0.0001**
Irrigation	0.3547	0.5835
Horizon	**<0.0001**	**<0.0001**
Fert. x Irr.	0.1109	0.4898
Fert. x Horizon	0.1650	**<0.0001**
Irr. x Horizon	0.3900	0.9401

Table 2.6. Mean loblolly pine and sweetgum forest floor mass and total-N and -P concentration and content at Mt. Pleasant (n=3). Coefficients of variation are in parentheses. Means in a row followed by different letters are significantly different (Treatment capital, Species lowercase). † Indicates overall treatment effect not specific to any horizon.

Horizon	Control			Irrigation			Fertilization + Irrigation			Control			Irrigation			Fertilization + Irrigation		
	Loblolly									Sweetgum								
	Forest Floor Mass Mg ha^{-1}																	
Oi	5.6	(37.4)		2.9	(27.3)		3.7	(25.2)		2.9	(51.1)		1.9	(58.5)		3.8	(24.4)	
Oe	10.8	(43.0)	a	6.6	(22.2)	a	9.8	(27.0)	a	2.9	(48.9)	b	1.7	(48.2)	b	6.9	(18.3)	b
Oa	0.1	(29.7)		0.6	(33.5)		1.5	(25.1)		-	-		-	-		-	-	
Total	16.6	(35.8)	a	10.1	(24.2)	a	15.1	(21.8)	a	5.8	(45.5)	b	3.6	(51.9)	b	10.8	(9.6)	b
	† total-N g kg^{-1}																	
Oi	5.29	(5.9)		4.58	(9.1)		7.16	(30.1)		7.81	(3.6)		6.77	(8.4)		8.03	(20.5)	
Oe	9.08	(3.4)		7.36	(6.3)		10.55	(19.2)		10.72	(4.5)		8.80	(2.9)		14.04	(15.1)	
Oa	64.01	(22.3)		46.25	(26.6)		64.56	(26.6)		-	-		-	-		-	-	
	total-N kg ha^{-1}																	
Oi	29	(33.9)		13	(31.3)		28	(51.8)		23	(54.0)		13	(62.5)		32	(40.7)	
Oe †	99	(44.9)	AB,a	49	(23.0)	B,a	107	(41.9)	A,a	31	(52.5)	AB,b	15	(49.2)	B,b	97	(19.1)	A,b
Oa	8	(43.8)	B	27	(3.7)	B	97	(40.5)	A	-	-		-	-		-	-	
Total	136	(39.1)	B,a	89	(18.4)	B,a	233	(7.2)	A,a	54	(47.3)	B,b	28	(54.0)	B,b	128	(19.7)	A,b
	total-P mg kg^{-1}																	
Oi	613	(10.7)		616	(10.8)		761	(23.2)		692	(3.8)		647	(5.3)		734	(8.2)	
Oe	690	(10.4)	A	568	(6.2)	B	699	(15.6)	A	772	(3.7)	A	658	(6.9)	B	977	(10.9)	A
Oa	4593	(16.5)	A	3002	(7.9)	B	2990	(13.2)	B	-	-		-	-		-	-	
	total-P kg ha^{-1}																	
Oi	3.4	(29.8)		1.7	(21.5)		3.0	(45.2)		2.0	(52.1)		1.2	(57.1)		2.8	(30.6)	
Oe	7.7	(49.3)	AB,a	3.7	(17.7)	B,a	7.1	(39.0)	A,a	2.3	(53.2)	AB,b	1.1	(52.1)	B,b	6.7	(15.7)	A,b
Oa	0.6	(44.1)	B	1.8	(25.2)	B	4.4	(15.3)	A	-	-		-	-		-	-	
Total	11.7	(40.6)	AB,a	7.3	(20.2)	B,a	14.4	(22.8)	A,a	4.3	(48.2)	AB,b	2.3	(53.0)	B,b	9.6	(15.2)	A,b

Table 2.7. Repeated measures main and interaction effect p-values for Mt. Pleasant forest floor mass and total-N and -P concentration and content. Statistically significant p-values are shown in bold.

Effect	p-value	
	Forest Floor Mass	
	Mg ha^{-1}	
Treatment	**0.0004**	
Species	**<0.0001**	
Horizon	**<0.0001**	
Trt. x Species	**0.0175**	
Trt. x Horizon	0.0567	
Species x Horizon	**0.0009**	
	total-N	
	g kg^{-1}	kg ha^{-1}
Treatment	**0.0161**	**<0.0001**
Species	0.3264	**<0.0001**
Horizon	**<0.0001**	**<0.0001**
Trt. x Species	0.9970	0.3939
Trt. x Horizon	0.1417	**0.0002**
Species x Horizon	0.9425	**<0.0001**
	total-P	
	mg kg^{-1}	kg ha^{-1}
Treatment	**0.0017**	**0.0001**
Species	0.3079	**<0.0001**
Horizon	**<0.0001**	**<0.0001**
Trt. x Species	0.9478	0.1176
Trt. x Horizon	**0.0017**	**0.0076**
Species x Horizon	0.3834	**0.0023**

Table 2.8. Mean mineral soil total-N concentration and content and CN ratio at SETRES (n=4). Italicized total-N concentration values were below instrument detection limits. Coefficients of variation are in parentheses. Means in a row followed by different letters are significantly different (Fertilization capital, Irrigation lowercase).

Depth (cm)	Control			Irrigation			Fertilization			F x I		
	total-N g kg^{-1}											
0-3.75	0.504	(10.3)		0.655	(40.4)		0.674	(4.7)		0.526	(23.0)	
3.75-7.5	0.299	(27.7)		0.321	(8.3)		0.343	(8.4)		0.302	(10.1)	
7.5-15	0.155	(14.7)		0.163	(12.3)		0.187	(13.4)		0.173	(10.8)	
15-30	0.070	(19.4)		0.055	(68.8)		0.075	(12.4)		0.068	(71.2)	
30-60	*0.023*	*(200.0)*		*0.028*	*(117.5)*		*0.048*	*(70.2)*		0.067	(24.6)	
60-90	*0.034*	*(117.1)*		0.068	(27.4)		*0.036*	*(115.5)*		0.052	(74.4)	
90-120	*0.000*	-		*0.000*	-		*0.000*	-		*0.000*	-	
120-150	*0.000*	-		*0.000*	-		*0.013*	*(200.0)*		*0.000*	-	
	total-N kg ha^{-1}											
0-3.75	231	(14.6)		300	(48.3)		305	(8.8)		241	(27.1)	
3.75-7.5	137	(28.8)		144	(15.8)		156	(9.8)		138	(12.6)	
7.5-15	154	(16.7)		156	(7.3)		185	(13.4)		169	(12.7)	
15-30	141	(19.2)		106	(69.9)		152	(11.8)		138	(71.4)	
30-60	98	(200.0)	B	116	(117.5)	B	204	(70.3)	A	286	(24.8)	A
60-90	145	(116.6)		285	(29.9)		157	(115.7)		221	(73.7)	
90-120	*0*	-		*0*	-		*0*	-		*0*	-	
120-150	*0*	-		*0*	-		60	(200.0)		*0*	-	
Total	907	(27.4)	B	1107	(19.5)	B	1219	(13.9)	A	1193	(18.1)	A
	CN Ratio											
0-3.75	38.4	(8.4)		33.2	(24.7)		34.2	(5.6)		32.5	(5.0)	
3.75-7.5	38.2	(13.2)		41.1	(4.0)		37.8	(5.2)		38.5	(3.0)	
7.5-15	38.0	(9.5)		39.7	(5.9)		45.0	(12.0)		33.4	(16.2)	
15-30	43.6	(45.1)		33.7	(76.2)		54.0	(39.3)		34.1	(81.9)	
30-60	3.4	(200.0)		12.2	(115.5)		21.1	(68.2)		29.6	(6.1)	
60-90	7.4	(118.1)		11.9	(19.8)		9.3	(115.8)		12.0	(74.9)	
90-120	0.0	-		0.0	-		0.0	-		0.0	-	
120-150	0.0	-		0.0	-		4.4	(200.0)		0.0	-	

Table 2.9. Mean mineral soil Mehlich 3-P concentration and content at SETRES (n=4). Coefficients of variation are in parentheses. Means in a row followed by different letters are significantly different (Fertilization capital, Irrigation lowercase).

Depth (cm)	Control			Irrigation			Fertilization			F x I		
	Mehlich 3-P mg kg^{-1}											
0-3.75	3.54	(25.5)	B	3.13	(17.5)	B	22.81	(20.6)	A	23.33	(38.7)	A
3.75-7.5	3.44	(28.8)	B	2.73	(13.3)	B	28.46	(35.5)	A	31.40	(17.1)	A
7.5-15	2.45	(19.6)	B	2.93	(25.9)	B	23.61	(19.1)	A	24.83	(51.5)	A
15-30	2.14	(13.6)	B	2.45	(30.2)	B	6.66	(48.5)	A	6.91	(43.7)	A
30-60	1.05	(64.5)		1.21	(17.0)		2.49	(30.4)		2.17	(42.4)	
60-90	2.19	(21.1)		2.55	(12.9)		1.80	(17.4)		3.32	(32.4)	
90-120	2.93	(25.8)		3.69	(18.7)		2.58	(17.3)		3.86	(21.2)	
120-150	4.43	(46.7)		3.96	(23.3)		3.29	(45.4)		4.83	(35.4)	
	Mehlich 3-P kg ha^{-1}											
0-3.75	1.6	(29.5)		1.4	(5.8)		10.4	(23.2)		10.6	(39.5)	
3.75-7.5	1.6	(36.4)	B	1.2	(15.5)	B	12.9	(37.4)	A	14.4	(20.2)	A
7.5-15	2.4	(18.3)	B	2.8	(22.6)	B	23.3	(19.0)	A	24.4	(52.7)	A
15-30	4.3	(15.1)		4.8	(25.4)		13.4	(47.7)		13.9	(44.9)	
30-60	4.5	(65.6)		5.1	(13.8)		10.7	(27.8)		9.2	(42.8)	
60-90	9.3	(21.3)		10.7	(11.2)		7.8	(16.3)		14.1	(32.5)	
90-120	12.4	(25.2)		15.6	(20.5)		11.1	(15.5)		16.4	(21.8)	
120-150	18.9	(46.9)		16.7	(24.2)		14.0	(43.7)		20.5	(35.6)	
Total	55.0	(28.4)	B	58.2	(11.8)	B	103.5	(21.3)	A	123.5	(30.1)	A

Table 2.10. Repeated measures main and interaction effect p-values for SETRES mineral soil total-N and Mehlich 3-P concentration and content and CN ratio. Statistically significant p-values are shown in bold.

Effect	p-value	
	total-N	
	g kg^{-1}	kg ha^{-1}
Fertilization	0.3066	**0.0136**
Irrigation	0.9354	0.2596
Depth	**<0.0001**	**<0.0001**
Fert. x Irr.	**0.0446**	0.1462
Fert. x Depth	0.9890	**0.1005**
Irr. x Depth	0.9928	0.6423
	Mehlich 3-P	
	mg kg^{-1}	kg ha^{-1}
Fertilization	**<0.0001**	**0.0031**
Irrigation	0.5655	0.3918
Depth	**<0.0001**	**<0.0001**
Fert. x Irr.	0.6051	0.5300
Fert. x Depth	**<0.0001**	**<0.0001**
Irr. x Depth	0.9979	0.5035
	CN Ratio	
Fertilization	0.0973	
Irrigation	0.3931	
Depth	**<0.0001**	
Fert. x Irr.	0.2931	
Fert. x Depth	0.1324	
Irr. x Depth	0.0798	

Table 2.11. Mean mineral soil Mehlich 3-P and total-P concentration (mg kg^{-1}) at SETRES. Also shown is the Mehlich 3-P percentage of total-P. The 0-15 cm depth represents the mean of the three depth classes.

Depth (cm)	Control			Irrigation			Fertilization			F x I		
	M3-P	total-P	%	M3-P	total-P	%	M3-P	total-P	%	M3-P	total-P	%
0-3.75	3.54	70.9	5.0	3.13	33.7	9.3	22.8	142.4	16.0	23.3	63.3	36.8
3.75-7.5	3.44	73.6	4.7	2.73	102.4	2.7	28.5	75.4	37.7	31.4	122.6	25.6
7.5-15	2.45	60.1	4.1	2.93	54.2	5.4	23.6	94.3	25.0	24.8	93.4	26.6
0-15	3.14	68.2	4.6	2.93	63.4	5.8	25.0	104.0	26.3	26.5	93.1	29.7

Table 2.12. Mean loblolly pine and sweetgum mineral soil total-N concentration and content and CN ratio at Mt. Pleasant (n=3). Italicized total-N concentration values were below instrument detection limits. Coefficients of variation are in parentheses.

Depth (cm)	Control		Irrigation		Fertilization + Irrigation		Control		Irrigation		Fertilization + Irrigation	
	Loblolly						Sweetgum					
	total-N g kg^{-1}											
0-3.75	0.529	(54.0)	0.278	(43.5)	0.488	(26.1)	0.530	(31.8)	0.622	(41.1)	0.502	(21.5)
3.75-7.5	0.447	(47.2)	0.213	(3.5)	0.359	(24.4)	0.401	(27.4)	0.372	(11.7)	0.326	(16.1)
7.5-15	0.413	(46.9)	0.185	(2.7)	0.298	(40.2)	0.344	(46.3)	0.304	(7.4)	0.284	(29.8)
15-30	0.165	(41.1)	0.137	(6.2)	0.165	(12.4)	0.149	(24.8)	0.173	(32.2)	0.170	(24.6)
30-60	0.081	(5.5)	0.073	(13.1)	0.138	(38.6)	0.087	(12.7)	0.154	(47.5)	0.083	(101.4)
60-90	*0.039*	*(86.6)*	*0.000*	-	*0.000*	-	*0.041*	*(86.6)*	*0.000*	-	0.293	(173.2)
90-120	*0.037*	*(87.3)*	*0.000*	-	*0.000*	-	*0.019*	*(173.2)*	*0.000*	-	*0.000*	-
120-150	0.057	(87.2)	*0.000*	-	0.050	(173.2)	*0.017*	*(173.2)*	*0.019*	*(173.2)*	*0.000*	-
	total-N kg ha^{-1}											
0-3.75	182	(49.9)	114	(43.3)	189	(26.1)	197	(25.0)	259	(41.0)	171	(21.4)
3.75-7.5	164	(40.3)	90	(3.3)	151	(24.2)	149	(20.3)	155	(11.7)	130	(16.2)
7.5-15	393	(44.9)	182	(2.5)	293	(40.2)	341	(43.1)	303	(7.6)	288	(29.7)
15-30	369	(40.8)	301	(6.1)	362	(12.3)	331	(23.1)	389	(32.2)	377	(24.9)
30-60	365	(5.9)	325	(12.9)	625	(38.6)	390	(13.7)	692	(47.4)	368	(101.3)
60-90	177	(86.6)	0	-	0	-	181	(86.6)	0	-	1285	(173.2)
90-120	161	(87.5)	0	-	0	-	83	(173.2)	0	-	0	-
120-150	253	(87.2)	0	-	212	(173.2)	75	(173.2)	85	(173.2)	0	-
Total	2065	(35.7)	1012	(8.4)	1833	(22.0)	1747	(25.9)	1883	(29.0)	2620	(75.5)
	CN Ratio											
0-3.75	40.0	(38.2)	32.8	(11.9)	27.2	(16.4)	26.7	(3.2)	27.1	(7.2)	24.8	(1.8)
3.75-7.5	32.4	(6.9)	35.3	(3.7)	33.2	(5.5)	30.8	(1.5)	31.1	(8.0)	30.6	(13.0)
7.5-15	30.9	(8.7)	43.2	(5.8)	33.5	(23.0)	30.4	(3.6)	31.4	(19.6)	34.7	(3.6)
15-30	31.2	(6.9)	43.1	(9.5)	35.0	(14.4)	40.3	(15.8)	31.8	(14.5)	34.7	(35.1)
30-60	22.4	(37.5)	44.0	(20.0)	33.0	(16.5)	29.0	(13.8)	33.3	(31.4)	22.4	(94.0)
60-90	12.4	(97.7)	0.0	-	0.0	-	13.6	(86.7)	0.0	-	0.3	(173.2)
90-120	9.1	(88.1)	0.0	-	0.0	-	6.0	(173.2)	0.0	-	0.0	-
120-150	11.8	(93.0)	0.0	-	0.8	(173.2)	5.5	(173.2)	16.7	(173.2)	0.0	-

Table 2.13. Mean loblolly pine and sweetgum mineral soil Mehlich 3-P concentration and content at Mt. Pleasant (n=3). Coefficients of variation are in parentheses.

Depth (cm)	Control		Irrigation		Fertilization + Irrigation		Control		Irrigation		Fertilization + Irrigation	
	Loblolly						Sweetgum					
	Mehlich 3-P mg kg^{-1}											
0-3.75	32.97	(25.9)	17.52	(69.7)	38.76	(42.7)	42.83	(15.1)	37.81	(39.4)	41.27	(38.0)
3.75-7.5	32.18	(47.1)	12.31	(56.0)	48.85	(37.1)	33.34	(19.9)	28.75	(46.8)	43.05	(15.2)
7.5-15	30.68	(87.5)	10.28	(49.0)	41.74	(32.1)	21.97	(42.6)	19.49	(37.6)	31.80	(29.4)
15-30	45.71	(135.6)	10.12	(66.8)	31.38	(44.4)	16.00	(61.2)	28.08	(46.3)	28.34	(36.5)
30-60	28.80	(156.1)	6.75	(71.7)	24.11	(12.7)	6.52	(56.0)	19.39	(62.3)	15.89	(67.8)
60-90	3.50	(28.4)	3.25	(22.5)	4.57	(25.3)	3.56	(26.5)	4.05	(28.1)	3.76	(39.4)
90-120	6.45	(63.9)	2.85	(22.5)	3.79	(65.2)	3.49	(19.5)	3.39	(21.0)	3.87	(78.7)
120-150	19.60	(88.9)	5.79	(86.0)	8.37	(100.8)	6.03	(16.8)	16.66	(105.0)	15.18	(149.7)
	Mehlich 3-P kg ha^{-1}											
0-3.75	11.8	(33.4)	7.2	(69.3)	15.0	(43.0)	16.0	(9.5)	15.7	(39.6)	14.0	(37.8)
3.75-7.5	12.4	(50.0)	5.2	(55.6)	20.5	(37.1)	12.6	(23.1)	12.0	(46.7)	17.2	(15.1)
7.5-15	28.4	(80.0)	10.1	(48.9)	41.0	(32.0)	22.1	(45.9)	19.5	(37.7)	32.3	(29.4)
15-30	102.0	(135.5)	22.3	(66.9)	68.7	(44.4)	35.9	(62.3)	63.0	(46.3)	62.8	(36.5)
30-60	127.5	(155.8)	30.1	(71.7)	109.1	(12.7)	29.3	(55.6)	87.1	(62.3)	70.3	(67.8)
60-90	15.8	(28.3)	14.4	(22.6)	20.1	(25.4)	15.8	(26.7)	18.5	(28.1)	16.5	(39.6)
90-120	28.7	(65.3)	12.0	(22.7)	16.2	(65.3)	15.2	(20.8)	14.9	(21.0)	16.4	(78.6)
120-150	87.2	(89.1)	24.5	(86.0)	35.8	(100.8)	26.2	(16.2)	73.3	(105.0)	64.4	(149.7)
Total	413.9	(73.7)	125.8	(51.2)	326.4	(20.6)	173.3	(30.9)	304.0	(28.3)	294.0	(59.9)

Table 2.14. Repeated measures main and interaction effect p-values for Mt. Pleasant mineral soil total-N and Mehlich 3-P concentration and content and CN ratio. Statistically significant p-values are shown in bold.

Effect	p-value	
	total-N	
	g kg^{-1}	kg ha^{-1}
Treatment	0.2428	0.1172
Species	0.2364	0.1444
Depth	**<0.0001**	**<0.0001**
Trt. x Species	0.1832	0.2090
Trt. x Depth	0.7581	0.7224
Species x Depth	0.4878	0.5919
	Mehlich 3-P	
	mg kg^{-1}	kg ha^{-1}
Treatment	0.2053	0.3638
Species	0.9687	0.5848
Depth	**<0.0001**	**<0.0001**
Trt. x Species	0.2159	**0.0266**
Trt. x Depth	0.7650	0.9712
Species x Depth	0.6733	0.9836
	CN Ratio	
Treatment	0.0589	
Species	0.1542	
Depth	**<0.0001**	
Trt. x Species	0.7909	
Trt. x Depth	0.0529	
Species x Depth	0.6255	

Table 2.15. Mean mineral soil Mehlich 3-P and total-P concentration (mg kg^{-1}) at Mt. Pleasant. Also shown is the Mehlich 3-P percentage of total-P. The 0-15 cm depth represents the average of the three depth classes.

Depth (cm)	Control			Irrigation			Fertilization + Irrigation		
	Loblolly								
	M3-P	total-P	%	M3-P	total-P	%	M3-P	total-P	%
0-3.75	33.0	147.1	22.4	17.5	175.2	10.0	38.8	164.7	23.5
3.75-7.5	32.2	94.4	34.1	12.3	79.0	15.6	48.9	168.5	29.0
7.5-15	30.7	162.9	18.8	10.3	65.1	15.8	41.7	123.2	33.9
0-15	31.9	134.8	25.1	13.4	106.4	13.8	43.1	152.1	28.8
	Sweetgum								
	M3-P	total-P	%	M3-P	total-P	%	M3-P	total-P	%
0-3.75	42.8	149.0	28.7	37.8	188.7	20.0	41.3	103.9	39.7
3.75-7.5	33.3	124.6	26.7	28.7	91.4	31.5	43.1	98.5	43.7
7.5-15	22.0	113.6	19.3	19.5	109.3	17.8	31.8	141.8	22.4
0-15	32.7	129.1	24.9	28.7	129.8	23.1	38.7	114.7	35.3

Table 2.16. Nitrogen budget for SETRES and Mt. Pleasant. Unless otherwise noted, values are kg N ha^{-1}.

	SETRES			Mt. Pleasant					
	Loblolly			Loblolly			Sweetgum		
	Unfertilized	Fertilized	Difference	Unfertilized	Fertilized	Difference	Unfertilized	Fertilized	Difference
Tree	300	575	**275**	277	345	**68**	133	513	**380**
Forest Floor	242	542	**300**	113	233	**120**	41	128	**87**
Mineral Soil	1007	1206	**199**	1539	1833	**294**	1815	2620	**805**
Total	1549	2323	**774**	1929	2411	**482**	1989	3261	**1272**
Added			**1378**			**1128**			**1128**
Retention %			**56**			**43**			**113**

Chapter 3
Effects of fertilization and irrigation on nitrogen, phosphorus, and soluble carbon fractions in loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) foliage and litter

Abstract

Growth of forest plantations in the southeastern U.S. often increases following fertilization. Nutrients are cycled internally by translocation and externally by litter decomposition. The forest floor accumulates N over time while P is more readily released suggesting the forest floor is a sink for N and source of P. Our objective was to determine soluble and residual N and P fractions and soluble carbohydrate and phenol fractions in loblolly pine and sweetgum foliage and litter. Our hypothesis was that the forest floor N sink resulted from fertilization increasing N in the residual fraction and, in contrast, the forest floor was a source for fertilizer P due to fertilization increasing P in the soluble fraction. Study sites were a 25-year old loblolly pine (*Pinus taeda* L.) plantation in NC (SETRES) and 13-year old loblolly pine and sweetgum (*Liquidambar styraciflua* L.) plantations in GA (Mt. Pleasant). Both sites were fertilized with a mixture of macro- and micro-nutrients including N and P. At SETRES, fertilization was on-going and ended at Mt. Pleasant 4 years prior to sampling. Nitrogen and P concentrations in foliage and litter were increased by fertilization at SETRES. At Mt. Pleasant, foliar and litter N and P concentrations were similar across treatments suggesting no residual fertilizer effects. Foliage and litter N were similar among species; however, sweetgum foliage and litter P concentrations were higher than loblolly pine. Residual-N comprised 80-90% of unfertilized and fertilized foliage and litter N and was the fraction most increased by fertilization. In contrast, soluble inorganic-P and organic-P comprised 50-70% of

unfertilized and fertilized foliage and litter P and were the fractions most increased by fertilization. Fertilization served only to magnify the litter residual-N sink and soluble-P source that was otherwise present without fertilization. Soluble carbohydrates were not altered by fertilization but were higher in sweetgum. This was proposed to be related to faster decomposition and the less extensive forest floor in sweetgum compared to loblolly pine.

3.1. Introduction

Growth of forest plantations is often increased following fertilizer applications that increase foliar nutrient concentrations, photosynthetic capacity, and leaf area (Gough et al., 2004). Nitrogen (N) and phosphorus (P) are the most common nutrients that limit tree growth on sandy soils in the southeastern U.S. Fertilization with N and P has been found to increase the growth of loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) on these soils (Albaugh et al., 1998; Cobb et al., 2008). Nutrients are cycled by translocation from foliage prior to senescence (internal) and by decomposition of litter (external). In a study of 20-year old loblolly pine, Switzer and Nelson (1972) suggest that internal cycling supplied 39% and 60% of the trees annual N and P requirements, respectively, while forest floor decomposition supplied 40% and 23%, respectively. In a study of 15-year old loblolly pine, Piatek and Allen (2000) propose that 64% and 70% of N and P, respectively, cycled internally.

In a study that evaluated forest floor and mineral soil N and P pools at two sites, Kiser (Chapter 2) found that the forest floor of both loblolly pine and sweetgum retained more N relative to the mineral soil while more P was retained by the mineral soil. This effect was enhanced by fertilization. Fertilization increased the forest floor N accumulations by 100% to 200%. The forest floor was determined to accumulate N, which supports that N accumulated during loblolly pine Oi horizon decomposition (Piatek and Allen, 2001; Sanchez, 2001). In a study that examined release of N from loblolly pine and sweetgum forest floors, cumulative growing season NH_4-N ranged from 0.3 to 1.0 kg ha^{-1} suggesting supply of N from forest floor decomposition to be very low (Chapter 4). Polglase et al. (1992a) found that N was immobilized during decomposition of loblolly pine Oi horizon and released in only modest amounts from Oe horizons. In contrast to N, P was more readily released from decomposing forest floor horizons increasing the supply of P to trees resulting in a sustained increase in productivity following fertilization. Further investigation revealed that the greater release of P from the forest floor relative to N results from leaching of inorganic P (Polglase et al., 1992b). The increase in litter P from fertilization was found in a soluble fraction suggesting fertilization will result in enhanced supply of P from litter (Polglase et al., 1992c). In addition, this fertilizer response was carried over to soil organic P residues while soil organic N residues remained unaffected (Polglase et al., 1992d). These studies suggest that N is retained in the forest floor due to a slow release of N during decomposition while P release proceeds at a faster rate.

Fertilization can also alter soluble carbon (C) fractions in foliage and litter. Studies suggest fertilization tended to alter phenols instead of carbohydrates. Fertilization of European beech (*Fagus sylvatica* L.) with N resulted in no change in foliar sugar and starch (Balsberg-Pahlsson, 1992). However, foliar phenolic compounds were decreased and this decrease was suggested to explain the greater susceptibility of trees to parasitic attack. Fertilization was found to increase foliar N and decrease foliar phenol of flowering crabapple (*Malus* ‘Sutyzam’) leading to increased herbivory by various caterpillar larvae and decreased resistance to drought stress (Lloyd et al., 2006). In a study of carbohydrates and phenols in loblolly pine and slash pine (*Pinus elliottii* Engelm.), fertilization decreased Oi horizon phenols but did not alter carbohydrates (Polglase et al., 1992c). Nitrogen fertilization was also found to not alter foliar starch in another loblolly pine study (Adams et al., 1986).

The N sink and general lack of a P sink in the forest floor creates the need to determine why these processes occur in order to build a more complete understanding of N and P cycling and the effects of fertilization on cycling. In the study by Polglase et al. (1992c), unlike P, information on the effects of fertilization on N fractions was not provided since an increase in litter N from fertilization was not present. The potential impact of alteration of C fractions creates the need to examine fertilization effects on these fractions as well. Our objective was to determine fertilization effects on soluble and residual N and P fractions and soluble carbohydrate and phenol fractions. Our hypothesis was that the forest floor N sink resulted from fertilization increasing N in the residual fraction and, in contrast, the forest floor was a source for fertilizer P due to fertilization increasing P in the soluble fraction. The hypothesis was tested at two plantations with similar soils and total fertilizer application but different in terms of stand age, species, and fertilizer regime.

3.2. Materials and Methods

3.2.1. Site Descriptions

SETRES

The Southeast Tree Research and Education Site (SETRES) was established in 1985 in order to investigate the effects of irrigation and fertilization on loblolly pine growing on an infertile, excessively-drained sandy soil. The site was located in the Sandhills of Scotland County, NC 17 km north of Laurinburg, NC (34° 54′N, 79° 28′W). Mean annual precipitation was 120 cm and the mean minimum and maximum temperatures were 10.4 and 23.6 °C, respectively. Prior to planting of loblolly pine in 1985, the site was a native longleaf pine (*Pinus palustris* Mill.) forest. The stand was 25-years old when sampling began for this study.

The soil was mapped as the Wakulla series; an infertile, excessively drained, sandy, siliceous, thermic Psammentic Hapludult. The experimental design at SETRES was a 2^2 factorial randomized complete block with 4 blocks (n=4, N=16) with fertilization and irrigation treatments. In 1992, 50 x 50 m treatment plots containing interior 30 x 30 m measurement plots were established. Prior to initial treatment application, plots were thinned and competing vegetation was controlled. Treatments included fertilization (no addition and optimal foliar nutrition based on target foliar nutrient concentration) and irrigation (no addition and addition of 2.5 cm water week^{-1} March to November). Treatment levels included a control (Ct), irrigation (I), fertilization (F), and fertilization x irrigation (FI). Fertilization was conducted by applying a balance of macro- and micro-nutrients to provide optimal foliar nutrition and irrigation was conducted to maintain a soil water content >40% field capacity. Fertilization began in

March 1992 and irrigation began in April 1993 and continues to the present. Fertilization was conducted each spring with a broadcast surface application of various fertilizer sources including granular urea, boron-coated urea, ammonium sulfate, diammonium phosphate, triple super phosphate, potassium chloride, dolomitic lime, Epsom salts, calcium sulfate, and borax (Albaugh et al., 2008). Total N and P applications from 1992 to 2010 were 1490 kg ha^{-1} and 168 kg ha^{-1}, respectively (Table 3.1). Nutrient additions significantly increased tree growth with volume increased by 100% after 13 yrs. of fertilization (Albaugh et al., 1998; Albaugh et al., 2004).

Mt. Pleasant, GA

A similar study designed to evaluate the effects of irrigation and fertilization on a number of pine and hardwood species was established in Mt. Pleasant, GA on an infertile, excessively-drained sandy soil. The site was located in the southeastern Georgia coastal plain in Wayne Co. (31°23′N, 81°43′W). Mean annual precipitation was 127 cm and the mean minimum and maximum temperatures were 12.0 and 26.0 °C, respectively. Prior to establishment of the study in 1997, the site was a slash pine plantation. After harvesting of the previous pine stand, the site was cleared, disked, limed, and planted with separate plots containing loblolly pine, slash pine, sweetgum, and sycamore (*Platanus occidentalis* L.). This paper includes data from only the loblolly pine and sweetgum plots. The stands were 13-years old when sampling began for this study. The soil was mapped as the Klej series; a mesic, coated Aquic Quartzipsamment.

The study was a randomized complete block design with 3 blocks (n=3, N=18). In 1997, 35 x 25.5 m treatment plots containing interior 22.2 x 13.5 m measurement plots were established. Treatment levels included a control (Ct), irrigation (I), and 3 levels of fertilization combined with irrigation (FI). Only the high fertilization rate was evaluated in this study where annual nutrient additions averaged 113 N, 17 P, and 62 K kg ha^{-1} yr^{-1} (Cobb et al., 2008). Fertilization and irrigation treatments began in 1997. Irrigation was conducted by applying 3.05 cm water $week^{-1}$ for 28 weeks $year^{-1}$ (April to November) from 1997 to 2003. Fertilization from 1997 to 2003 was conducted by applying nutrients in solution in the irrigation water throughout the 28 weeks of irrigation each year. Nitrogen was supplied as ammonium nitrate (NH_4NO_3). From 2004 to 2006, surface applications of N, P, and micronutrients were conducted in the spring. Nitrogen and P were applied as diammonium phosphate (DAP) and urea. Total N and P applications from 1997 to 2006 were 1128 kg ha^{-1} and 169 kg ha^{-1} (Table 3.2), respectively, which were comparable to total fertilizer N and P applied at SETRES. Nutrient additions increased aboveground biomass (Cobb et al., 2008). After 9 yrs. of fertilization, loblolly pine volume increased by 30% and sweetgum volume increased by 300%.

3.2.2. Sampling

At both SETRES and Mt. Pleasant, foliage samples were collected in mid-August 2010 from the upper 1/3 of the canopy at 3 randomly selected trees within each plot. Loblolly pine samples were taken from the first flush of the previous year’s growth. Litter samples were obtained at SETRES by collecting subsamples from existing litterfall traps (8 m^2 $plot^{-1}$) that are part of ongoing studies at the site. Litter samples were collected at Mt. Pleasant in litterfall traps placed at two random locations in each plot (0.51 m^2 $plot^{-1}$). Litter collection was timed to obtain a sample no more than one week after the litter had fallen in order to minimize possible leaching of water soluble fractions. Litter was collected at Mt. Pleasant and SETRES on October 23, 2010 and October 24, 2010, respectively. Foliage and litter samples were oven-dried at 60 ^{0}C for approximately 1 week and ground using a Thomas-Wiley Model 4 mill (Thomas Scientific, USA).

3.2.3. Chemical Analysis

Fractionation of soluble and residual N, P, and C was carried out according to the procedures of Kedrowski (1983) and Polglase et al. (1992c). In brief, total-N concentration in the litter was determined by dry-combustion with a Vario MAX CN analyzer (Elementar, Hanau, Germany). Total-P in the litter was determined by dry-ashing at 500 ^{0}C followed by dissolution with 6N HCl. Phosphorus in solution was analyzed with inductively-coupled plasma atomic emission spectrophotometry (ICP-AES) on a Varian Vista MPX **(**Varian, Palo Alto, CA, USA). A sequential cold 0.30 M trichloroacetic acid (TCA) and hot 0.15 M TCA extraction was performed on duplicate 0.30 g samples to extract soluble N, P, sugars, and phenols. The residual concentration was calculated as the total concentration minus the total soluble fraction concentration. To fractionate soluble inorganic and organic-N and P, soluble total-P and N in the extracts were determined by H_2SO_4/H_2O_2 digestion and analysis with ICP-AES and the indophenol blue method (Keeney and Nelson, 1982), respectively. Inorganic-N and P in the extracts were determined with the indophenol blue method and the procedure of Murphy and Riley (1962), respectively. Similar to the results of Polglase et al. (1992c), initial analysis revealed inorganic-N to be an insignificant component and was dropped from further analysis. The soluble organic P fraction was calculated as the total soluble P fraction minus the soluble inorganic P fraction. Carbohydrates were determined by the procedure of Dubois et al. (1956) and were expressed as glucose equivalents. Carbohydrates were reported as soluble sugar, soluble starch, and the sum of these two fractions, total soluble carbohydrate. Phenols were determined by the Prussian blue method (Price and Butler, 1977) and were expressed as tannic acid equivalents. Phenols were reported as soluble phenol, soluble tannin, and the sum of these two fractions, total soluble phenol. According to Chapin and Kedrowski (1983), organic-P soluble in cold TCA is phytate and other ester P while organic-P soluble in hot TCA is phytate and nucleic acid P. Organic-N soluble in cold TCA is amino acid N while organic-N soluble in hot TCA is nucleic acid N. Residual N is protein N. Sugars and phenols soluble in cold TCA represent soluble components while in hot TCA, soluble sugars represent starches and soluble phenols represent tannins.

3.2.4. Statistical Analysis

Treatment and species effects on N, P, and C fractions were determined with a general linear model (PROC GLM, SAS Institute Inc., Cary, NC, USA). At Mt. Pleasant, trees in the block 3 loblolly FI plot were killed by the Ips beetle (*Ips spp.*). Therefore, samples could not be collected in this plot and n=2 for loblolly FI while n=3 for all other species/treatment combinations. At Mt. Pleasant, analysis was conducted on the Type III sums of squares due to the unbalanced design. Pairwise comparisons were conducted with the Tukey test. Prior to analysis, normality of the data was tested and log transformations were used when necessary.

3.3. Results

3.3.1. Nitrogen

At SETRES, fertilization increased all N fractions and irrigation decreased nucleic-N in foliage (Fig. 3.1A; Table 3.3). However in litter, only residual and total-N were higher in fertilized treatments suggesting amino-N and nucleic-N were re-translocated prior to senescence (Fig. 3.1B; Table 3.3).

At Mt. Pleasant, foliar amino-N was higher in loblolly than in sweetgum. (Fig. 3.1C; Table 3.4). Foliar nucleic-N in loblolly I was higher than all sweetgum treatments. Sweetgum Ct foliar nucleic-N was lower than sweetgum I. Foliar and litter residual-N and total-N did not vary by treatment or species (Table 3.4; Fig. 3.1C and D). Litter amino-N and nucleic-N were higher in loblolly than in sweetgum.

The residual, protein-N fraction contributed 80-90% of total-N concentration for both loblolly pine and sweetgum foliage and litter. Foliar total-N concentration at SETRES was >12.0 g kg^{-1} in fertilized treatments and approximately 8.0 g kg^{-1} in unfertilized treatments. Foliar total-N concentration in all treatment/species combinations at Mt. Pleasant had fallen to 8.0 g kg^{-1} from concentrations of 12.7 g kg^{-1} and 17.0 g kg^{-1} for loblolly and sweetgum, respectively, reported by Cobb et al. (2008) during fertilization. Fertilization ended at Mt. Pleasant 4 years prior to sampling and the foliar N response observed during fertilizer applications by Cobb et al. (2008) had disappeared. Foliar total-N concentration in all treatment/species combinations at Mt. Pleasant and in unfertilized loblolly pine at SETRES was similar at approximately 8.0 g kg^{-1}. Litter total-N concentration was approximately 50% of foliar total-N concentration in SETRES unfertilized treatments and in all treatment/species combinations at Mt. Pleasant. In SETRES fertilized treatments, litter total-N concentration was approximately 30% of foliar total-N concentration suggesting greater translocation.

3.3.2. Phosphorus

At SETRES, fertilization increased foliar inorganic-P, organic-P, and total-P (Fig. 3.2A; Table 3.3). Similar results were found for SETRES litter with the addition of higher residual-P in SETRES fertilized treatments (Fig. 3.2B; Table 3.3).

At Mt. Pleasant, only species effects were found for all foliar and litter P fractions (Table 3.4; Fig. 3.2C and D). All P fractions were higher in sweetgum compared to loblolly. Litter organic-P exhibited additional responses (Table 3.4). Litter organic-P in the sweetgum I was higher than all other treatment/species combinations.

Foliar total-P concentrations were higher at Mt. Pleasant compared to SETRES. Loblolly and sweetgum foliar total-P at Mt. Pleasant was approximately 1500 mg kg^{-1} and 2200 mg kg^{-1}, respectively. At SETRES, foliar total-P concentration was approximately 700 mg kg^{-1} and 975 mg kg^{-1} for unfertilized and fertilized treatments, respectively. At Mt. Pleasant, the inorganic-P and residual-P fractions contributed the majority of foliage and litter total-P concentration in loblolly pine and sweetgum (Fig 3.2C and D). The exception was that in sweetgum, the proportion of organic-P increased from foliage to litter (Fig. 3.2D). The same was generally true at SETRES with the exception of a greater contribution of organic-P in the fertilized treatments (Fig. 3.2A and B). At SETRES, litter concentration was approximately 25% of foliar total-P concentration. At Mt. Pleasant, loblolly litter concentration was approximately 50% of foliar total-P concentration and sweetgum litter concentration was approximately 90% of foliar total-P concentration. Translocation of P appeared to be higher at SETRES. However, this could have arisen from timing of sample collection. Litter samples were collected at SETRES the day after collection at Mt. Pleasant. SETRES was located at higher latitude, litterfall occurred earlier, and litter was on the ground longer compared to Mt. Pleasant. Although it is unknown, precipitation could have occurred at SETRES and not at Mt. Pleasant subjecting litter to leaching of P.

3.3.3. Soluble Carbohydrate

At SETRES, foliar carbohydrate fractions did not differ among treatments (Tables 3.3). However, irrigation increased soluble sugar and total soluble carbohydrate in SETRES litter leading to the highest concentration in the FI treatment (Table 3.3; Fig. 3.3B). At Mt. Pleasant, a TxS interaction effect indicated that soluble sugar and total soluble carbohydrate in loblolly pine foliage increased from Ct to I to FI whereas in sweetgum, the opposite trend was observed (Table 3.3; Fig. 3.3C). In the litter, this effect had disappeared. Sweetgum litter soluble sugar and total soluble carbohydrate were higher than in loblolly pine (Table 3.4; Fig. 3.3D).

Foliar total soluble carbohydrate concentration was comparable across sites and species and ranged from 120 g kg^{-1} to 150 g kg^{-1} (Fig. 3.3A and C). Litter total soluble carbohydrate concentration results were also comparable across sites and species and ranged from 80 g kg^{-1} to 90 g kg^{-1} (Fig. 3.3B and D). Litter total soluble carbohydrate concentration was approximately 65% of foliar total soluble carbohydrate concentration at both sites.

3.3.4. Soluble Phenol

At SETRES, foliar soluble tannin and total soluble phenol were higher in unfertilized treatments (Fig. 3.4A; Table 3.3). Litter soluble phenol, soluble tannin, and total soluble phenol concentration were also higher in unfertilized treatments (Fig. 3.4B;

Table 3.3). Also in the litter, soluble phenol was higher in the FI treatment than the F treatment as indicated by the FxI interaction effect.

At Mt. Pleasant, a TxS interaction effect was observed for foliar soluble phenol, soluble tannins, and total soluble phenol (Table 3.4). These fractions increased from loblolly Ct to I to FI (Fig. 3.4C). These fractions were similar among loblolly FI and sweetgum Ct and I and lowest in sweetgum FI (Fig. 3.4C). Also, these fractions were equivalent among loblolly Ct and sweetgum FI. This trend had disappeared in litter and only species effects were observed for litter soluble phenol, soluble tannins, and total soluble phenol (Fig. 3.4D; Table 3.4). These variables were higher in sweetgum than in loblolly.

Foliar total soluble phenol concentration in loblolly FI and sweetgum Ct and I treatments at Mt. Pleasant was nearly 4 times greater than that observed at SETRES (Fig. 3.4). However, loblolly litter total soluble phenol concentration at Mt. Pleasant was approximate to SETRES while sweetgum total soluble phenol concentration remained 2 times greater. At SETRES, litter total soluble phenol concentration was 61% to 77% of foliar total soluble phenol concentration. At Mt. Pleasant, loblolly litter total soluble phenol concentration was 15% to 54% of foliar total soluble phenol concentration. Sweetgum litter total soluble phenol concentration was 48% to 200% of foliar total soluble phenol concentration with the accumulation in the FI treatment.

3.4. Discussion

The hypothesis that the forest floor was a sink for fertilizer N due to fertilization increasing N in the residual fraction and a source for fertilizer P due to fertilization increasing P in the soluble fraction was accepted. It is important to note that residual-N and soluble-P comprised the majority of total-N and -P in unfertilized treatments as well. This suggests that fertilization serves only to magnify the litter residual-N sink and soluble-P source that would otherwise be present without fertilization. Polglase et al. (1992c) also found residual-N comprised the majority of litter total-N in a study where N was not increased by fertilization.

An accumulation of P during decomposition of loblolly pine Oi horizon reported by Piatek and Allen (2001) and Sanchez (2001) was inconsistent with our results and those of Polglase et al. (1992) who suggested this material was a source for P. Conflicting results likely arose from differences in methodology. We used the methodology of Polglase et al. (1992c) and determined soluble P by extraction with TCA. In the studies by Piatek and Allen (2001) and Sanchez (2001), litterbags were incubated for periods of 2 and 3 years, respectively. Unlike the studies that assessed extractable P, each year, newly fallen litter was allowed to collect on top of the litterbags. This resulted in an input of P to the litterbags and likely resulted in the observance of a net accumulation of P. Achat et al. (2010) suggest microbes immobilize phosphate ions and inorganic-P was found to be readily released from loblolly pine litter by leaching (Polglase et al., 1992b). A decline in C inputs from litter was suggested to decrease N assimilation by microbial biomass (Hart et al., 1994; Bradley et al., 2000). This suggests that annual C inputs from litter aided microbes in the assimilation of P. We propose that in the litterbag incubation studies, P was released into the litterbags from the newly fallen litter each year and immobilized in the microbial biomass. This process was aided by C inputs from the newly fallen litter.

In a study of fertilization effects on *Eucalyptus globulus* Labill. foliage and litter N and P fractions, Hooda and Weston (1999) found that fertilization effects were sustained on P but not N at 4 years after the last fertilizer application. Kiser (Chapter 4) suggested that mineral soil N availability was still elevated in fertilized plots at Mt. Pleasant 3 years after fertilization ended. Mineral soil N availability was elevated due to a sustained increase in supply from the forest floor. Additionally, it was also suggested that mineral soil N availability was declining over time as indicated by differences in N cycling processes among Mt. Pleasant and a site still receiving fertilizer applications. At 4 years following cessation of fertilization in this study, foliar N was no longer elevated. This was in agreement with the findings of Hooda and Westion (1999) and also suggests that N availability has continued to decline in fertilized plots at Mt. Pleasant from 3 years after fertilization cessation to 4 years after fertilization cessation.

At SETRES, P fertilizer was last applied in 2002, 8 years prior to sampling in this study. Fertilization was reported to result in increased mineral soil P at SETRES (Kiser, Chapter 2). Results suggest that this mineral soil pool supplied higher P to the trees as evidenced by sustained elevated P concentrations in foliage. The allocation of at least 50% of litter P in soluble fractions suggests that P will be readily released from the litter into the mineral soil. The high proportion (80-90%) of litter N in the residual fraction suggests the opposite for N. A sustained growth response to P fertilization has been observed in numerous studies (Ballard, 1978; Gentle et al., 1986; Comerford et al., 2002; Crous et al., 2007; Everett and Palm-Leis, 2009). Results suggest this sustained growth response to P fertilization was due to efficient cycling of P from the mineral soil into the foliage and then, release from the litter back into the soil to begin the cycle again. Our results regarding N and P cycling were in agreement with cycling processes outlined by Switzer and Nelson (1972) who proposed that while N cycling was predominantly biochemical, cycling within the plant and through forest floor decomposition, P cycling was also biogeochemical, biochemical with the addition of cycling through the mineral soil.

Fertilization has been reported to not alter foliage and litter carbohydrate fractions (Adams et al., 1986; Balsberg-Pahlsson, 1992; Polglase et al., 1992c). Results from SETRES were consistent with these studies. Ludovici et al. (2002) suggested that fertilization at SETRES decreased foliar starch. However, the methodology used by Ludovici et al. (2002) differed from that used in this study in that we determined soluble starch not total starch. Decreased foliar phenol from fertilization at SETRES reported at stand age 9 following 2 years of fertilization reported by Booker and Maier (2001) was still present at stand age 25 following 18 years of fertilization.

The most interesting C fraction results were found at Mt. Pleasant. To reiterate briefly, foliar soluble sugar, phenol, and tannin increased from loblolly pine Ct to I to FI treatments. In sweetgum, the opposite trend was generally observed. Sweetgum also differed from loblolly pine in that Ct and I treatments were similar. In litter, the trend found in foliage had disappeared and these C fractions were higher in sweetgum than in loblolly pine. Osono et al. (2003) suggested the growth of fungal hyphae on litter was dependent on the availability of soluble carbohydrates. Higher soluble sugar concentrations in sweetgum likely explained the faster decomposition of the sweetgum forest floor litter. Fresh litter inputs can also produce a positive priming effect (Kalbitz et al., 2007) stimulating decomposition of the existing forest floor resulting in less forest floor mass in sweetgum relative to loblolly pine. Although litter production in FI treatments was 500 kg ha^{-1} greater in sweetgum than in loblolly pine (data not shown), Kiser (Chapter 2) reported that forest floor mass of loblolly pine was 15.1 Mg ha^{-1} while sweetgum forest floor mass was 10.8 Mg ha^{-1}. In addition, unlike loblolly pine, the Oa horizon was absent in the sweetgum forest floor. At SETRES, litter N concentration was higher in fertilized treatments while no difference was found in litter soluble sugar, soluble starch, and total soluble carbohydrates. Studies have reported that endogenous N availability does not alter litter decomposition rates (Prescott, 1995; Sanchez, 2001). Based on our results and those of Osono et al. (2003), it is possible that higher decomposition rates observed in hardwoods relative to pines are driven by higher soluble sugar in litter. In other words, decomposition is limited by the availability of C not N. This was supported by Klotzbucher et al. (2011) who suggest decomposition of recalcitrant litter lignin required availability of easily decomposable C sources.

The divergent trend observed in loblolly pine and sweetgum foliar soluble sugars, phenols, and tannins may be explained by differences in plant responses to water stress. However, we can only offer speculation. In loblolly pine, tree size and foliage mass increased from Ct to I to FI treatments (Cobb et al., 2008). In sweetgum, tree size and foliage mass were highest in FI and more or less equal in Ct and I treatments. Water demand likely follows the same trend. It is important to remember that irrigation ended at Mt. Pleasant in 2003. Water stress was therefore, assumed to be influenced only by precipitation and would increase with water demand. Shure et al. (1998) found that a number of hardwood species decreased foliar phenols in response to water stress. Induced drought in European white birch (*Betula pendula* Roth.) contributed to thickening of the leaf epidermis, reduced starch, and increased tannin depositions (Paakkonen et al., 1998). Results of these studies suggest that hardwoods respond to water stress by decreasing soluble leaf compounds in order to thicken the epidermis. Conifers, however, appear to respond to water stress by increasing soluble compounds and leaf osmotic potential since they already possess a thick epidermis. Green et al. (1994) found that loblolly pine increased foliar hexose in response to water stress. Horner (1990) found that foliar tannin concentration of Douglas-fir [*Pseudotsuga menziesii* (Mirb.) Franco, Pinaceae] increased with increasing water stress. Increases in foliar N have been accompanied by decreases in foliar phenols and have been reported to increase the susceptibility of trees to attack from insects and parasites (Balsberg-Pahlsson, 1992; Lloyd et al., 2006). Results of these studies may offer an explanation for the Ips beetle (*Ips spp.*) infestation in one of the loblolly FI plots that led to almost 100% mortality.

3.5. Conclusions

In forest ecosystems, nutrients taken up by trees are returned to the soil in litter. Cycling of N will proceed slowly relative to P due to residual-N comprising the majority of litter N. Cycling of P will proceed quickly due to soluble-P comprising the majority of litter P. This was found in both unfertilized and fertilized systems suggesting fertilization only serves to magnify the forest floor N sink and P source. While fertilization increases litter N, decomposition of litter is likely not altered since fertilization does not alter soluble carbohydrates. Higher soluble carbohydrate in sweetgum litter explains faster decomposition of sweetgum litter and a less extensive forest floor relative to loblolly pine.

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3.7. Tables and Figures

Table 3.1. Nutrient additions at SETRES from 1992 to 2010. Granular fertilizer was applied to the surface on each application date.

Application Date	N	P	K	Ca	Mg	S	B
	kg ha^{-1}						
Mar-92	224	56	112	0	0	0	0
Apr-92	0	0	0	134	56	0	0
Jun-92	0	0	0	0	0	0	2
Mar-93	0	28	0	0	0	0	0
Apr-93	26	22	21	0	0	0	0
Jun-93	0	0	92	0	56	120	0
Aug-93	56	0	0	0	0	0	0
Mar-94	112	0	0	0	0	0	0
Mar-95	56	28	56	24	34	74	0
May-95	0	0	0	0	0	0	1
Mar-96	112	11	56	10	0	15	0
Apr-96	0	0	0	0	0	0	1
Apr-97	135	0	0	0	0	0	0
Mar-98	56	6	0	0	0	0	0
Mar-99	69	0	0	0	0	0	0
Mar-00	56	6	0	0	0	0	0
Apr-01	56	6	0	0	0	0	0
Apr-02	56	6	0	0	0	0	0
May-02	0	0	56	0	0	0	0
Apr-03	56	0	0	0	0	0	0
Mar-04	56	0	0	0	0	64	0
Mar-05	84	0	0	0	0	0	1
Mar-06	56	0	0	0	0	64	0
Apr-07	56	0	0	0	0	0	0
Apr-08	56	0	0	0	0	0	0
Apr-09	56	0	0	0	0	0	0
Apr-10	56	0	56	0	0	0	0
Total	1490	168	449	168	146	337	6

Table 3.2. Nutrient additions at Mt. Pleasant from 1997 to 2006. From 1997 to 2003, soluble fertilizer was applied over a 28 week period in the irrigation water. From 2004 to 2006, a single surface application of granular fertilizer was conducted in the spring.

Year	N	P	K	Cu	Mn	Mo	S	B	Fe
	kg ha^{-1}								
1997	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
1998	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
1999	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
2000	170	26	94	0.0	0.0	0.0	0.0	0.0	0.0
2001	51	8	28	0.0	0.0	0.0	0.0	0.0	0.0
2002	116	17	64	0.0	0.0	0.0	0.0	0.0	0.0
2003	116	17	64	0.0	0.0	0.0	0.0	0.0	0.0
2004	112	17	62	0.6	1.4	0.0	1.4	0.6	3.4
2005	112	17	62	0.7	1.6	0.5	0.0	0.7	3.8
2006	112	17	62	0.7	1.6	0.5	0.0	0.7	3.8
Total	1128	169	621	2.0	4.6	1.0	1.4	2.0	11.0

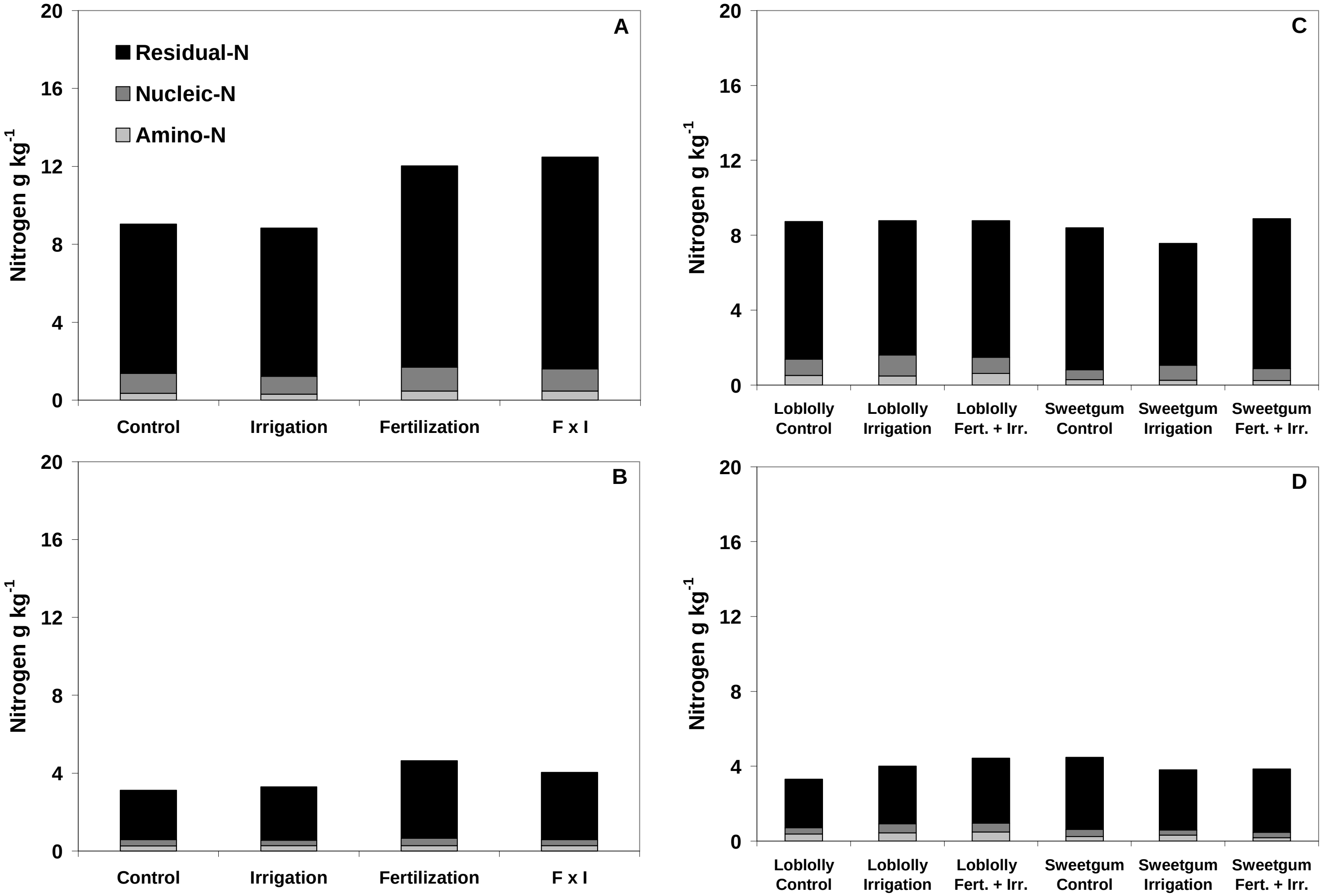

Fig. 3.1. Mean nitrogen fraction concentrations for SETRES foliage (A), SETRES litter (B), Mt. Pleasant foliage (C), and Mt. Pleasant litter (D). Amino-N is cold 0.30M extractable and nucleic-N is hot 0.15M extractable. Residual-N is protein-N and was calculated as the difference between total extractable-N and total-N.

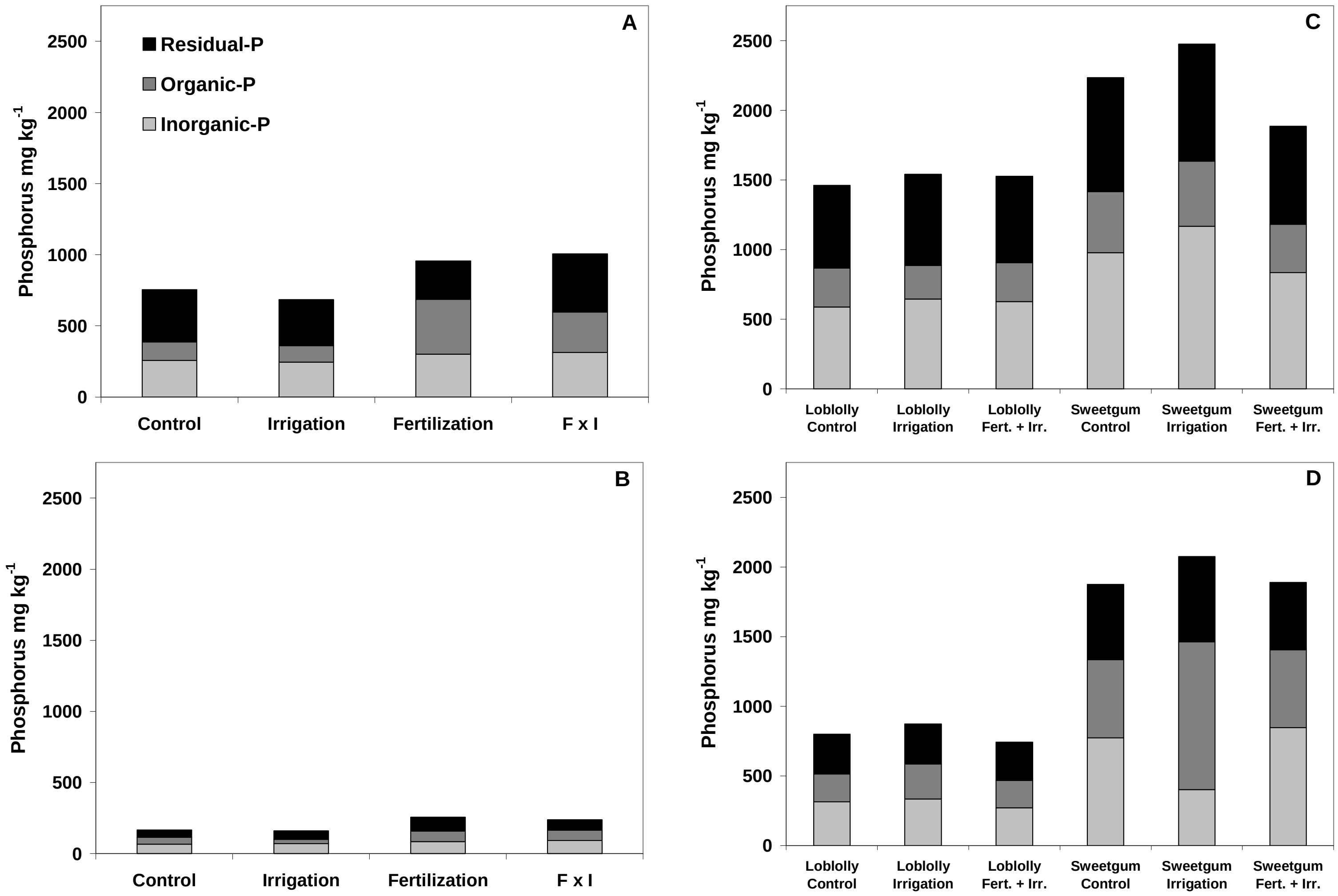

Fig. 3.2. Mean phosphorus fraction concentrations for SETRES foliage (A), SETRES litter (B), Mt. Pleasant foliage (C), and Mt. Pleasant litter (D). Cold and hot extractable inorganic-P and organic-P fractions were pooled. Residual-P was calculated as the difference between total extractable-P and total-P.

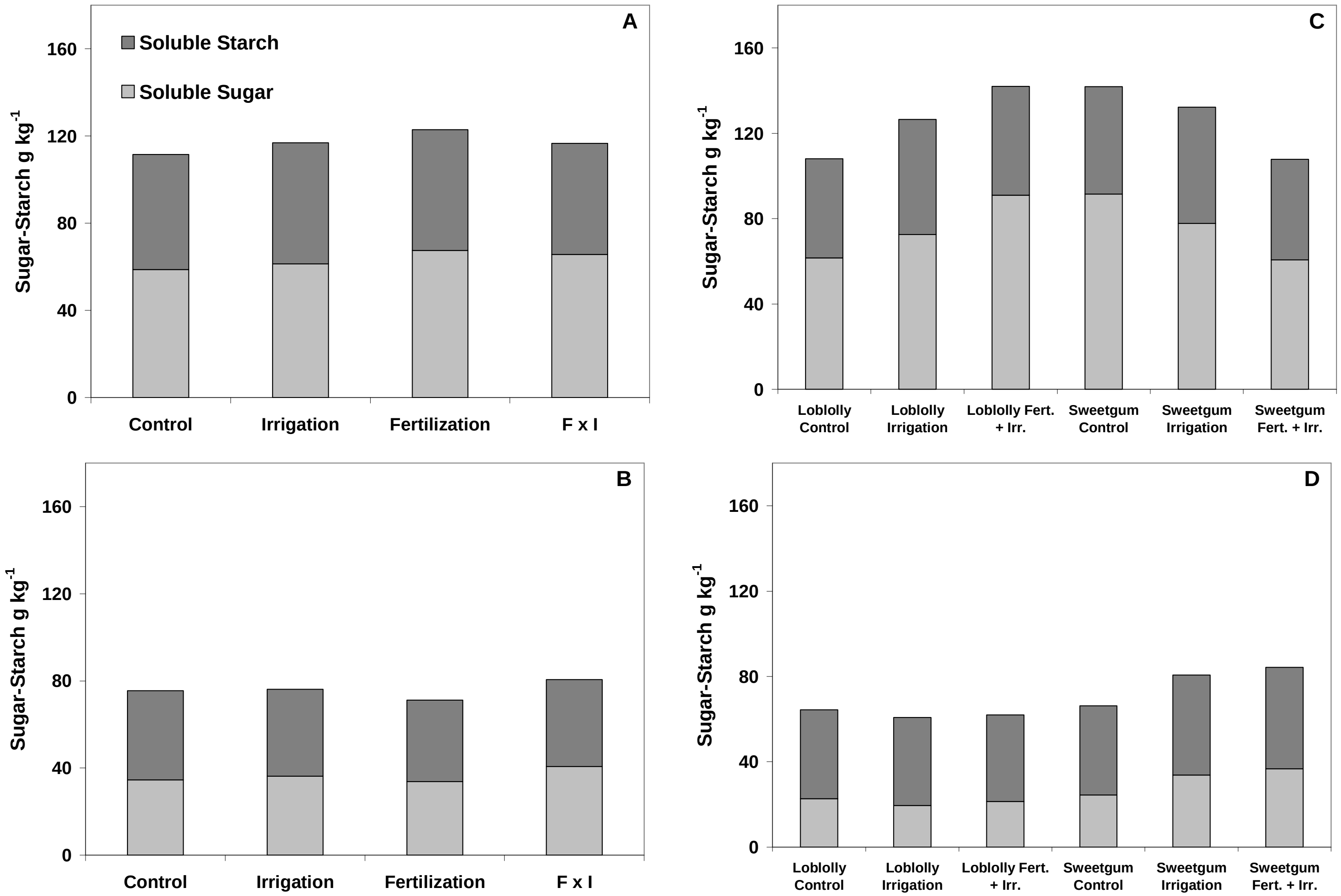

Fig. 3.3. Mean carbohydrate fraction concentrations for SETRES foliage (A), SETRES litter (B), Mt. Pleasant foliage (C), and Mt. Pleasant litter (D). Soluble sugar is cold 0.30M extractable and soluble starch is hot 0.15M extractable.

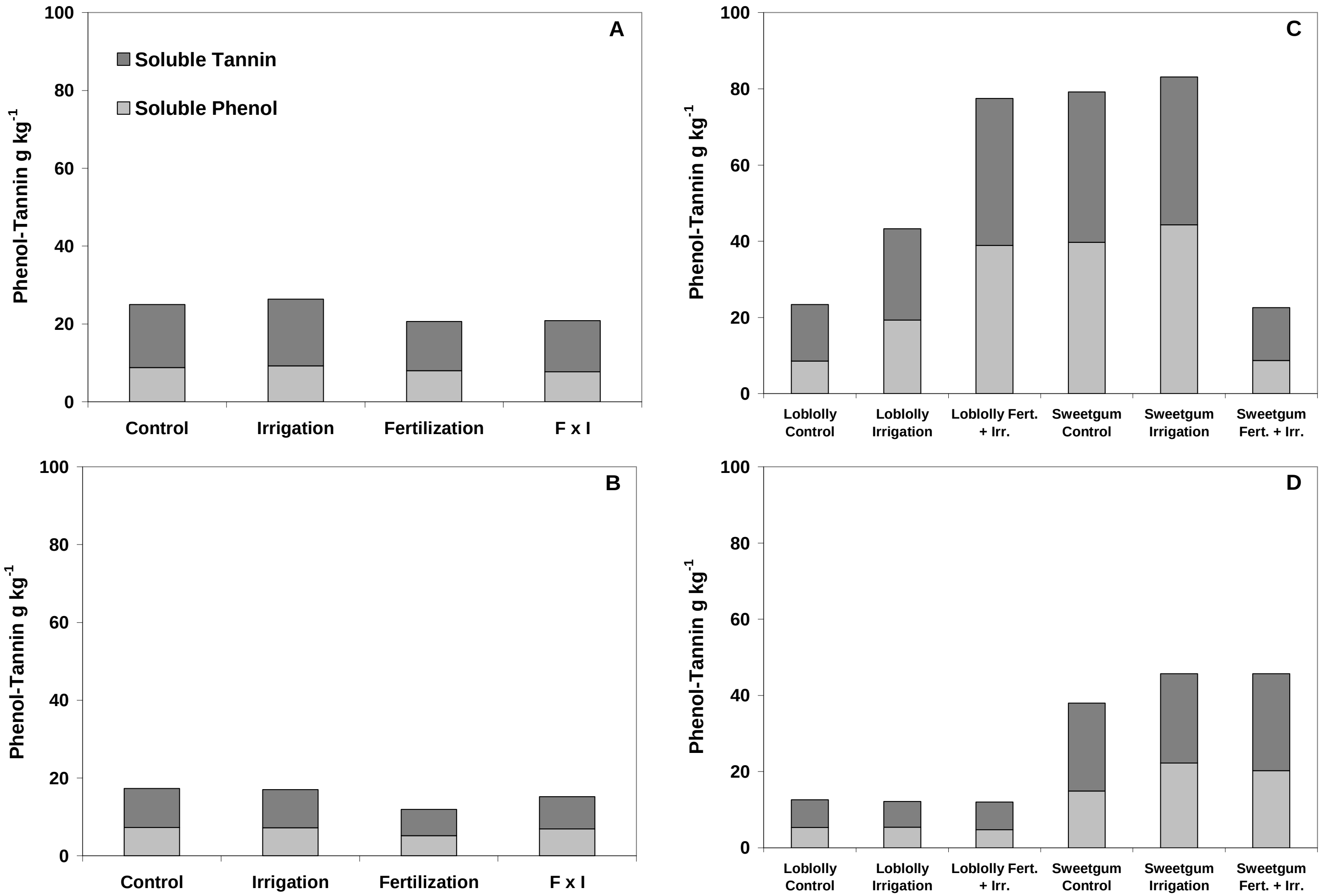

Fig. 3.4. Mean phenol and tannin fraction concentrations for SETRES foliage (A), SETRES litter (B), Mt. Pleasant foliage (C), and Mt. Pleasant litter (D). Soluble phenol is cold 0.30M extractable and soluble tannin is hot 0.15M extractable.

Table 3.3. SETRES foliage and litter p-values for general linear model effects of fertilization, irrigation, and the interaction (FxI). Statistically significant p-values are shown in bold. † Indicates statistical analysis performed on log transformed data.

	Foliage	Litter	Foliage	Litter	Foliage	Litter	Foliage	Litter
	Amino-N		Inorganic-P		Soluble Sugar		Soluble Phenol	
Fertilization	**0.0085**	0.7759	**0.0012**	**<0.0001**	0.0972	0.1787	0.0555	**† 0.0138**
Irrigation	0.5896	0.8866	0.9692	**0.0139**	0.9240	**0.0080**	0.8723	0.0501
F x I	0.6524	0.8866	0.3200	0.4650	0.5350	0.0642	0.5104	**0.0429**
	Nucleic-N		Organic-P		Soluble Starch		Soluble Tannin	
Fertilization	**<0.0001**	0.2231	**0.0006**	**0.0068**	0.7002	0.2398	**0.0001**	**0.0025**
Irrigation	**0.0122**	0.1476	0.1921	0.3247	0.7848	0.6075	0.2634	0.2869
F x I	0.7828	0.5507	0.3170	0.4118	0.2319	0.2641	0.7208	0.1729
	Residual N		Residual-P		Total Carbohydrate		Total Phenol	
Fertilization	**<0.0001**	**0.0001**	0.8969	**0.0009**	0.3365	0.9950	**0.0004**	**0.0042**
Irrigation	0.3853	0.3407	0.3869	0.3065	0.9392	**0.0279**	0.4291	0.1476
F x I	0.3078	**0.0605**	0.1161	0.0309	0.3006	**0.0463**	0.5394	0.0980
	Total-N		Total-P					
Fertilization	**<0.0001**	**0.0002**	**<0.0001**	**<0.0001**				
Irrigation	0.6779	0.2934	0.7272	0.2542				
F x I	0.3138	0.0741	0.0542	0.5129				

Table 3.4. Mt. Pleasant foliage and litter p-values for general linear model effects of treatment, species, and the interaction (TxS). Statistically significant p-values are shown in bold. † Indicates statistical analysis performed on log transformed data.

	Foliage	Litter	Foliage	Litter	Foliage	Litter	Foliage	Litter
	Amino-N		Inorganic-P		Soluble Sugar		Soluble Phenol	
Treatment	0.1880	0.6253	0.2269	† 0.1944	0.9536	0.4233	† 0.2158	0.2044
Species	**<0.0001**	**0.0120**	**0.0014**	**0.0004**	0.6659	**0.0124**	0.0589	**<0.0001**
T x S	**0.0277**	0.4508	0.4416	0.0520	**0.0066**	0.2295	**0.0004**	0.1875
	Nucleic-N		Organic-P		Soluble Starch		Soluble Tannin	
Treatment	**0.0016**	0.8313	0.5373	**0.0186**	0.1938	0.8038	0.3660	0.8791
Species	**<0.0001**	**0.0018**	**0.0030**	**<0.0001**	0.8151	0.2156	0.1356	**<0.0001**
T x S	0.7729	**0.0111**	0.3526	**0.0456**	0.6896	0.6485	**0.0007**	0.8290
	Residual N		Residual-P		Total Carbohydrate		Total Phenol	
Treatment	0.5149	0.6944	0.2947	0.2327	0.7642	0.6026	† 0.2241	0.6303
Species	0.9912	0.1417	**0.0037**	**<0.0001**	0.6767	**0.0467**	0.0771	**<0.0001**
T x S	0.7662	0.1293	0.4853	0.2306	**0.0193**	0.3848	**0.0004**	0.5192
	Total-N		Total-P					
Treatment	0.6528	0.7418	0.3033	0.7789				
Species	0.4213	0.6847	**0.0016**	**0.0002**				
T x S	0.8154	0.0773	0.4427	0.9598				

Chapter 4
Residual effects of nitrogen fertilization on nitrogen availability in loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) plantations

Abstract

Fertilization with N is utilized in the southeastern U.S. to improve growth of forest plantations. Nitrogen cycling in forest ecosystems following N fertilization determines the long-term impact of fertilization on N availability and tree growth. The movement of N from the forest floor to the mineral soil is a key component of the N cycle. Our objective was to determine whether higher N in the forest floor from fertilization resulted in increased release of N from the forest floor and increased mineral soil N availability. Our hypothesis was that higher N in the forest floor did not result in increased release of N from the forest floor and elevated mineral soil N availability due to studies suggesting N accumulations in the forest floor. Forest floor and mineral soil N availability were measured at a 23-year old loblolly pine (*Pinus taeda* L.) plantation in NC (SETRES) and 12-year old loblolly pine and sweetgum (*Liquidambar styraciflua* L.) plantations in GA (Mt. Pleasant) growing on sandy soils. At SETRES, 1378 kg N ha^{-1} were applied over 16 years with 56 kg N ha^{-1} applied to the surface of the forest floor as granular urea-N. At Mt. Pleasant, 1128 kg N ha^{-1} were applied over 9 years with the final application conducted 3 years prior to the measurement period. Fertilizer N and N released from decomposition of the forest floor were assessed with ion exchange membranes (IEM) placed at the Oa horizon/mineral soil interface. Mineral soil extractable NH_4-N and NO_3-N and mineralization and nitrification were measured using sequential soil cores. At SETRES, previous fertilizer applications that included an

application of 56 kg N ha^{-1} in the spring before sampling elevated N at the forest floor/mineral soil interface for 5 months and resulted in increased cumulative NH_4-N and NO_3-N. This application resulted in only small increases in mineral soil extractable NH_4-N and NO_3-N and suggested that fertilizer N entering the mineral soil from the forest floor was first immobilized in the microbial biomass before being released. The release was detected as a large increase in cumulative nitrification. At Mt. Pleasant, N detected at the forest floor/mineral soil interface was probably derived solely from forest floor decomposition. NH_4-N release from the forest floor was higher in fertilized treatments but was of a much smaller magnitude than at SETRES. Mineral soil NO_3-N was elevated in fertilized treatments. The increase in mineral soil NO_3-N was disproportionate to the increase in NH_4-N release from the forest floor suggesting release of DON from the forest floor may have also contributed to elevated mineral soil NO_3-N. Nitrogen fertilization had residual effects on increased N availability from forest floor decomposition and in the mineral soil at 3 years following cessation of fertilization. However, the increase was small and it is uncertain whether the effect was biologically significant and resulted in increased foliar N.

4.1. Introduction

In the southeastern U.S., forest plantations are frequently established on soils that are sandy textured and infertile. Fertilization with nitrogen (N) is necessary to increase tree growth on these soils (Allen, 1987; Albaugh et al., 2007; Fox et al., 2007). As a stand matures, N content increases in the trees and forest floor (Switzer and Nelson, 1972). On these sandy textured soils, fertilization results in greater accumulations of N in the forest floor relative to the mineral soil (Miller, 1981; Piatek and Allen, 2001; Chapter 2).

Nitrogen fertilization on soils with low levels of total-N can significantly increase the amount of N within the ecosystem. Fertilization with N was reported to increase leaf area and foliar N concentration for loblolly pine (*Pinus taeda* L.) (Albaugh et al., 1998; Cobb et al., 2008) and sweetgum (*Liquidambar styraciflua* L.) (Cobb et al., 2008) resulting in a growth response to fertilization. In a study that evaluated the effects of fertilization on forest floor and mineral soil N from these same two sites, fertilization increased forest floor N more than mineral soil N (Chapter 2). In the 23-year old loblolly pine plantation (SETRES), fertilization with 1378 kg N ha^{-1} increased forest floor N from 240 to 540 kg ha^{-1}. In the 12-year old plantations (Mt. Pleasant), fertilization of loblolly pine and sweetgum with 1128 kg N ha^{-1} increased forest floor N from 89 to 233 kg ha^{-1} and 28 to 128 kg ha^{-1}, respectively.

Due to these large accumulations of N in the forest floor, the need to quantify the release of N from this forest floor pool arises in order to determine whether this pool increased availability of N for the trees. The two forest plantation fertilization and irrigation experiments examined in this study provide an opportunity to assess N release from the forest floor that has accumulated during more than a decade of stand development and N fertilizer applications. Our objective was to determine whether higher N in the forest floor from fertilization resulted in increased release of N from the forest floor and increased mineral soil N availability. Increased mineral soil N availability was assumed to originate from forest floor decomposition N release due to no increase in mineral soil N from fertilization. Our hypothesis was that higher N in the forest floor did not result in increased release of N from the forest floor and elevated mineral soil N availability due to studies suggesting N accumulations in the forest floor. Fertilization at

SETRES was on-going and ceased at Mt. Pleasant 3 years prior to sampling. This enabled us to test our hypothesis in a system where forest floor N was elevated by fertilization and 56 kg N ha^{-1} was added and in a system where forest floor N was elevated by previous fertilization but no N was added recently.

4.2. Materials and Methods

4.2.1. Site Descriptions

SETRES

The Southeast Tree Research and Education Site (SETRES) was established in 1985 in order to investigate the effects of irrigation and fertilization on loblolly pine growing on an infertile, excessively-drained sandy soil. The site was located in the Sandhills of Scotland County, NC 17 km north of Laurinburg, NC (34° 54′N, 79° 28′W). Mean annual precipitation was 120 cm and the mean minimum and maximum temperatures were 10.4 and 23.6 °C, respectively. Prior to planting of loblolly pine in 1985, the site was a native longleaf pine (*Pinus palustris* Mill.) forest. The stand was 23-years old when sampling began for this study.

The soil was mapped as the Wakulla series; an infertile, excessively drained, sandy, siliceous, thermic Psammentic Hapludult. The experimental design at SETRES was a 2^2 factorial randomized complete block with 4 blocks (n=4, N=16) with fertilization and irrigation treatments. In 1992, 50 x 50 m treatment plots containing interior 30 x 30 m measurement plots were established. Prior to initial treatment application, plots were thinned and competing vegetation was controlled. Treatments included fertilization (no addition and optimal foliar nutrition based on target foliar nutrient concentration) and irrigation (no addition and addition of 2.5 cm water $week^{-1}$

March to November). Treatment levels included a control (Ct), irrigation (I), fertilization (F), and fertilization x irrigation (FI). Fertilization was conducted by applying a balance of macro- and micro-nutrients to provide optimal foliar nutrition and irrigation was conducted to maintain a soil water content >40% field capacity. Fertilization began in March 1992 and irrigation began in April 1993 and continues to the present. Fertilization was conducted each spring with a broadcast surface application of various fertilizer sources including granular urea, boron-coated urea, ammonium sulfate, diammonium phosphate, triple super phosphate, potassium chloride, dolomitic lime, Epsom salts, calcium sulfate, and borax (Albaugh et al., 2008). Total N and P applications from 1992 to 2009 were 1434 kg ha^{-1} and 168 kg ha^{-1}, respectively (Table 4.1). Nutrient additions significantly increased tree growth with volume increased by 100% after 13 yrs. of fertilization (Albaugh et al., 1998; Albaugh et al., 2004).

Mt. Pleasant, GA

A similar study designed to evaluate the effects of irrigation and fertilization on a number of pine and hardwood species was established in Mt. Pleasant, GA on an infertile, excessively-drained sandy soil. The site was located in the southeastern Georgia coastal plain in Wayne Co. (31°23′N, 81°43′W). Mean annual precipitation was 127 cm and the mean minimum and maximum temperatures were 12.0 and 26.0 °C, respectively. Prior to establishment of the study in 1997, the site was a slash pine (*Pinus elliottii* Engelm.) plantation. After harvesting of the previous pine stand, the site was cleared, disked, limed, and planted with separate plots containing loblolly pine, slash pine, sweetgum, and sycamore (*Platanus occidentalis* L.). This paper includes data from only the loblolly pine and sweetgum plots. The stands were 12-years old when sampling began for this study. The soil was mapped as the Klej series; a mesic, coated Aquic Quartzipsamment.

The study was a randomized complete block design with 3 blocks (n=3, N=18). In 1997, 35 x 25.5 m treatment plots containing interior 22.2 x 13.5 m measurement plots were established. Treatment levels included a control (Ct), irrigation (I), and 3 levels of fertilization combined with irrigation (FI). Only the high fertilization rate was evaluated in this study where annual nutrient additions averaged 113 N, 17 P, and 62 K kg ha^{-1} yr^{-1} (Cobb et al., 2008). Fertilization and irrigation treatments began in 1997. Irrigation was conducted by applying 3.05 cm water $week^{-1}$ for 28 weeks $year^{-1}$ (April to November) from 1997 to 2003. Fertilization from 1997 to 2003 was conducted by applying nutrients in solution in the irrigation water throughout the 28 weeks of irrigation each year. Nitrogen was supplied as ammonium nitrate (NH_4NO_3). From 2004 to 2006, surface applications of N, P, and micronutrients were conducted in the spring. Nitrogen and P were applied as diammonium phosphate (DAP) and urea. Total N and P applications from 1997 to 2006 were 1128 kg ha^{-1} and 169 kg ha^{-1} (Table 4.2), respectively, which were comparable to total fertilizer N and P applied at SETRES. Nutrient additions increased aboveground biomass (Cobb et al., 2008). After 9 yrs. of fertilization, loblolly pine volume increased by 30% and sweetgum volume increased by 300%.

4.2.2. N Availability

Forest floor *in situ* N availability was assessed with cation and anion exchange membranes (IEM) (Ionics Inc., Watertown, MA, USA) placed at the forest floor/mineral soil interface in random locations within each plot. For both cation and anion IEM, two replicate membranes measuring 81 cm^2 were placed in each plot. Reported data were expanded to 1 m^2 of membrane. Membranes were removed after 30 days and a new set was placed in random locations within each plot. Soil adhering to the membranes was removed and membranes were transported on ice to the laboratory where they were stored at 4 °C until extraction. Membranes were extracted in 1M KCl for one hour on a reciprocal shaker. Extracts were collected in scintillation vials and frozen prior to chemical analysis.

Mineral soil *in situ* N mineralization and nitrification in the 0-15 cm mineral soil depth were assessed with the sequential core method (Raison, 1987; Gurlevik et al., 2004). Two replicate cores were collected and incubated at random locations within each plot at 30 day intervals. Soil samples were transported on ice to the laboratory where they were stored at 4 °C until extraction in 2M KCl with the method of Bremner (1965). Extracts were filtered through Whatman #42 into scintillation vials and frozen prior to chemical analysis. Moisture content of soil core samples was determined in order to report N concentration on a dry weight basis. Measurements of *in situ* N availability were carried out from June 2008 to November 2008 (SETRES cores) and May 2009 to November 2009 (SETRES membranes; Mt. Pleasant cores and membranes). At SETRES 56 kg N ha^{-1} were applied in April 2008 and 2009.

4.2.3. Chemical Analysis

Membrane and soil core extracts were analyzed for NH_4-N and NO_3-N concentration using a TRAACS 2000 analytical console (Bran & Luebbe, Norderstedt, Germany).

4.2.4. Statistical Analysis

Treatment and species effects on both mineral soil sequential core and IEM NH_4-N and NO_3-N data were determined with a repeated measures analysis using the PROC MIXED procedure (Littell et al., 1998) (SAS Institute Inc., Cary, NC, USA). At SETRES, irrigation was ceased in blocks 2 and 3 in 2009. This was anticipated to influence the outcome of IEM N measurements due to differences in transport of N in the irrigation water and adjustments were made to account for the lack of irrigation in these blocks. Therefore, n=2 for I and F x I treatments and n=6 for C and F treatments for the IEM data. Mineral soil sequential core N availability was measured in 2008 and n=4 for all treatments. At Mt. Pleasant, trees in the block 3 loblolly FI plot were killed by the Ips beetle (*Ips spp.*). Therefore, this plot was dropped from IEM and sequential core measurements and n=2 for loblolly FI while n=3 for all other species/treatment combinations.

4.3. Results

SETRES

Nitrogen at the forest floor/mineral soil interface measured with IEM was increased by fertilization (Tables 4.3 and 4.4). Fertilization increased NH_4-N in months 1-3 and month 5 (Fig. 4.1A) and cumulative NH_4-N (Fig. 4.2A). Cumulative NH_4-N was approximately 3.0 g m^{-2} IEM for fertilized treatments and 0.14 g m^{-2} IEM for unfertilized treatments (Fig. 4.2A). Fertilization increased NO_3-N (Table 4.1; Fig. 4.1B) and cumulative NO_3-N (Table 4.2) in months 4-7 (Fig. 4.2B). Cumulative NO_3-N was approximately 0.5 g m^{-2} IEM for fertilized treatments and 0.1 g m^{-2} IEM for unfertilized treatments (Fig. 4.2B).

Fertilization increased mineral soil extractable NH_4-N but not extractable NO_3-N (Table 4.3; Fig. 4.3). Fertilization increased mineral soil cumulative nitrification NO_3-N but not cumulative mineralization NH_4-N (Table 4.4; Fig. 4.4). Cumulative mineralization NH_4-N averaged 28.4 kg ha^{-1} across treatments (Fig. 4.4A). Fertilization increased cumulative nitrification NO_3-N from 2.2 kg ha^{-1} for unfertilized treatments to 19.2 kg ha^{-1} for fertilized treatments (Fig. 4.4B).

Mt. Pleasant

Nitrogen at the forest floor/mineral soil interface detected by IEM was less than at SETRES. NH_4-N was highest in fertilized loblolly pine and sweetgum in month 1 indicating N supply was elevated by forest floor decomposition in fertilized treatments (Table 4.3; Fig. 4.5A). NO_3-N was generally higher in sweetgum than in loblolly pine (Table 4.3; Fig. 4.5B). Cumulative NH_4-N was approximately 0.10 g m^{-2} IEM for loblolly FI and sweetgum I and FI and was 0.03 g m^{-2} IEM for loblolly I (Table 4.4; Fig. 4.6). Cumulative NO_3-N did not differ among treatments and species (Table 4.4; Fig. 4.6).

Mineral soil extractable NO_3-N varied by treatment and species but extractable NH_4-N did not (Table 4.3; Fig. 4.7). In months 2 and 7, extractable NO_3-N was higher in loblolly pine than in sweetgum (Fig. 4.7A). In months 4 and 7, extractable NO_3-N was higher in fertilized treatments of both species (Fig. 4.7A). Mineral soil cumulative mineralization NH_4-N did not vary by treatment or species and was characterized by immobilization throughout most of the growing season (Table 4.4; Fig. 4.8A). Mineral soil cumulative nitrification NO_3-N varied by treatment and species and decreased on the order of loblolly FI (10.7 kg ha^{-1}) > loblolly I (5.5 kg ha^{-1}) > sweetgum FI (2.2 kg ha^{-1}) >

sweetgum I (0.5 kg ha^{-1}) (Fig. 4.8B). Mineral soil extractable and cumulative NO_3-N was generally elevated in loblolly pine relative to sweetgum.

4.4. Discussion

Results suggested that after fertilizer application to the surface of the forest floor as conducted at SETRES, fertilizer is not immobilized in the forest floor, but rather, fertilizer N travels through to the forest floor/mineral soil interface. After an application of 56 kg N ha^{-1} in April, forest floor IEM NH_4-N immediately increased in fertilized treatments and remained elevated for 5 months suggesting the fertilizer had travelled through the forest floor in this time frame. This pattern of elevated N was consistent with results of Mudano (1986) following fertilization of loblolly pine. In the absence of a recent fertilizer application at Mt. Pleasant, membranes most likely only detected N released from forest floor decomposition. Although higher in fertilized plots, the quantity of N entering the forest floor/mineral soil interface from decomposition of the forest floor was much lower compared to SETRES.

The magnitude of the increase in mineral soil extractable N at SETRES from fertilization was not as large as one would anticipate given the increase in IEM N from fertilization. While mineral soil extractable N was not increased as one would expect, mineral soil cumulative nitrification was greatly increased suggesting fertilization results in elevated mineral soil N availability. Nannipieri et al. (1990), Ledgard et al. (1989), and Blankenau et al. (2000) suggested that fertilizer N that entered the mineral soil was first temporarily immobilized by the microbial biomass. Yevdokimov and Blagodatsky (1993) suggest that in the mineral soil, decreases in mineral N were accompanied by increases in microbial N. In addition, at least 70% of ^{15}N in plant was derived from re-mineralized microbial N. Based on these findings, it is possible the discrepancy among quantities of mineral soil extractable N and nitrification in fertilized treatments at SETRES arose from microbial immobilization. We hypothesize that fertilizer N entered the mineral soil and was immobilized by the microbial biomass leading to lower than expected extractable mineral-N. Upon incubation of the soil cores, this N immobilized in the microbial biomass was released and nitrification occurred. Raison et al. (1992) suggested this same process occurred and after labile C was depleted, N was released from the microbial biomass. This was also suggested by Chang (1996) in a ^{15}N tracer study. This led us to believe that fertilization results in elevated mineral soil N that may not be detectable by measuring extractable NH_4-N and NO_3-N. Instead, mineralization and nitrification must be measured since fertilizer N was first temporarily sequestered in the microbial biomass.

At Mt. Pleasant where fertilizer was last applied 3 years prior to sampling, decomposition of the forest floor increased release of N and elevated mineral soil N with this increase attributable only to NO_3-N. Therefore, the hypothesis that higher N in the forest floor did not result in increased release of N from the forest floor and elevated mineral soil N was refuted. Results suggest that increases in forest floor N from past fertilization resulted in elevated mineral soil N beyond the immediate effects of fertilizer application. However, the magnitude of elevated mineral soil N was greatly diminished relative to SETRES and while the differences in mineral soil NO_3-N were statistically significant, they were likely not biologically significant since foliar N concentration in the following year was no longer higher in fertilized plots (Chapter 3). The increase in mineral soil NO_3-N observed in fertilized treatments appeared proportionately large compared to the increase in IEM N from forest floor decomposition. Since IEM only detected mineral-N, it is possible that the forest floor in fertilized treatments also released greater quantities of dissolved organic N (DON) which resulted in the observed elevated mineral soil N in the fertilized treatments. According to Kalbitz et al. (2007), the quantity of forest floor dissolved organic matter (DOC) was greatest from the more highly decomposed Oe/Oa horizons. At Mt. Pleasant, Oe and Oa horizon mass was reported to be higher in the fertilized treatments suggesting DOC and DON export may have been higher as well (Chapter 2).

A species effect in the mineral soil indicated higher NO_3-N in loblolly pine compared to sweetgum even though forest floor IEM N was similar. This suggests that higher NO_3-N in loblolly pine may also have resulted from higher DON export from the more highly decomposed Oe and Oa horizons. Another explanation may be due to differences in labile C among loblolly pine and sweetgum litter affecting N cycling processes. Kiser (Chapter 3) found that soluble carbohydrate and phenols concentrations were higher in sweetgum than in loblolly pine. When labile C inputs were higher, microbes immobilized more NH_4-N reducing NO_3-N production (Davidson et al., 1992; Hart et al., 1994). According to Raison et al. (1992), available labile C results in microbial N immobilization and delays N mineralization until labile C is depleted. It is possible that higher labile C sources in sweetgum resulted in more N immobilization compared to loblolly pine and accounted for lower NO_3-N in the mineral soil of sweetgum plots.

Schimel and Bennett (2004) hypothesized that N cycling follows a gradient of N availability. At the highest level of N availability, net mineralization and nitrification become active. This likely occurred at SETRES where 56 kg N ha^{-1} were applied and was assumed to have occurred at Mt. Pleasant while fertilization was active. Results suggest that while fertilization was active, supply of N was adequate to meet the demands of both microbes and trees as indicated by high nitrification. Fertilization ceased 3 years prior to sampling at Mt. Pleasant and N cycling has likely shifted to processes hypothesized by Schimel and Bennett (2004) to occur at lower levels of N availability where N rich micro-sites mineralize and N poor micro-sites immobilize. When fertilization ended, supply of N was diminished and microbes and plants began to compete for available N as indicated by immobilization. However, supply of N was still elevated enough for nitrification to occur.

Growth responses to a single application of N have been reported to last for 6 to 10 years (Fox et al., 2007). At Mt. Pleasant, N availability was higher in fertilized plots 3 years after the last fertilizer application. Foliar N concentration was not higher in fertilized plots. We hypothesize that when fertilization ends, the decomposing forest floor in fertilized plots supplies greater quantities of N relative to unfertilized plots. This effect gradually diminishes over time until N supply and cycling processes become equivalent to unfertilized systems where NO_3-N pools are very small (Vitousek and Matson, 1985). At SETRES, a fertilizer application of 56 kg N ha^{-1} increased site N levels to the extent that production exceeded consumption resulting in a net release of N. At Mt. Pleasant, 3 years after fertilizer was last applied; site N levels were diminishing indicated by production not exceeding consumption.

4.5. Literature Cited

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4.6. Tables and Figures

Table 4.1. Nutrient additions at SETRES from 1992 to 2009. Granular fertilizer was applied to the surface on each application date.

Application Date	N	P	K	Ca	Mg	S	B
	kg ha^{-1}						
Mar-92	224	56	112	0	0	0	0
Apr-92	0	0	0	134	56	0	0
Jun-92	0	0	0	0	0	0	2
Mar-93	0	28	0	0	0	0	0
Apr-93	26	22	21	0	0	0	0
Jun-93	0	0	92	0	56	120	0
Aug-93	56	0	0	0	0	0	0
Mar-94	112	0	0	0	0	0	0
Mar-95	56	28	56	24	34	74	0
May-95	0	0	0	0	0	0	1
Mar-96	112	11	56	10	0	15	0
Apr-96	0	0	0	0	0	0	1
Apr-97	135	0	0	0	0	0	0
Mar-98	56	6	0	0	0	0	0
Mar-99	69	0	0	0	0	0	0
Mar-00	56	6	0	0	0	0	0
Apr-01	56	6	0	0	0	0	0
Apr-02	56	6	0	0	0	0	0
May-02	0	0	56	0	0	0	0
Apr-03	56	0	0	0	0	0	0
Mar-04	56	0	0	0	0	64	0
Mar-05	84	0	0	0	0	0	1
Mar-06	56	0	0	0	0	64	0
Apr-07	56	0	0	0	0	0	0
Apr-08	56	0	0	0	0	0	0
Apr-09	56	0	0	0	0	0	0
Total	1434	168	393	168	146	337	6

Table 4.2. Nutrient additions at Mt. Pleasant from 1997 to 2006. From 1997 to 2003, soluble fertilizer was applied over a 28 week period in the irrigation water. From 2004 to 2006, a single surface application of granular fertilizer was conducted in the spring.

Year	N	P	K	Cu	Mn	Mo	S	B	Fe
	kg ha^{-1}								
1997	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
1998	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
1999	113	17	62	0.0	0.0	0.0	0.0	0.0	0.0
2000	170	26	94	0.0	0.0	0.0	0.0	0.0	0.0
2001	51	8	28	0.0	0.0	0.0	0.0	0.0	0.0
2002	116	17	64	0.0	0.0	0.0	0.0	0.0	0.0
2003	116	17	64	0.0	0.0	0.0	0.0	0.0	0.0
2004	112	17	62	0.6	1.4	0.0	1.4	0.6	3.4
2005	112	17	62	0.7	1.6	0.5	0.0	0.7	3.8
2006	112	17	62	0.7	1.6	0.5	0.0	0.7	3.8
Total	1128	169	621	2.0	4.6	1.0	1.4	2.0	11.0

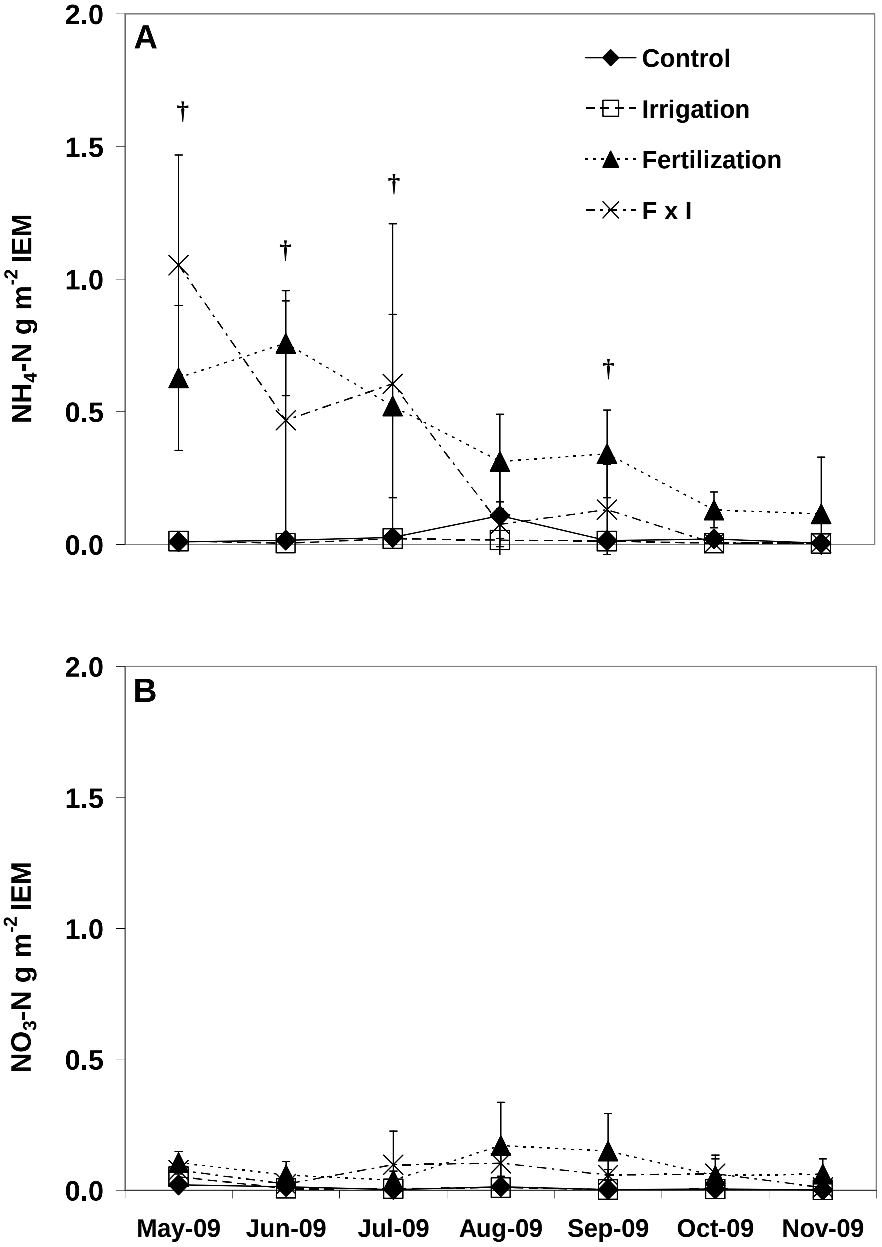

Fig. 4.1. Mean forest floor ion exchange membrane (IEM) NH_4-N (A) and NO_3-N (B) at SETRES in g m^{-2} IEM. Error bars represent 1 standard deviation. † Indicates months with a significant fertilization effect ($p < 0.05$).

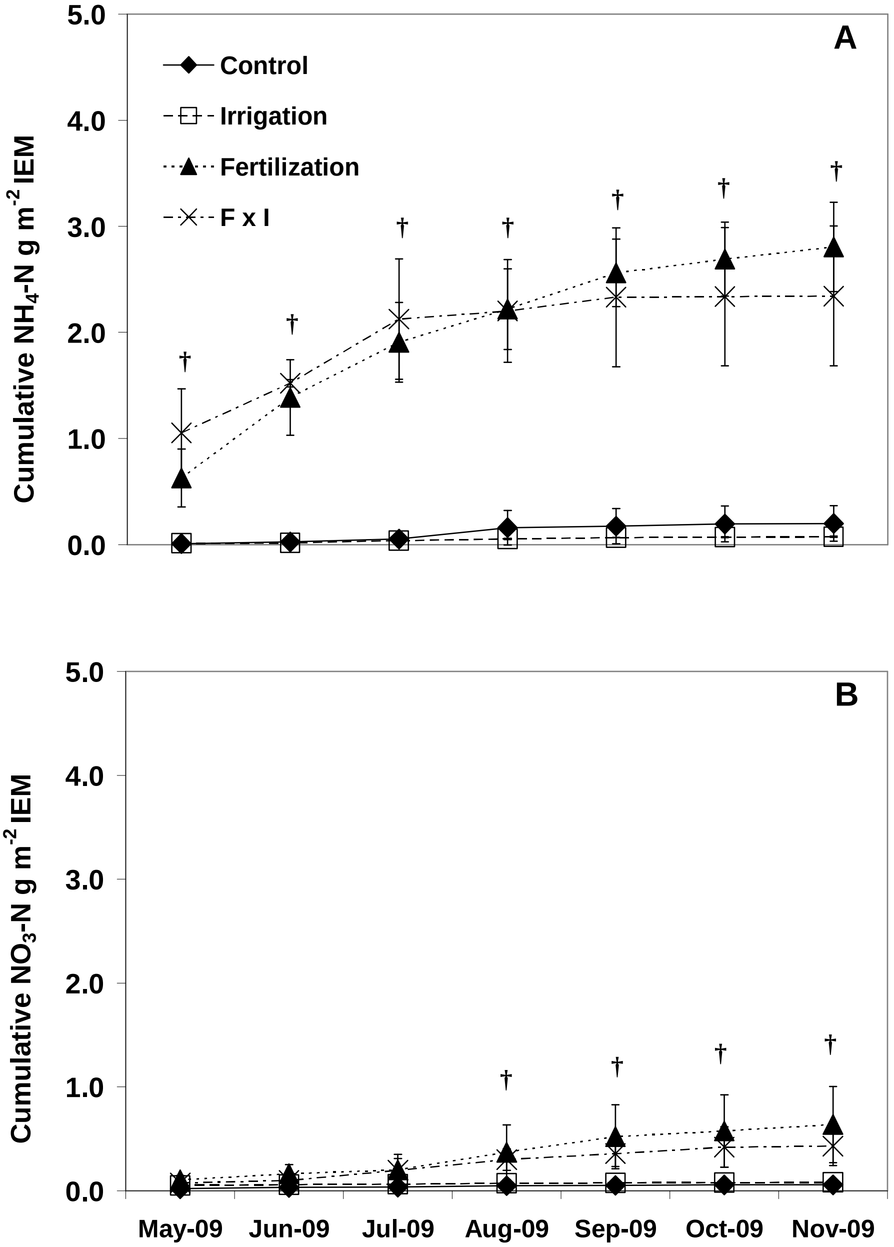

Fig. 4.2. Mean cumulative forest floor ion exchange membrane (IEM) NH_4-N (A) and NO_3-N (B) at SETRES in g m^{-2} IEM. Error bars represent 1 standard deviation.
† Indicates months with a significant fertilization effect ($p < 0.05$).

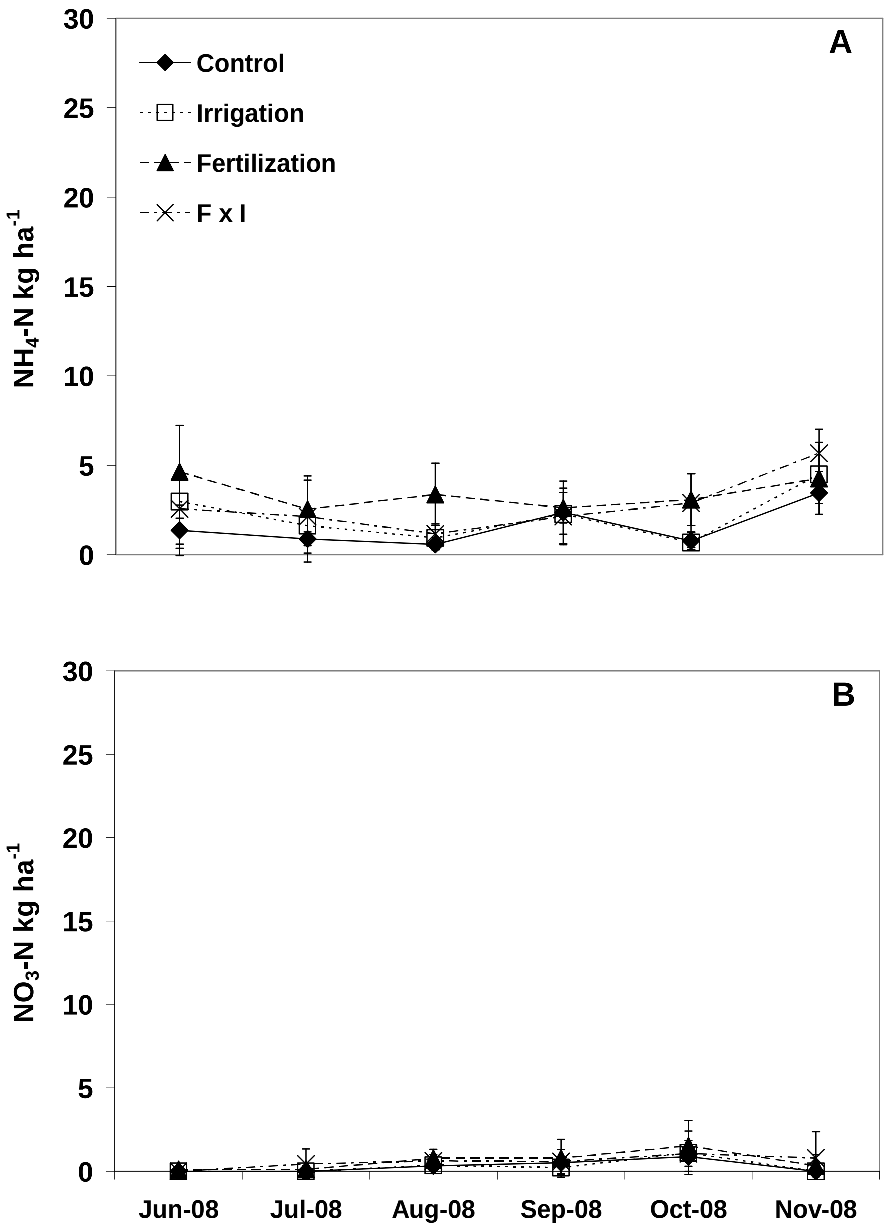

Fig. 4.3. Mean mineral soil extractable NH_4-N (A) and nitrification NO_3-N (B) at SETRES in kg ha^{-1}. Error bars represent 1 standard deviation.

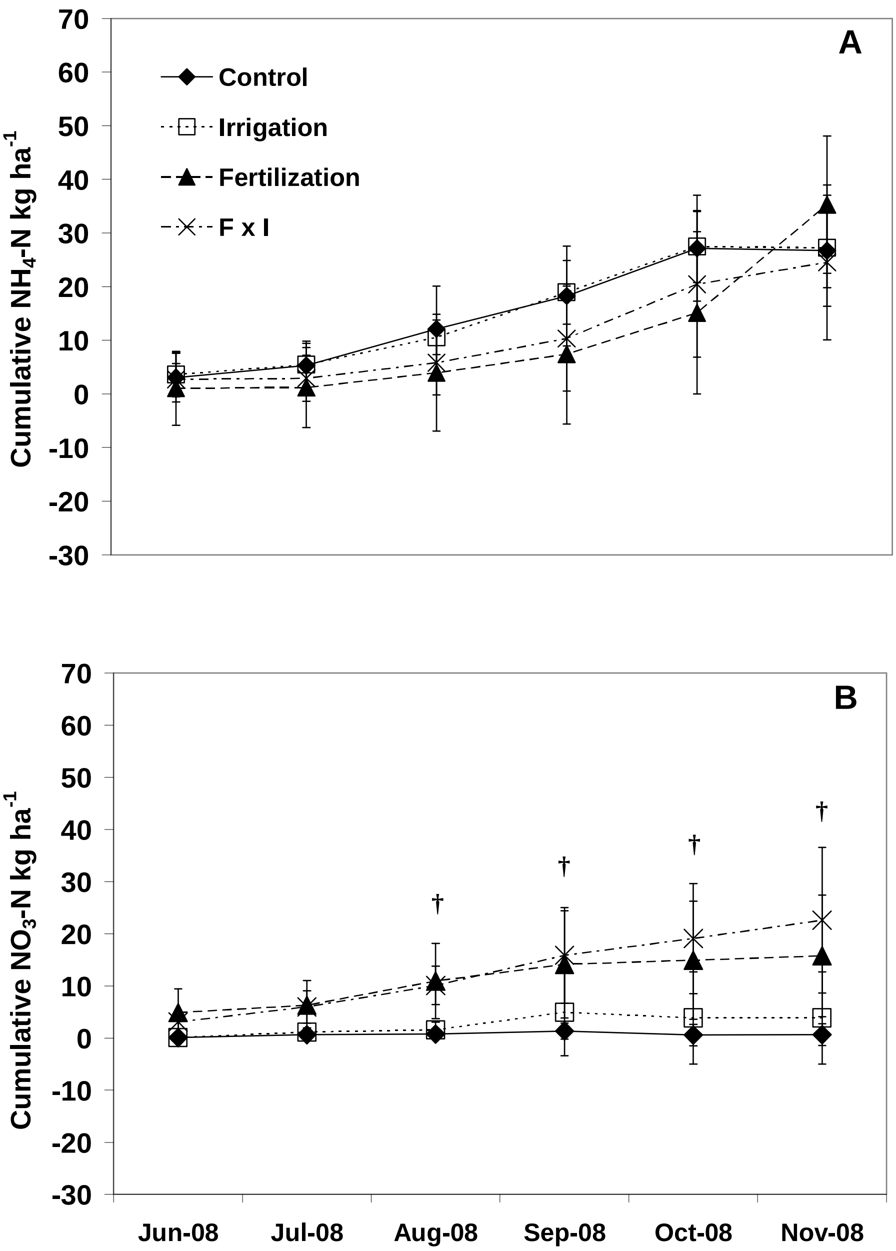

Fig. 4.4. Mean cumulative mineral soil mineralization NH_4-N (A) and nitrification NO_3-N (B) at SETRES in kg ha^{-1}. Error bars represent 1 standard deviation. † Indicates months with a significant fertilization effect ($p < 0.05$).

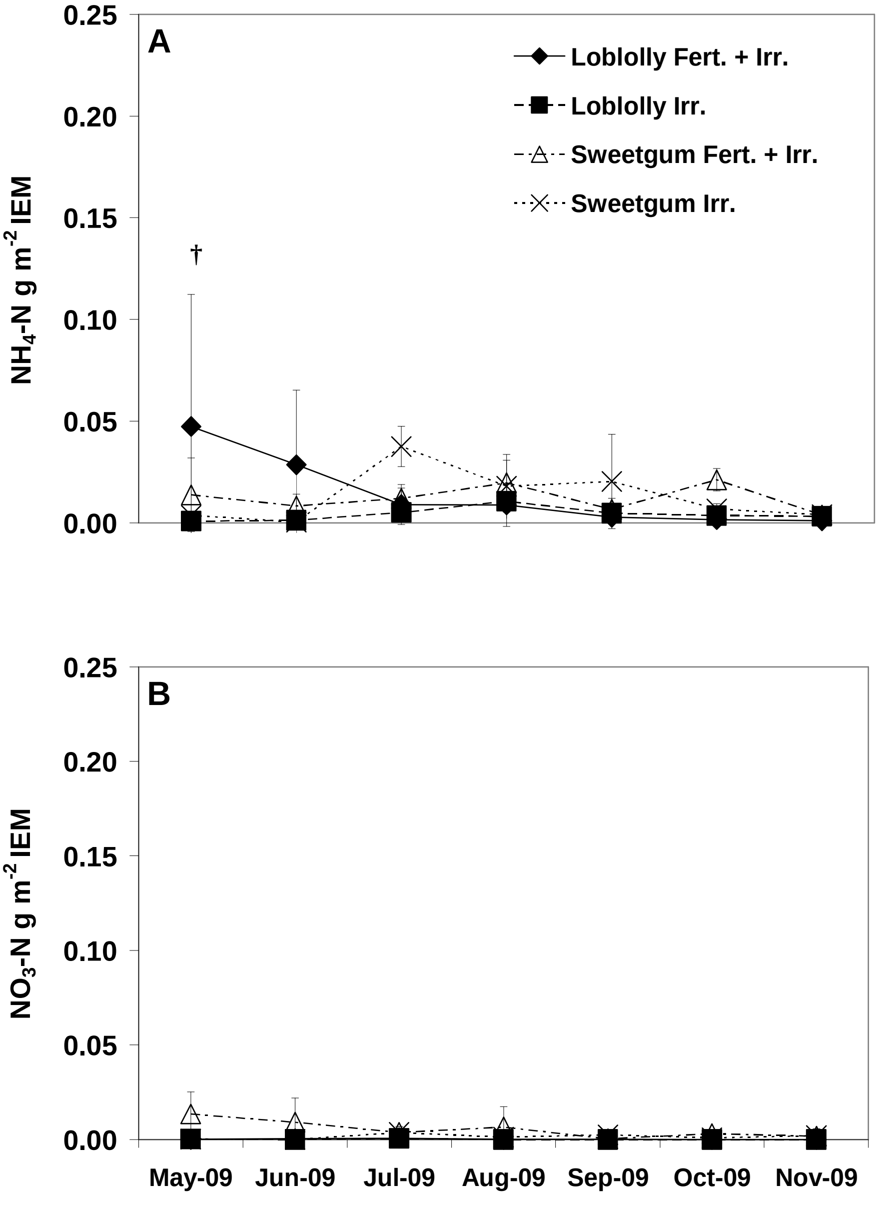

Fig. 4.5. Mean forest floor ion exchange membrane (IEM) NH_4-N (A) and NO_3-N (B) at Mt. Pleasant in g m^{-2} IEM. Error bars represent 1 standard deviation. † Indicates months with a significant treatment effect ($p < 0.05$).

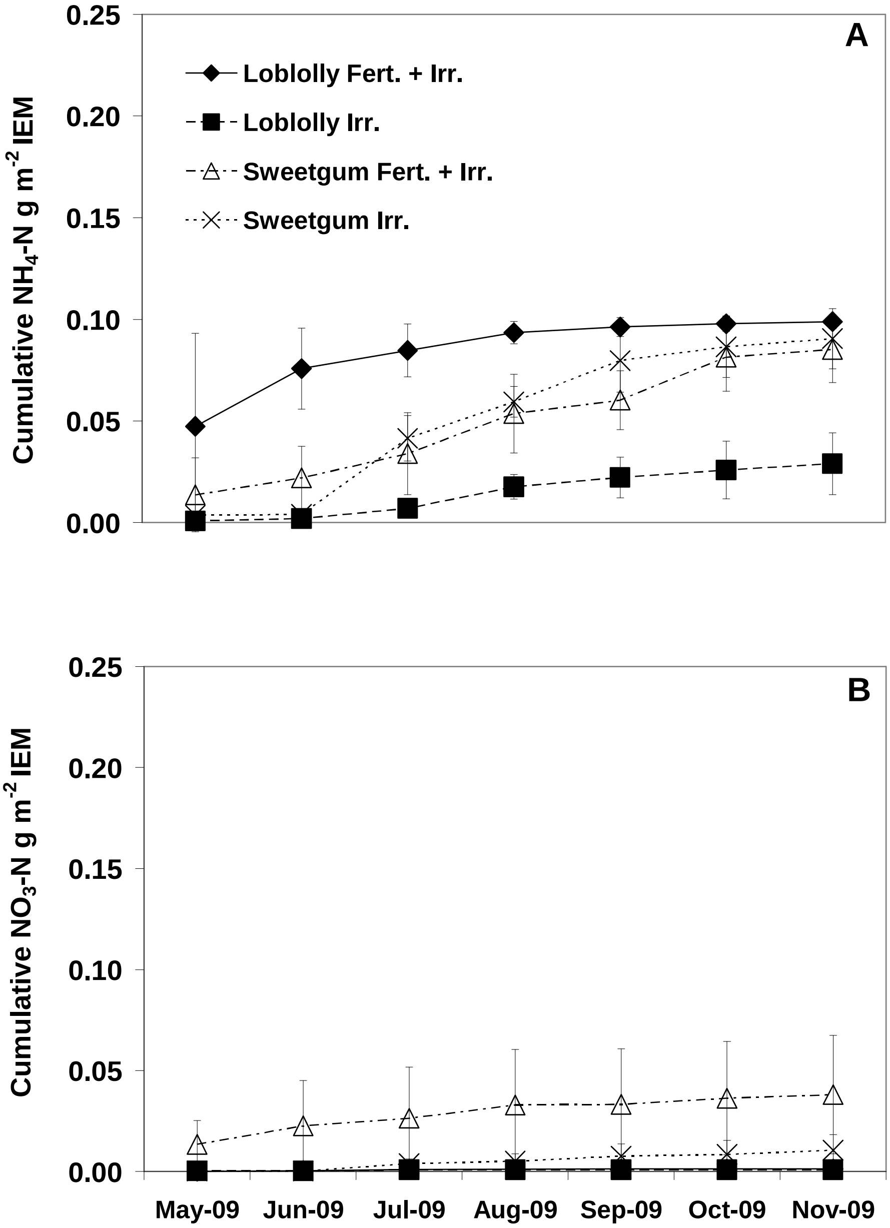

Fig. 4.6. Mean cumulative forest floor ion exchange membrane (IEM) NH_4-N (A) and NO_3-N (B) at Mt. Pleasant in g m^{-2} IEM. Error bars represent 1 standard deviation.

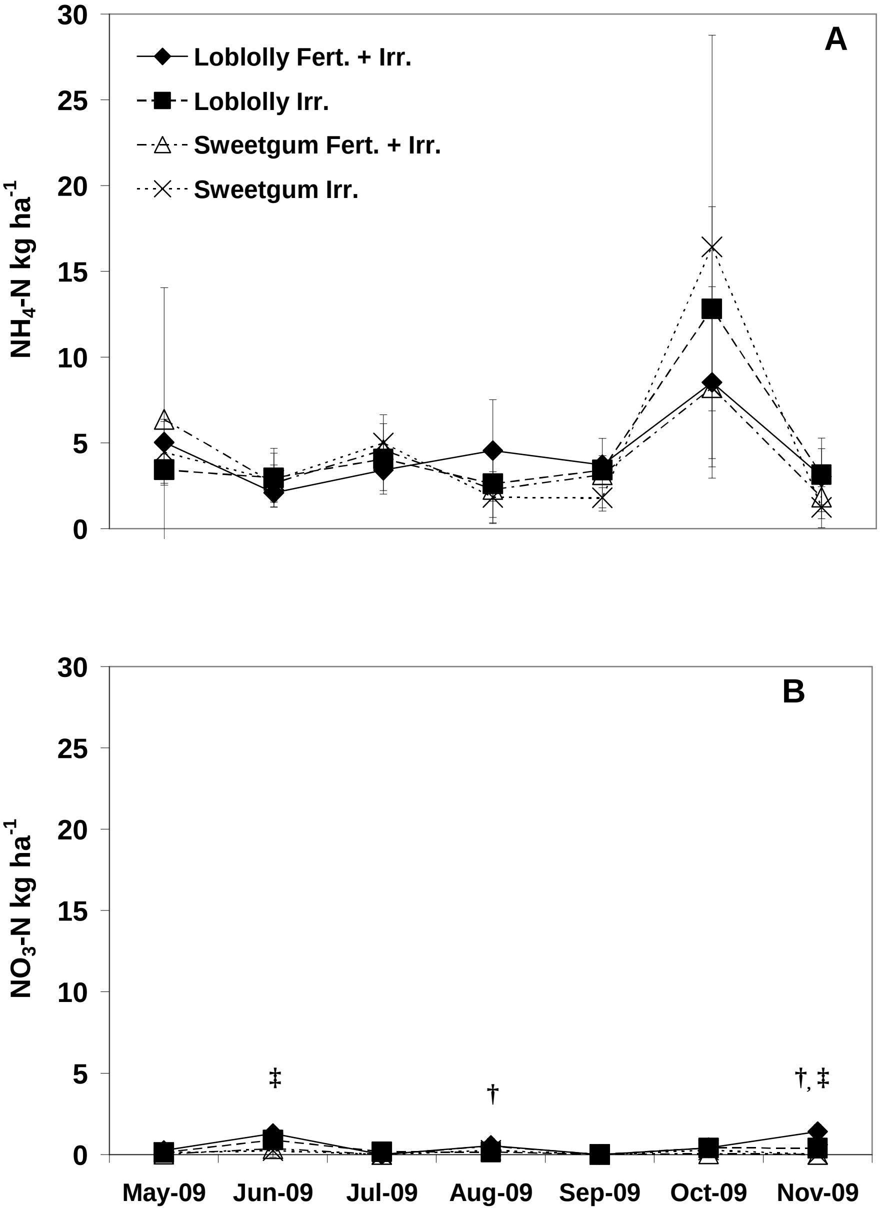

Fig. 4.7. Mean mineral soil extractable NH_4-N (A) and NO_3-N (B) at Mt. Pleasant in kg ha^{-1}. Error bars represent 1 standard deviation. † Indicates months with a significant treatment effect and ‡ indicates months with a significant species effect ($p < 0.05$).

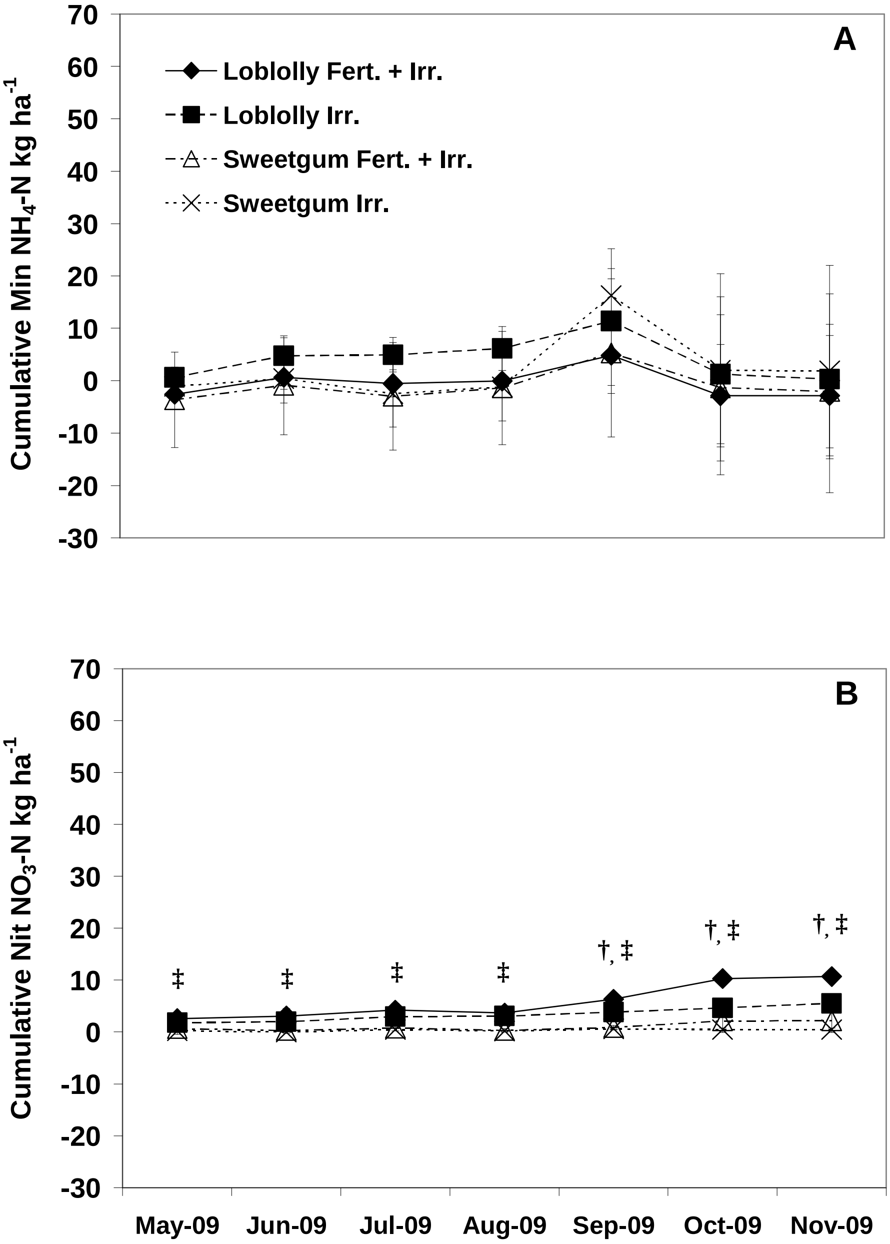

Fig. 4.8. Mean cumulative mineral soil mineralization NH_4-N (A) and nitrification NO_3-N (B) at Mt. Pleasant in kg ha^{-1}. Error bars represent 1 standard deviation. † Indicates months with a significant treatment effect and ‡ indicates months with a significant species effect ($p < 0.05$).

Table 4.3. Repeated measures main and interaction effect p-values for SETRES and Mt. Pleasant forest floor and mineral soil NH_4-N and NO_3-N. Mineral soil N represents extractable N.

Forest Floor				Mineral Soil			
SETRES		Mt. Pleasant		SETRES		Mt. Pleasant	
Effect	p-value	Effect	p-value	Effect	p-value	Effect	p-value
NH_4-N g m^{-2} IEM				NH_4-N kg ha^{-1}			
Fertilization	**<0.0001**	Treatment	**0.0385**	Fertilization	**0.0018**	Treatment	0.4893
Irrigation	0.4203	Species	0.1051	Irrigation	0.9208	Species	0.8609
Month	**<0.0001**	Month	0.0970	Month	**<0.0001**	Month	**<0.0001**
Fert. x Month	**<0.0001**	Trt. x Month	**0.0033**	Fert. x Month	0.4658	Trt. x Month	0.2471
Irr. x Month	0.1509	Species x Month	**0.0107**	Irr. x Month	0.5538	Species x Month	0.8958
Fert. x Irr.	0.5385	Trt. x Species	**0.0170**	Fert. x Irr.	0.0795	Trt. x Species	0.9961
NO_3-N g m^{-2} IEM				NO_3-N kg ha^{-1}			
Fertilization	**<0.0001**	Treatment	0.1162	Fertilization	0.1049	Treatment	**0.0411**
Irrigation	0.3521	Species	**0.0297**	Irrigation	0.9416	Species	**<0.0001**
Month	0.2710	Month	0.8173	Month	**0.0007**	Month	**<0.0001**
Fert. x Month	0.4969	Trt. x Month	0.3487	Fert. x Month	0.9588	Trt. x Month	**0.0328**
Irr. x Month	0.7890	Species x Month	0.8373	Irr. x Month	0.9292	Species x Month	**<0.0001**
Fert. x Irr.	0.2348	Trt. x Species	0.1873	Fert. x Irr.	0.9721	Trt. x Species	0.0517

Table 4.4. Repeated measures main and interaction effect p-values for SETRES and Mt. Pleasant forest floor and mineral soil cumulative NH_4-N and NO_3-N. Mineral soil cumulative N represents mineralization and nitrification from incubated soil cores.

Forest Floor				Mineral Soil			
SETRES		Mt. Pleasant		SETRES		Mt. Pleasant	
Effect	p-value	Effect	p-value	Effect	p-value	Effect	p-value
Cumulative NH_4-N g m^{-2} IEM				Cumulative NH_4-N kg ha^{-1}			
Fertilization	**<0.0001**	Treatment	**0.0159**	Fertilization	0.0749	Treatment	0.7342
Irrigation	0.5677	Species	0.9064	Irrigation	0.9048	Species	0.7076
Month	**<0.0001**	Month	**<0.0001**	Month	**<0.0001**	Month	**<0.0001**
Fert. x Month	**<0.0001**	Trt. x Month	0.1293	Fert. x Month	**0.0044**	Trt. x Month	0.5732
Irr. x Month	0.3718	Species x Month	**0.0164**	Irr. x Month	0.2068	Species x Month	0.5405
Fert. x Irr.	0.8083	Trt. x Species	**0.0125**	Fert. x Irr.	0.9340	Trt. x Species	0.9129
Cumulative NO_3-N g m^{-2} IEM				Cumulative NO_3-N kg ha^{-1}			
Fertilization	**0.0091**	Treatment	0.1255	Fertilization	**0.0027**	Treatment	**<0.0001**
Irrigation	0.7957	Species	0.0655	Irrigation	0.5098	Species	**<0.0001**
Month	**<0.0001**	Month	0.1589	Month	**<0.0001**	Month	**<0.0001**
Fert. x Month	**<0.0001**	Trt. x Month	0.6289	Fert. x Month	**0.0031**	Trt. x Month	**0.0005**
Irr. x Month	0.2656	Species x Month	0.2802	Irr. x Month	0.3665	Species x Month	**<0.0001**
Fert. x Irr.	0.7620	Trt. x Species	0.1560	Fert. x Irr.	0.9571	Trt. x Species	**0.0011**

Chapter 5
Loblolly pine (*Pinus taeda* L.) forest floor decomposition and nutrient release following a simulated disturbance

Abstract

Fertilization of loblolly pine (*Pinus taeda* L.) is conducted to increase growth on sites with low soil fertility. As a stand develops, the forest floor acts as a sink with mass and N and P contents increasing through time. This forest floor sink for nutrients is magnified when stands are fertilized. When a stand is harvested changes in environmental conditions can increase forest floor decomposition and release a flush of nutrients, the Assart effect. A litterbag experiment was conducted to examine forest floor decomposition in a simulated disturbance. The experiment was conducted at SETRES, a 24-year old loblolly pine 2^2 factorial irrigation and fertilization study located in the Sandhills region of NC. Treatment levels include a control, irrigation, fertilization, and fertilization x irrigation. Annual fertilization has greatly increased forest floor Oi and Oe horizon mass and nutrient contents. Forest floor Oi + Oe horizon samples were collected from each treatment, placed in mesh bags and then placed in an adjacent, open area without an overstory canopy to simulate environmental conditions likely to occur following thinning or harvest. Our hypothesis was that increased endogenous N and P from fertilization would not increase the rate of decomposition and nutrient release from the loblolly pine forest floor. Mass loss and nutrient proportions released did not vary by treatment and forest floor Oi and Oe horizons lost 80% mass within 1 year. Nutrient content released was greatest in fertilized treatments. Release of C, N, P, K, and Ca after 1 year as the mean of fertilized treatments was 13.0 Mg ha^{-1}, 255 kg ha^{-1}, 13.0 kg ha^{-1}, 7.7 kg ha^{-1}, and 63 kg ha^{-1} respectively. Following thinning, increased forest floor

decomposition and nutrient availability is likely to have beneficial effects on tree growth. Following harvest of stands growing on sandy mineral soils, increased forest floor decomposition can result in loss of a significant amount of site N capital due to the inability of the soil to retain mineral-N. Results suggest management practices that minimize loss of this significant forest floor N pool are necessary.

5.1. Introduction

Loblolly pine (*Pinus taeda* L.) plantations grown on nutrient deficient soils in the southeastern U.S. require nitrogen (N) and phosphorus (P) fertilization to increase growth (Albaugh et al., 2007; Fox et al., 2007). Potassium (K) and calcium (Ca) are also often added to meet optimal foliar nutrition requirements. Fertilization increases growth by increasing foliar nutrients and leaf area (Albaugh et al., 1998) which also results in higher litterfall quantities. Nutrients sequestered in foliage accumulate in the forest floor as the stand ages and forest floor mass increases (Switzer and Nelson, 1972). Nutrient content also increases during the life of the stand (Switzer and Nelson, 1972). In fertilized systems, more nutrients accumulate in the forest floor due to higher inputs from litterfall and increased nutrient concentrations.

Studies that examined loblolly pine Oi horizon decomposition and nutrient release with *in situ* incubation of litterbags under the existing overstory canopy found an accumulation of N and P during decomposition (Piatek and Allen, 2001; Sanchez, 2001). However, Sanchez (2001) found this litter released K and Ca. Polglase et al. (1992a) determined soluble and insoluble N and P fractions in loblolly pine litter and concluded that litter was a net source of P, in conflict with other studies (Piatek and Allen, 2001; Sanchez, 2001). Decomposition rates of loblolly pine (Sanchez, 2001) and lodgepole pine

(*Pinus contorta* var. *latifolia* Engelm.) (Prescott, 1995) litter was not increased by higher endogenous N availability. Rather, decomposition was increased by higher labile carbon (C) availability (Osono et al., 2003; Kalbitz et al., 2007; Klotzbucher et al., 2011). Fertilization did not increase soluble carbohydrates in loblolly pine litter (Kiser, Chapter 3).

Nutrient accumulations in loblolly pine forest floors greater than 500 kg N ha^{-1} and 25 kg P ha^{-1} have been reported (Will et al., 2006; Chapter 2). While Sanchez (2001) suggests release of K and Ca occurred under the canopy, an accumulation of N and P reported by Piatek and Allen (2001) and Sanchez (2001) suggests the forest floor will not release or only slowly releases these important nutrients under the overstory canopy. This was supported in terms of N by Kiser (Chapter 4) who suggested release of N from forest floor decomposition proceeded slowly under the overstory canopy. In contrast, this release of P from the forest floor was suggested to proceed readily compared to N (Polglase, 1992a; Polglase, 1992b; Chapter 3). Quick release of N has been suggested to require disturbance of the overstory canopy such that occurs following thinning or harvest. When a stand is harvested, forest floor decomposition and nutrient mineralization is increased due to changes in environmental conditions resulting in a large flush of nutrients, the Assart effect (Kimmins, 1997).

Nitrogen availability is increased following a harvest disturbance through increased decomposition and mineralization of the forest floor and slash (Binkley, 1984; Vitousek and Matson, 1985; Fox et al., 1986). Evaluating the rate and magnitude of the release of nutrients from the forest floor following disturbance is important since the forest floor pool contains a substantial amount of N added through fertilization (Will et al., 2006; Chapter 2). Increased forest floor decomposition and nutrient release following a thinning disturbance has beneficial effects due to high tree demand. Thus, retention of nutrients is likely. In contrast, after harvest, rapid release of these accumulated nutrients early in the next rotation likely exceeds seedling nutrient demand (Fox et al., 2007). These conditions result in a potential loss of a significant amount of site N capital through N leaching.

Our objective was to determine loblolly pine forest floor decomposition and release of nutrients in a simulated disturbance environment. Our hypothesis was that increased endogenous N and P from fertilization would not increase the rate of decomposition and nutrient release from the loblolly pine forest floor.

5.2. Materials and Methods

5.2.1. Site Description

The Southeast Tree Research and Education Site (SETRES) was established in 1985 in order to investigate the effects of irrigation and fertilization on loblolly pine growing on an infertile, excessively-drained sandy soil. The site was located in the Sandhills of Scotland County, NC 17 km north of Laurinburg, NC (34° 54′N, 79° 28′W). Mean annual precipitation was 120 cm and the mean minimum and maximum temperatures were 10.4 and 23.6 °C, respectively. Prior to planting of loblolly pine in 1985, the site was a native longleaf pine (*Pinus palustris* Mill.) forest. The stand was 23-years old when sampling began for this study.

The soil was mapped as the Wakulla series; an infertile, excessively drained, sandy, siliceous, thermic Psammentic Hapludult. The experimental design at SETRES was a 2^2 factorial randomized complete block with 4 blocks (n=4, N=16) with fertilization and irrigation treatments. In 1992, 50 x 50 m treatment plots containing interior 30 x 30 m measurement plots were established. Prior to initial treatment application, plots were thinned and competing vegetation was controlled. Treatments included fertilization (no addition and optimal foliar nutrition based on target foliar nutrient concentration) and irrigation (no addition and addition of 2.5 cm water week^{-1} March to November). Treatment levels included a control (Ct), irrigation (I), fertilization (F), and fertilization x irrigation (FI). Fertilization was conducted by applying a balance of macro- and micro-nutrients to provide optimal foliar nutrition and irrigation was conducted to maintain a soil water content >40% field capacity. Fertilization began in March 1992 and irrigation began in April 1993 and continues to the present. Fertilization was conducted each spring with a broadcast surface application of various fertilizer sources including granular urea, boron-coated urea, ammonium sulfate, diammonium phosphate, triple super phosphate, potassium chloride, dolomitic lime, Epsom salts, calcium sulfate, and borax (Albaugh et al., 2008). Total N and P applications from 1992 to 2009 were 1434 kg ha^{-1} and 168 kg ha^{-1}, respectively (Table 5.1). Nutrient additions significantly increased tree growth with volume increased by 100% after 13 yrs. of fertilization (Albaugh et al., 1998; Albaugh et al., 2004).

5.2.2. Simulated Disturbance Environment Experiment

Litterbags (20 x 40 cm) were constructed of white nylon mesh with a 2 mm opening. Forest floor Oi and Oe horizons were collected in each plot in early May 2009. Beginning in late May 2009, litterbags containing approximately 100 g of forest floor material were placed on the mineral soil in an open area with no overstory canopy adjacent to the experimental plots replicating the layout of the plots. Four replicates were prepared for each of the 16 plots resulting in 64 total litterbags. One litterbag representing each of the 16 plots was collected and destructively sampled every 3 months. Samples were not bulked by treatment. Remaining mass and N and P concentrations were then determined. Samples were oven-dried at 60 ^{0}C for 5 days prior to mass determination and then ground using a Thomas-Wiley Model 4 mill (Thomas Scientific, USA) prior to chemical analysis. The final litterbags were sampled in May 2010.

5.2.3. Chemical Analysis

Total-N and -C were determined by dry combustion with a Vario MAX CNS analyzer (Elementar, Hanau, Germany). Total-P, K, and Ca were determined by dry-ashing at 500 °C and dissolution in 6N HCl. Total-P, K, and Ca in solution were analyzed with inductively-coupled plasma atomic emission spectrophotometry (ICP-AES) on a Varian Vista MPX **(**Varian, Palo Alto, CA, USA).

5.2.4. Statistical Analysis

Proportion of nutrients released was calculated following the method of Schlesinger and Hasey (1981). Nutrient content released was estimated by multiplying the proportion released from each litterbag with mass of the Oi + Oe horizons for each block/treatment combination. The decay rate constant (k month^{-1}) and mean residence time (MRT) for 99% mass loss were calculated (Olson, 1963). Treatment effects on mass remaining, the decay rate, and nutrient proportion and content released were determined with repeated measures ANOVA using the PROC MIXED procedure (Littell et al., 1998) (SAS Institute Inc., Cary, NC, USA).

5.3. Results

No treatment effects were observed for percent mass remaining and the decay rate constant (Table 5.2). The overall decay rate constant was determined for all the data (k = 0.411; R^2=0.95) and indicated a MRT of 12.2 months. Litterbags lost approximately 50% mass within 6 months and 80% within 1 year (Fig. 5.1). The proportion of C, N, P, and Ca released did not vary by treatment (Table 5.2). An irrigation effect was found for the proportion of K released and indicated a higher release in the control treatment for the first sampling interval. Treatment effects were observed for nutrient contents released (Table 5.2). At the end of the 1 year decomposition period, C content released from unfertilized and fertilized treatments was 7.1 and 13.0 Mg ha^{-1}, respectively (Fig. 5.2B). Nitrogen content released from unfertilized and fertilized treatments was 92 and 255 kg ha^{-1}, respectively (Fig. 5.3B). Phosphorus content released from unfertilized and fertilized treatments was 4.5 and 13.0 kg ha^{-1}, respectively (Fig. 5.4B). Potassium content released from unfertilized and fertilized treatments was 3.7 and 7.7 kg ha^{-1}, respectively (Fig. 5.5B). Calcium content released from unfertilized and fertilized treatments was 41.9 and 62.9 kg ha^{-1}, respectively (Fig. 5.6B).

5.4. Discussion

The decomposition rate of the forest floor was not increased by higher endogenous N availability from fertilization supporting our hypothesis and consistent with results of Prescott (1995) and Sanchez (2001). Decomposition was not different among treatments possibly due to no differences in soluble carbohydrates (Chapter 3). The combination of results from this study and those from Kiser (Chapter 3) support Osono et al. (2003), Kalbitz et al. (2007), and Klotzbucher et al. (2011) who proposed that decomposition is increased by increased availability of labile C. However, decomposition rates were greatly increased over rates reported by Piatek and Allen (2001) and Sanchez (2001). They reported similar decay rate constants that indicated a MRT for 99% mass loss of approximately 150 months whereas incubation of litter under environmental conditions likely to occur following disturbance in this study indicated a MRT of 12.2 months. Piatek and Allen (2001) and Sanchez (2001) incubated Oi horizon litterbags under the overstory canopy while in this study, Oi + Oe horizon litterbags were incubated in an open area with no overstory canopy. Release of dissolved organic matter (DOC) and nitrogen (DOC) was greater from more highly decomposed Oe/Oa horizons compared to the Oi horizon (Kalbitz et al., 2007) offering one explanation for the higher release of nutrients. However, different environmental conditions under the overstory canopy and open-sky were likely the main factors driving the large differences in decomposition rates. Comparison of under-canopy and on-site weather station data from 1998 revealed that annual mean temperature and relative humidity differed by only 5% among the open area and under the overstory canopy. However, annual mean photosynthetically active radiation and net radiation at the weather station were 17% and 58% higher than under-canopy. This comparison of environmental data suggests that at SETRES, higher light levels rather than differences in temperature and moisture accelerated decomposition. Studies that examined decomposition of various plant materials without an overstory canopy concluded that photodegradation from ultraviolet (UV) radiation increased decomposition (Austin and Vivanco, 2006; Brandt et al., 2010). While Brandt et al. (2009) suggest abiotic photochemical mineralization was a primary mechanism by which C was lost from decomposing oak and grass litter, the breakdown of organic matter molecules into smaller constituent pieces resulting in an acceleration of microbial decomposition may also be a key process according to Brandt et al. (2010). Brandt et al. (2010) conducted an experiment comparing decomposition of grass litter among litterbags protected from sunlight and exposed to sunlight. Exposing grass litter to sunlight was reported to increase decomposition and loss of the hemicellulose fraction. Extracellular enzyme activity was not higher in sunlight exposed litterbags leading Brandt et al. (2010) to propose increased decomposition was independent of photodegradation effects on microbial activity. It was alternatively postulated that equal extracellular enzyme activity among protected and exposed litterbags did not indicate a difference in microbial activity but instead indicated less energy was required to decompose the photodegraded substrate. While these studies did not evaluate loblolly pine litter and dealt with semi-arid ecosystems, these same process may have occurred in our experiment and resulted in the greatly accelerated decomposition observed in an open area without an overstory canopy.

Under the overstory canopy, decomposing loblolly pine Oi horizon litterbags were found to accumulate N and P and release K and Ca (Piatek and Allen, 2001; Sanchez, 2001). Decomposition of loblolly pine Oi + Oe horizon litterbags in this study also detected a release of K and Ca that was greatly accelerated. We also detected a rapid release of N and P with approximately 80% of the N and P released within one year. Results suggest that a disturbance that removes the overstory canopy will greatly accelerate forest floor decomposition and nutrient release.

A quick release of nutrients from the decomposing forest floor is expected to occur following removal of the overstory canopy at harvest. However, other studies suggest our results may be specific to the southeastern U.S. In contrast to our results from a loblolly pine plantation in NC, a release of nutrients was not observed in a mixed boreal forest for 3-4 years following clearcutting (Jerabkova et al., 2006). In another boreal forest clearcut study, Pacific silver fir (*Abies amabilis* [Dougl.] Forbes), western hemlock (*Tsuga heterophylla* [Raf.] Sarg.), and western red cedar (*Thuja plicata* Donn.) forest floor mass and N content did not differ from the uncut site until 6 years after harvesting (Martin et al., 2002). In another similar study, Western hemlock forest floor lost approximately 5% mass 1 year after harvest and fresh litter lost approximately 25% mass (Titus et al., 2006). After 2 years, canopy removal in a study of red pine (*Pinus resinosa* Sol. ex Aiton) litter decomposition was also found to not alter mass loss and nutrient release (Kim et al. 1996).

Quick release of nutrients from the decomposing forest floor may also occur following canopy removal from thinning (Prescott, 2002). However, whether this occurs is dependent upon the size of the canopy gap created by thinning. In a review of studies examining the influence of the forest canopy on nutrient cycling, Prescott (2002) found that single tree thinning does not increase N availability while canopy gaps from 0.07 ha to 0.25 ha and a removal of 15 trees did increase N availability. This suggests the potential for a mid-rotation thinning to result in increased nutrient availability at a time when tree demand is high. Further studies are warranted that investigate the influence of different thinning regimes on increases in nutrient availability from forest floor decomposition and the consequent influences on tree growth.

Nitrogen and P are the most growth limiting nutrients in southeastern U.S. loblolly pine plantations and represent the highest fertilizer cost. Efficient management of site N and P capital is therefore, essential. However, studies suggest that management of site P capital is much less of an issue relative to N. In acidic soils across of range of textures, fertilization resulted in an accumulation of P in the mineral soil suggesting increased site P availability and a long-term improvement in tree growth from higher P nutrition (Ballard, 1978; Gentle et al., 1986; Comerford et al., 2002; Crous et al., 2007; Everett and Palm-Leis, 2009, Chapter 2). However, on sandy mineral soils, fertilization with N results in higher increases in forest floor N compared to mineral soil N that were sustained for a longer period of time (Chapter 2). Kiser (Chapter 2) attributed the lack of a sustained increase in mineral soil N to the inability of sandy mineral soils to retain NH_4-N and NO_3-N due to low NH_4^+ fixation capacity and excessive drainage. Our results suggest that the forest floor N pool that has accumulated over the life of the stand will be quickly released following harvest. On acidic, sandy mineral soils, forest floor decomposition and N mineralization can potentially result in a loss of a substantial amount of site N capital. Nitrification and losses of N through nitrate leaching can be prevalent following harvest (Vitousek and Matson, 1984). The 255 kg N ha^{-1} released within 1 year of harvest represents a substantial amount of N particularly for nutrient poor sites creating the need for management strategies to minimize loss of this N. Site preparation techniques prior to planting of the next rotation determine what happens to the N pool that has accumulated over the life of the previous rotation. Fox et al. (1986) found that slash retention and incorporating the forest floor into the mineral soil by disking led to retention of N following harvest by inhibiting decomposition.

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5.6. Tables and Figures

Table 5.1. Nutrient additions at SETRES from 1992 to 2009. Granular fertilizer was applied to the surface on each application date.

Application Date	N	P	K	Ca	Mg	S	B
	kg ha^{-1}						
Mar-92	224	56	112	0	0	0	0
Apr-92	0	0	0	134	56	0	0
Jun-92	0	0	0	0	0	0	2
Mar-93	0	28	0	0	0	0	0
Apr-93	26	22	21	0	0	0	0
Jun-93	0	0	92	0	56	120	0
Aug-93	56	0	0	0	0	0	0
Mar-94	112	0	0	0	0	0	0
Mar-95	56	28	56	24	34	74	0
May-95	0	0	0	0	0	0	1
Mar-96	112	11	56	10	0	15	0
Apr-96	0	0	0	0	0	0	1
Apr-97	135	0	0	0	0	0	0
Mar-98	56	6	0	0	0	0	0
Mar-99	69	0	0	0	0	0	0
Mar-00	56	6	0	0	0	0	0
Apr-01	56	6	0	0	0	0	0
Apr-02	56	6	0	0	0	0	0
May-02	0	0	56	0	0	0	0
Apr-03	56	0	0	0	0	0	0
Mar-04	56	0	0	0	0	64	0
Mar-05	84	0	0	0	0	0	1
Mar-06	56	0	0	0	0	64	0
Apr-07	56	0	0	0	0	0	0
Apr-08	56	0	0	0	0	0	0
Apr-09	56	0	0	0	0	0	0
Total	1434	168	393	168	146	337	6

Table 5.2. Main and interaction effect p-values for repeated measures analysis of mass remaining, decay rate constant, and C, N, P, K, and Ca proportion and content released.

Effect	Mass Remaining (%)	Decay Rate k (month^{-1})	C Proportion Released	C Mass Released (Mg ha^{-1})
Fertilization	0.8727	0.7182	0.6746	**<0.0001**
Irrigation	0.7114	0.3359	0.3151	**0.0112**
Month	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**
Fert. x Month	0.1273	0.1990	0.1926	**<0.0001**
Irr. x Month	0.2028	**0.0272**	0.0657	0.2962
Fert. x Irr.	0.8758	0.6275	0.6501	0.4601
	N Proportion Released	N Mass Released (Mg ha^{-1})	P Proportion Released	P Mass Released (Mg ha^{-1})
Fertilization	0.5638	**<0.0001**	0.5091	**<0.0001**
Irrigation	0.9584	0.9190	0.5131	0.5498
Month	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**
Fert. x Month	0.2053	**<0.0001**	0.1486	**<0.0001**
Irr. x Month	0.2639	0.8743	0.2589	0.4835
Fert. x Irr.	0.1204	0.2512	0.2971	0.1256
	K Proportion Released	K Mass Released (Mg ha^{-1})	Ca Proportion Released	Ca Mass Released (Mg ha^{-1})
Fertilization	0.3416	**<0.0001**	0.9045	**<0.0001**
Irrigation	**0.0423**	**<0.0001**	0.5163	0.0662
Month	**<0.0001**	**<0.0001**	**<0.0001**	**<0.0001**
Fert. x Month	0.0551	**<0.0001**	0.4622	**<0.0001**
Irr. x Month	**0.0367**	0.2822	0.4067	0.6085
Fert. x Irr.	0.1345	0.5833	0.3503	0.3678

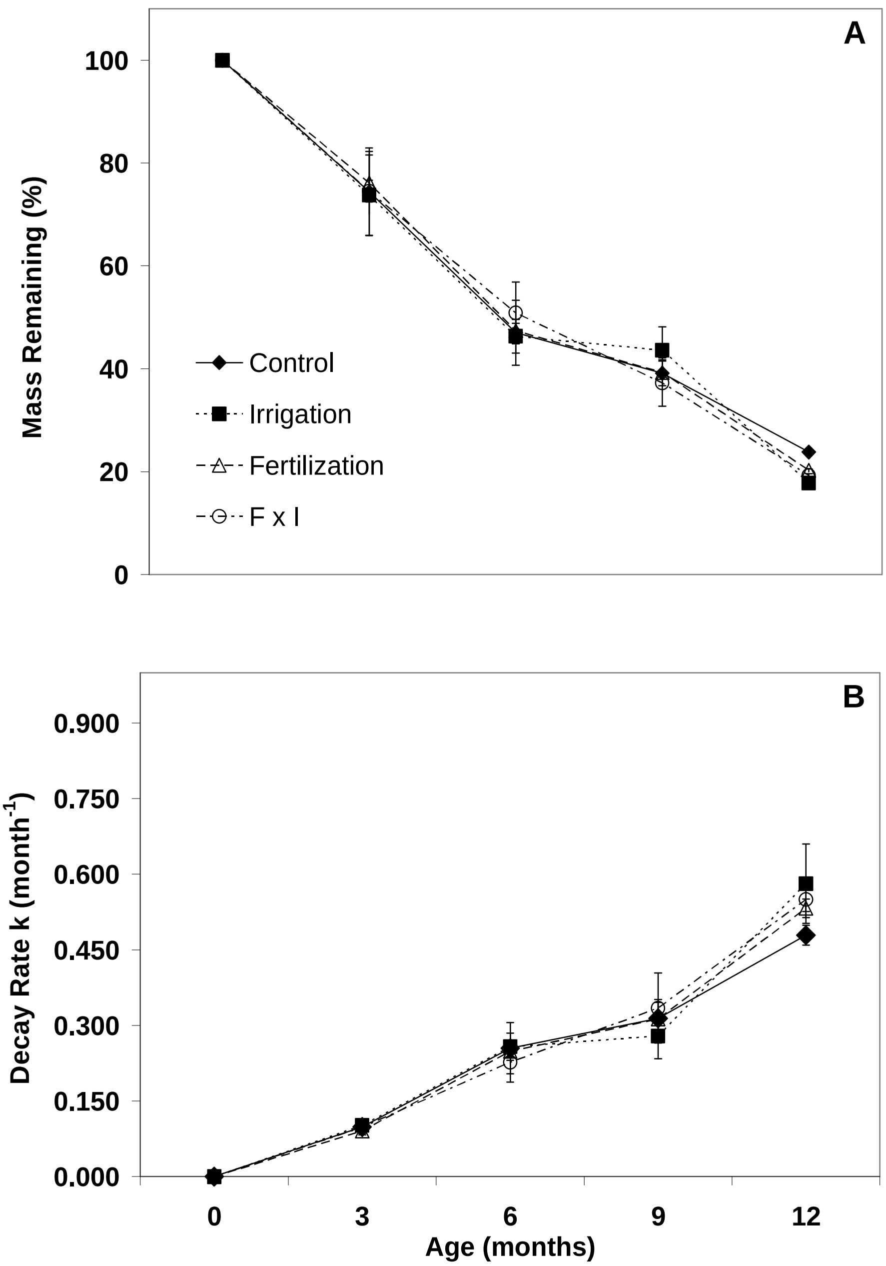

Fig. 5.1. Mean percent mass remaining (A) and decay rate constants (B) for forest floor Oi and Oe horizon material. Significant fertilization effect denoted by * (0.05), ** (0.001), *** (0.0001). Error bars represent 1 standard deviation.

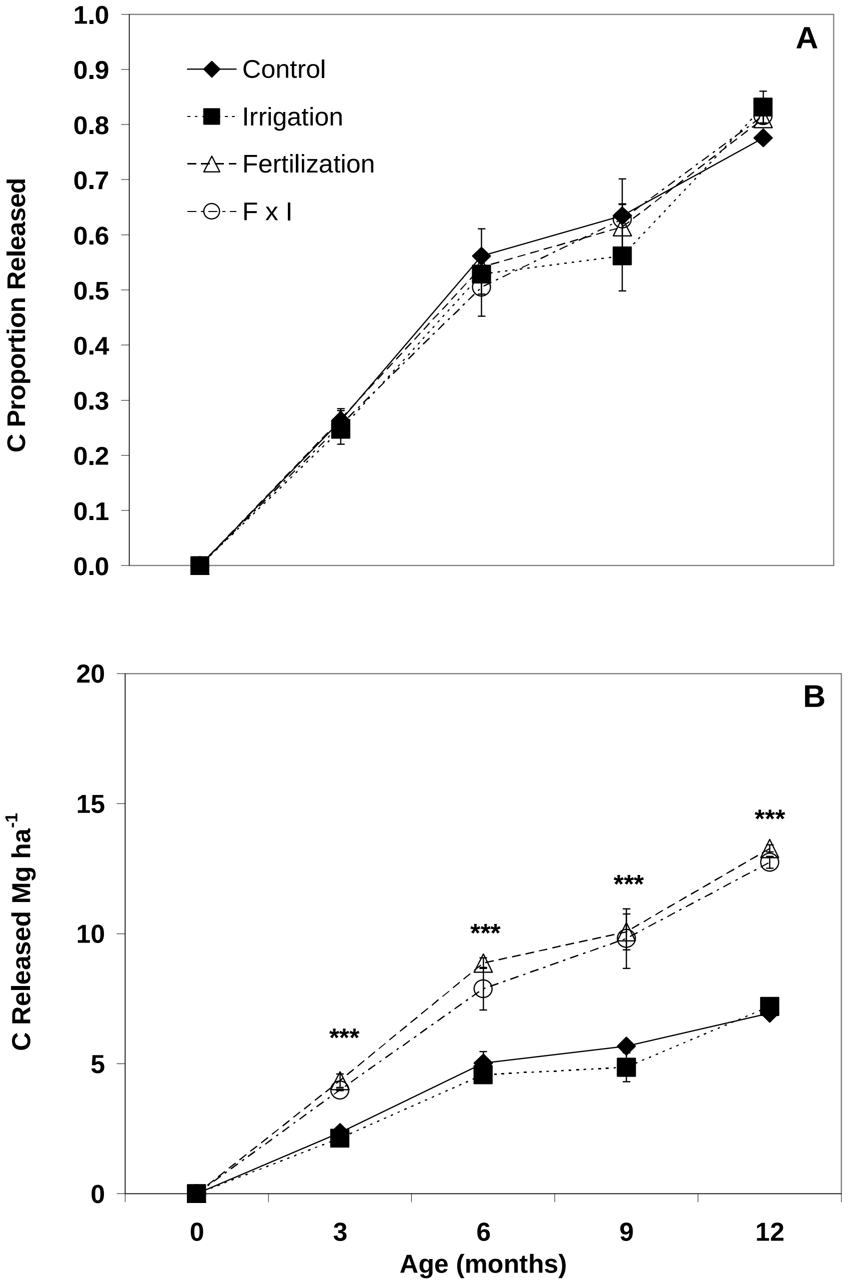

Fig. 5.2. Mean carbon proportion (A) and content (B) released from forest floor Oi and Oe horizon material. Significant fertilization effect denoted by * (0.05), ** (0.001), *** (0.0001). Error bars represent 1 standard deviation.

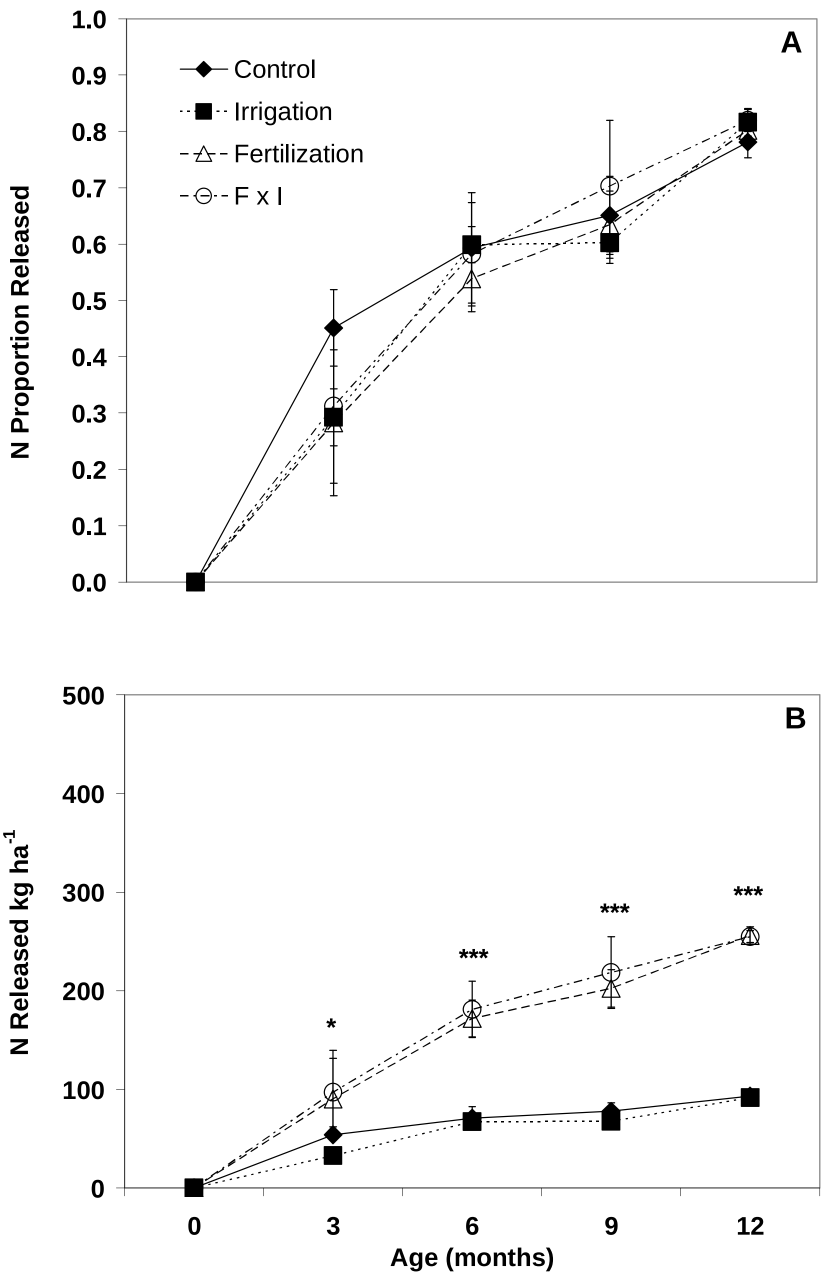

Fig. 5.3. Mean nitrogen proportion (A) and content (B) released from forest floor Oi and Oe horizon material. Significant fertilization effect denoted by * (0.05), ** (0.001), *** (0.0001). Error bars represent 1 standard deviation.

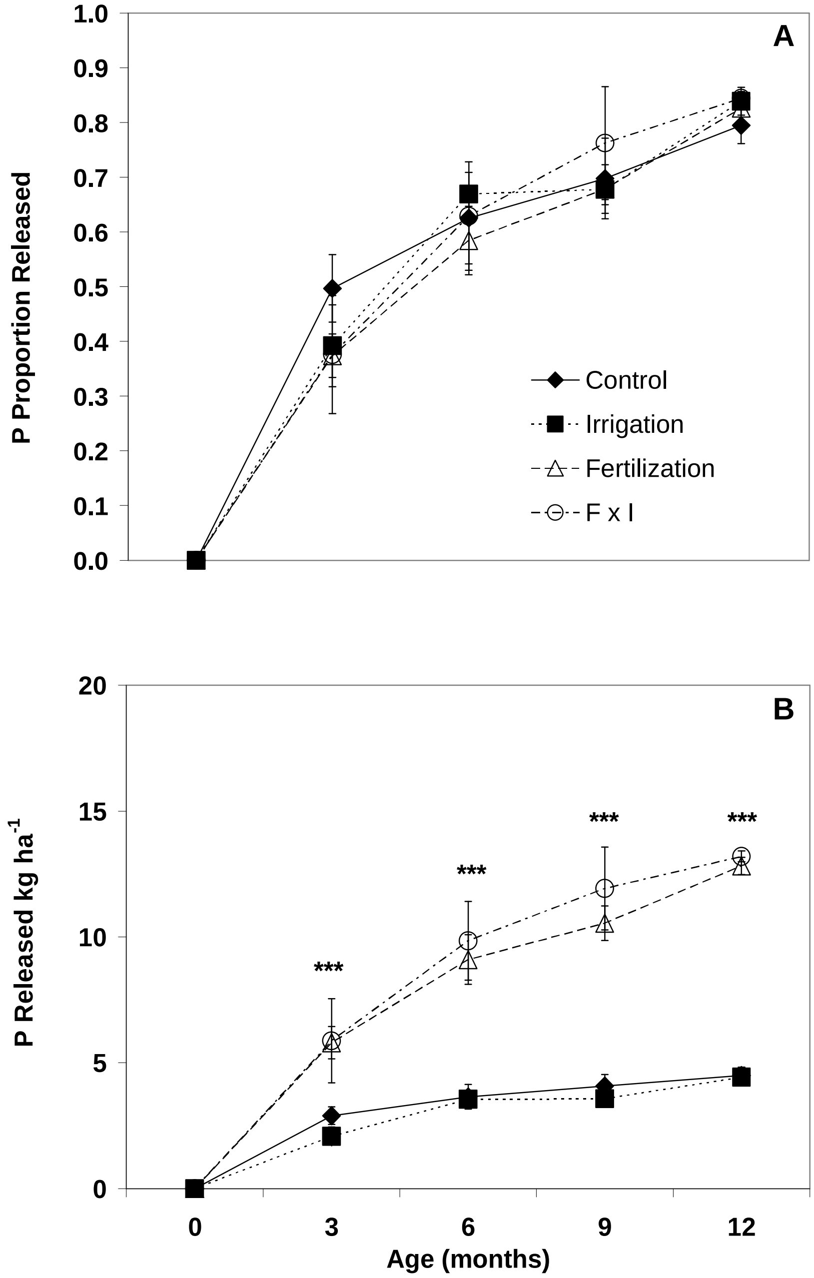

Fig. 5.4. Mean phosphorus proportion (A) and content (B) released from forest floor Oi and Oe horizon material. Significant fertilization effect denoted by * (0.05), ** (0.001), *** (0.0001). Error bars represent 1 standard deviation.

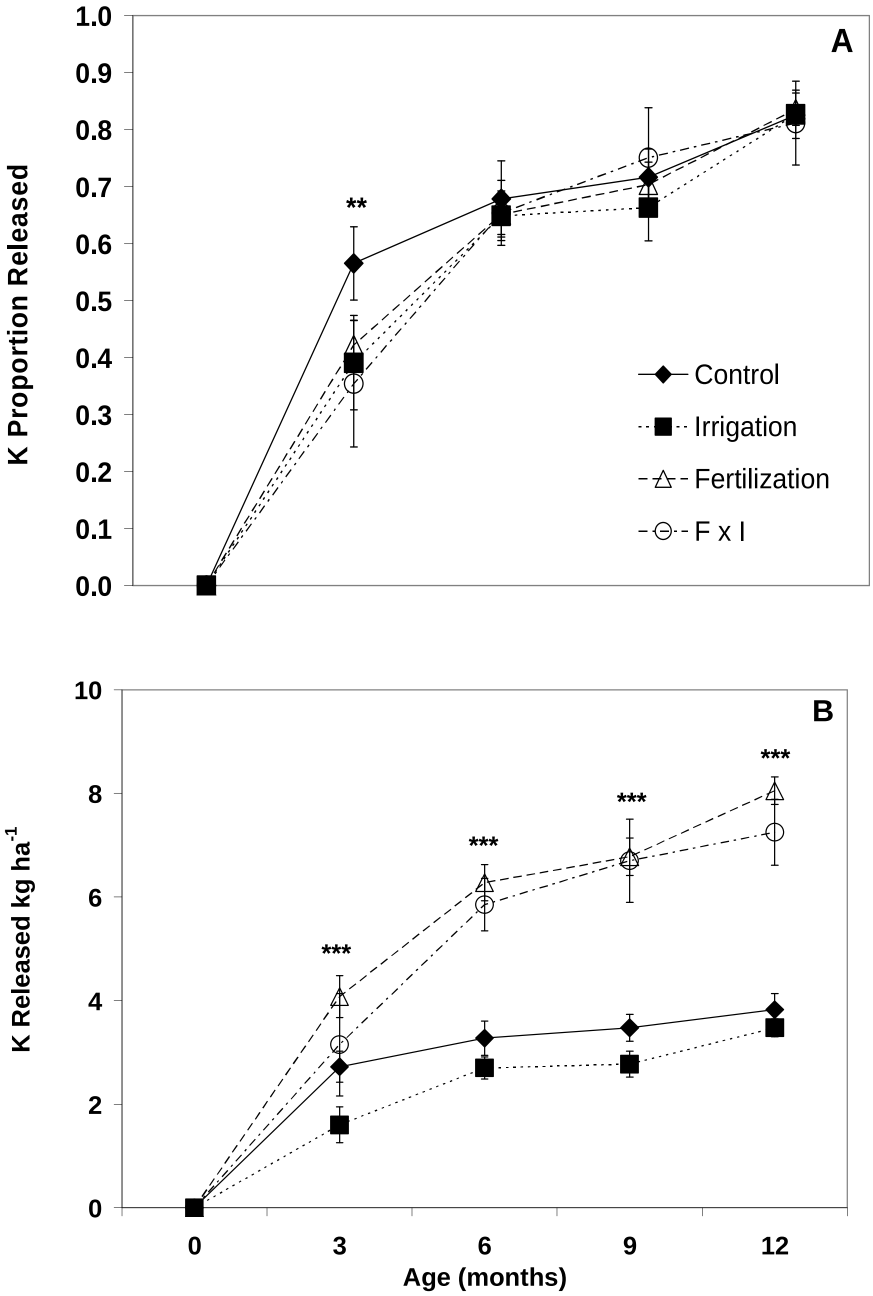

Fig. 5.5. Mean potassium proportion (A) and content (B) released from forest floor Oi and Oe horizon material. Significant fertilization effect denoted by * (0.05), ** (0.001), *** (0.0001). Error bars represent 1 standard deviation.

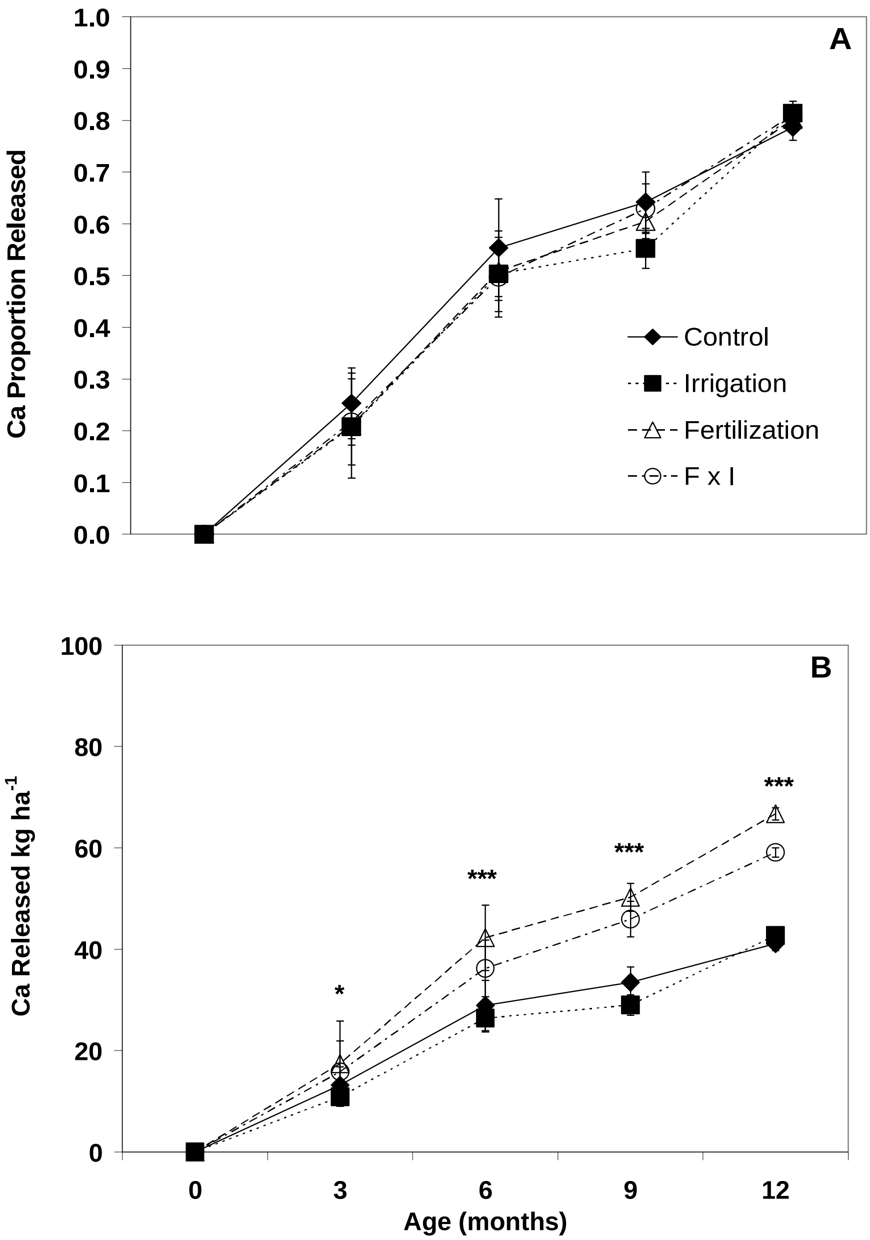

Fig. 5.6. Mean calcium proportion (A) and content (B) released from forest floor Oi and Oe horizon material. Significant fertilization effect denoted by * (0.05), ** (0.001), *** (0.0001). Error bars represent 1 standard deviation.

Chapter 6
Summary and Conclusions

6.1. Introduction

Fertilization of forest plantations is widely practiced in the southeastern U.S. to increase growth on infertile soils. These soils are typically both nitrogen (N) and phosphorus (P) limited. Fertilization with 225 kg N ha^{-1} and 28 kg P ha^{-1} have been shown to result in large and consistent growth increases of approximately 25% for loblolly pine (*Pinus taeda* L.) (Fox et al., 2007). Loblolly pine growth is increased through fertilization increasing foliar nutrient concentration resulting in a temporary increase in photosynthetic capacity that produces additional photosynthate used to increase leaf area (Gough et al., 2004) producing a growth response in fertilized trees (Albaugh et al., 1998). Similar foliar and productivity responses have been observed for sweetgum (*Liquidambar styraciflua* L.) following fertilization on nutrient limited soils (Scott et al., 2004; Cobb et al., 2008).

A key question that arises from fertilization of trees on these soils is whether the applied fertilizer benefits only the current trees in the stand or also improves long-term site quality. Long-term site quality would improve if fertilization increased mineral soil N and P and long-term N and P availability. Miller (1981) suggests that fertilization with N only benefits the current trees in the stand and does not increase N in a sandy soil. In contrast, others have found that fertilization with P benefits the trees and increases P in an array of soil textures including sands (Ballard, 1978; Gentle et al., 1986; Comerford et al., 2002; Crous et al., 2007; Everett and Palm-Leis, 2009). The loblolly pine forest floor is suggested to be the primary sink for N relative to the mineral soil due to an accumulation of N during decomposition (Polglase et al., 1992a; Piatek and Allen, 2001; Sanchez, 2001). In contrast, P is suggested to be readily released from loblolly pine litter soon after litterfall through leaching of inorganic-P (Polglase et al., 1992b). Results of these studies suggest a forest floor N sink and a potential mineral soil P sink that is related to the release of N and P from decomposing litter and mineral soil properties. These studies also suggest that long-term site quality and tree growth in terms of P nutrition will be improved by increasing mineral soil P but not N.

The overall objective of this research was to determine whether fertilization improves long-term site quality by increasing long-term mineral soil N and P availability and to assess factors contributing to this site response to N and P fertilization. Two forest plantation sites in the southeastern U.S. with similar soils that received similar total N and P applications were evaluated. One site was a 25-year old loblolly pine plantation (SETRES) while the other had 13-year old loblolly pine and sweetgum plantations (Mt. Pleasant). In the four previous chapters, five specific objectives were evaluated to compare accumulation of N and P in the forest floor and mineral soil among unfertilized and fertilized plantations (Chapter 2), determine soluble and residual N and P fractions and soluble carbohydrate and phenol fractions in foliage and litter (Chapter 3), determine whether higher N in the forest floor from fertilization resulted in increased release of N from the forest floor and increased mineral soil N availability (Chapter 4), determine loblolly pine forest floor decomposition rate and release of N and P expected to occur following disturbance (Chapter 5), and finally, to compare N and P accumulation and cycling among loblolly pine and sweetgum (Chapters 2, 3, and 4).

6.2. Chapter Results and Discussion

Chapter 2
Nitrogen and phosphorus retention in soils of fertilized loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) plantations

Our objective was to compare accumulation of N and P in the forest floor and mineral soil among unfertilized and fertilized plantations. Our hypothesis was that fertilization would primarily result in N accumulations in the forest floor with little N accumulation in the mineral soil. In contrast to N, fertilization would increase P in both the forest floor and mineral soil. Fertilization increased mineral soil N at SETRES (Table 2.8) and did not increase mineral soil N at Mt. Pleasant where fertilization ended 3 years prior to sampling (Table 2.12). Results suggested that the increase in mineral soil N observed at SETRES was temporary and would likely not be sustained following cessation of fertilization. Fertilization increased mineral soil P at SETRES (Table 2.9) and mineral soil P was elevated at Mt. Pleasant from past fertilizer applications (Table 2.13). Results showed that fertilization improves long-term nutrient availability only in terms of increased mineral soil P (Chapter 2). Nitrogen accumulations were primarily in the forest floor suggesting transient, not long-term, effects of N fertilization (Table 2.4, 2.6). At SETRES, fertilization increased the mineral soil Mehlich 3-P fraction (Table 2.8). At Mt. Pleasant, irrigation appeared to have increased P mobility in the mineral soil. Increased P mobility by irrigation was also suggested by a lower Mehlich 3 percentage of total-P in the loblolly I treatment (Table 2.15). Irrigation appeared to have also accelerated forest floor decomposition at Mt. Pleasant and decreased loblolly pine Oa horizon P (Table 2.6). Loblolly pine plantations appeared capable of capturing approximately 50% of N added through fertilization while sweetgum plantations appeared capable of capturing 100% of N (Table 2.16).

Soil responses supported the hypothesis that fertilization would primarily result in N accumulations in the forest floor with little N accumulation in the mineral soil and that fertilization would increase both forest floor and mineral soil P. Results suggested short-term rather than long-term effects of N fertilization. A lack of a long-term increase in N was likely due to the properties of the mineral soil. In contrast to N, the accumulation of P in the mineral soil was likely due to properties of the mineral soil promoting P retention.

Results indicated that irrigation appeared to have exerted a strong influence on the forest floor and mineral soil at Mt. Pleasant. Differences in forest floor responses to irrigation among sites suggests that forest floor decomposition of non-irrigated plots was limited by available moisture at Mt. Pleasant but was not limited at SETRES. Results provided evidence that irrigation not only accelerated forest floor decomposition at Mt. Pleasant, but also led to greater leaching of P from the forest floor. Results suggest that irrigation increased the mobility of mineral soil P. It is possible that at Mt. Pleasant, irrigation increased the decomposition of the forest floor and the quantity of forest floor leachate entering the mineral soil resulting in increased release of Al- and Fe-phosphate bound P through dissolution and ligand exchange. This effect was more pronounced in loblolly pine than in sweetgum due to the higher acidity of loblolly pine forest floor leachate. This process was supported by the reduction of the Mehlich 3-P percentage of total-P in the loblolly I treatment (Table 2.15).

Chapter 3
Effects of fertilization and irrigation on nitrogen, phosphorus, and soluble carbon fractions in loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) foliage and litter

In Chapter 2, it was suggested that fertilization results in only short-term increases in mineral soil and forest floor N while long-term increases were anticipated for P. This was attributed to mineral soil properties inhibiting N retention and promoting P retention. However, it was also essential to investigate fertilization effects on residual and soluble N, P, and C forms in foliage and litter. This was conducted in order to enable inferences to be made on differences in release of N and P and soluble C among unfertilized and fertilized systems. Our objective was to determine soluble and residual N and P fractions and soluble carbohydrate and phenol fractions in foliage and litter. Our hypothesis was that the forest floor N sink resulted from fertilization increasing N in the residual fraction and, in contrast, the forest floor was a source for fertilizer P due to fertilization increasing P in the soluble fraction.

At SETRES where fertilization was on-going to maintain optimal foliar N and P concentrations, fertilization was found to have altered N, P, and phenol fractions in foliage and litter. Fertilization increased foliar and litter N with the majority of this increase attributable to the residual protein-N fraction (Fig. 3.1A and B). Fertilization increased foliar and litter P with the majority of this increase attributable to the soluble inorganic- and organic-P fractions (Fig. 3.2A and B). Soluble carbohydrates were not altered by fertilization (Fig. 3.3A and B). Soluble phenols were highest in unfertilized treatments (Fig. 3.4A and B).

At Mt. Pleasant where fertilization was ceased 4 years prior to sampling, N was not higher in the fertilization + irrigation (FI) treatments and was comparable among loblolly pine and sweetgum (Fig. 3.1C and D). Phosphorus was also not higher in FI treatments but was higher in sweetgum than in loblolly pine (Fig. 3.2C and D). Foliar soluble carbohydrates and phenols exhibited responses attributed to water stress (Fig. 3.3C, 3.4C). In litter, these trends had disappeared and soluble carbohydrates and phenols were higher in sweetgum than in loblolly pine (Fig. 3.3D, 3.4D).

The hypothesis that the forest floor was a sink for fertilizer N due to fertilization increasing N in the residual fraction and a source for fertilizer P due to fertilization increasing P in the soluble fraction was accepted. It is important to note that residual-N and soluble-P comprised the majority of total-N and -P in unfertilized treatments as well. This suggests that fertilization served only to magnify the litter residual-N sink and soluble-P source that was otherwise present without fertilization.

At Mt. Pleasant, N fertilizer was last applied 4 years prior to sampling. Forest floor N and not mineral soil N was increased by fertilization (Chapter 2). At 3 years after fertilizer was last applied; mineral soil N availability was slightly higher in fertilized treatments (Chapter 4). However, foliar N concentration was no longer elevated 4 years after fertilizer was last applied suggesting either mineral soil N availability was no longer higher or the increase was not great enough to result in higher foliar N concentration. At SETRES, P fertilizer was last applied 8 years prior to sampling. Fertilization was reported to result in increased mineral soil P at SETRES (Chapter 2). Results suggest that this mineral soil pool supplied higher P to the trees as evidenced by sustained elevated P concentrations in foliage. The allocation of at least 50% of litter P in soluble fractions suggests that P will be readily released from the litter into the mineral soil. The allocation of 80-90% of litter N in the residual fraction suggests the opposite for N. Results suggest this sustained growth response to P fertilization was due to efficient cycling of P from the mineral soil into the foliage and then, release from the litter back into the soil to begin the cycle again.

The most interesting C fraction results were found at Mt. Pleasant. We speculated that foliage results appeared to indicate water stress. Litter results explained differences in forest floor mass among loblolly pine and sweetgum. Higher soluble carbohydrate concentrations in sweetgum likely result in faster decomposition and explain the lower sweetgum forest floor mass and lack of an Oa horizon. We propose higher decomposition rates observed in hardwoods relative to pines are driven by higher soluble carbohydrates in litter. Furthermore, based on results from SETRES where fertilization did not alter soluble carbohydrates in litter, decomposition rates of loblolly pine litter are not altered by fertilization. The divergent trend observed in loblolly pine and sweetgum foliar soluble carbohydrates, phenols, and tannins may be explained by differences in plant responses to water stress. Hardwoods respond to water stress by decreasing soluble leaf compounds in order to thicken the epidermis. Conifers, however, appear to respond to water stress by increasing soluble compounds and leaf osmotic potential since they already possess a thick epidermis.

Chapter 4
Residual effects of nitrogen fertilization on nitrogen availability in loblolly pine (*Pinus taeda* L.) and sweetgum (*Liquidambar styraciflua* L.) plantations

Results of Chapter 2 suggested large accumulations of N in the forest floor. In Chapter 3, it was determined that N accumulated in the forest floor due to residual-N comprising the largest fraction unlike P where soluble-P comprised the largest fraction. Due to large accumulations of N in the forest floor and that the majority of litter N was contained in a residual fraction, the need to quantify the release of N from this forest floor pool arose in order to determine whether this pool increased availability of N for the trees. Our objective was to determine whether higher N in the forest floor from fertilization resulted in increased release of N from the forest floor and increased mineral soil N availability. Our hypothesis was that higher N in the forest floor did not result in increased release of N from the forest floor and elevated mineral soil N availability. Fertilization at SETRES was on-going and ceased at Mt. Pleasant 3 years prior to sampling. This enabled us to test our hypothesis in a system where forest floor N was elevated by fertilization and 56 kg N ha^{-1} was added and in a system where forest floor N was elevated by fertilization and no N was added.

At SETRES, where 56 kg N ha^{-1} was applied prior to the measurement periods, mineral-N (NH_4-N and NO_3-N) detected by ion exchange membranes (IEM) at the forest floor/mineral soil interface was higher in fertilized treatments (Fig. 4.1, 4.2). The fertilizer application elevated IEM NH_4-N in months 1-3 and month 5. Cumulative growing season mineral-N was 3.5 g m^{-2} IEM in fertilized treatments. Fertilization increased mineral soil extractable NH_4-N but not NO_3-N (Fig. 4.3). Incubated soil cores detected an increase in cumulative nitrification. Fertilization increased cumulative nitrification NO_3-N from 2.2 kg ha^{-1} for unfertilized treatments to 19.2 kg ha^{-1} for fertilization treatments (Fig. 4.4B).

Fertilization ceased at Mt. Pleasant 3 years prior to the measurement period. Forest floor IEM NH_4-N was highest in fertilized loblolly pine and sweetgum in month 1

(Fig. 4.5A). NO_3-N was generally higher in sweetgum than in loblolly pine (Fig. 4.5B). Cumulative NO_3-N did not differ among treatments and species (Fig. 4.6). Cumulative NH_4-N was approximately 0.10 g m^{-2} IEM for loblolly FI and sweetgum I and FI and was 0.03 g m^{-2} IEM for loblolly I (Fig. 4.6). In months 4 and 7, mineral soil extractable NO_3-N was higher in fertilized treatments (Fig. 4.7A). Mineral soil cumulative mineralization did not vary by treatment or species and was characterized by immobilization throughout most of the growing season (Fig. 4.8A). Mineral soil cumulative nitrification NO_3-N varied by treatment and species and decreased on the order of loblolly FI (10.7 kg ha^{-1}) > loblolly I (5.5 kg ha^{-1}) > sweetgum FI (2.2 kg ha^{-1}) > sweetgum I (0.5 kg ha^{-1}) (Fig. 8B).

Results suggested that after fertilizer application to the surface of the forest floor as conducted at SETRES, fertilizer is not immobilized in the forest floor, but rather, fertilizer N travels through to the forest floor/mineral soil interface. After an application of 56 kg N ha^{-1}, forest floor IEM NH_4-N was no longer elevated in fertilized treatments after 5 months suggesting the fertilizer had travelled through the forest floor. In the absence of a fertilizer application at Mt. Pleasant, membranes detected N released from forest floor decomposition. The quantity of N entering the forest floor/mineral soil interface from decomposition of the forest floor was minimal compared to SETRES.

The magnitude of the increase in mineral soil extractable N at SETRES from fertilization was not as large as one would anticipate given the increase in IEM N from fertilization. While mineral soil extractable N was not increased as one would expect, mineral soil cumulative nitrification was greatly increased suggesting fertilization results in elevated mineral soil N availability. We propose it is possible that the discrepancy among quantities of mineral soil extractable N and nitrification in fertilized treatments at

SETRES arose from microbial immobilization. Fertilizer N entered the mineral soil and was immobilized by the microbial biomass leading to lower than expected extractable mineral-N. Upon incubation of the soil cores, this N immobilized in the microbial biomass was released and nitrification occurred. Fertilization results in elevated mineral soil N that may not be detectable by measuring extractable NH_4-N and NO_3-N. Instead, mineralization and nitrification must be measured since fertilizer N was first temporarily sequestered in the microbial biomass. At SETRES, a fertilizer application of 56 kg N ha^{-1} increased site N levels to the extent that production exceeded consumption resulting in a net release of N.

At Mt. Pleasant where fertilizer was last applied 3 years prior to sampling, decomposition of the forest floor resulted in increased release of N and elevated mineral soil N with this increase attributable only to NO_3-N. Mineralization measurements indicated NH_4-N immobilization throughout most of the growing season. Therefore, the hypothesis that higher N in the forest floor did not result in increased release of N from the forest floor and elevated mineral soil N was refuted. Results suggest that increases in forest floor N from fertilization result in elevated mineral soil N beyond the immediate effects of fertilizer application. However, the magnitude of elevated mineral soil N was greatly diminished relative to SETRES. Site N levels were diminishing indicated by production not exceeding consumption. Although mineral soil NO_3-N was higher in fertilized treatments, in the following year foliar N was not higher in fertilized treatments (Chapter 3) suggesting either this effect had disappeared one year later or the statistically significant increase was not great enough to be biologically significant. The increase in mineral soil NO_3-N observed in fertilized treatments appeared proportionately large compared to the increase in IEM N from forest floor decomposition. Since IEM only detected mineral-N, it is possible that the forest floor in fertilized treatments also released greater quantities of dissolved organic N (DON) which resulted in the observed elevated mineral soil N in the fertilized treatments. Loblolly pine exhibited higher NO_3-N in the mineral soil compared to sweetgum even though IEM N was similar. This could have occurred due to release of greater quantities of DON from the loblolly pine forest floor. Alternatively, this could have occurred due to greater immobilization of N in sweetgum due to higher soluble carbohydrates (Chapter 3) facilitating more microbial immobilization.

Chapter 5
Loblolly pine (*Pinus taeda* L.) forest floor decomposition and nutrient release following a simulated disturbance

Results from Chapter 2 indicated that fertilization results in an accumulation of N in the forest floor. In Chapter 3, it was determined that the majority of litter N is in a residual fraction contributing to an accumulation of N and slow release of N from the forest floor. Under the existing overstory canopy, *in situ* measurements of N release from the decomposing forest floor indicated N release proceeded slowly (Chapter 4). However, following a disturbance such as thinning or harvesting of the overstory, forest floor decomposition and release of N is increased. Evaluating the rate and magnitude of the release of nutrients from the forest floor following disturbance is important since the forest floor pool contains a substantial amount of N added through fertilization. Rapid release of these accumulated nutrients early in the next rotation may exceed seedling nutrient demand potentially resulting in a loss of a significant amount of site N capital.

However, increased forest floor decomposition and N release following a thinning disturbance has beneficial effects by increasing N availability to trees at a time when demand is relatively high. Our objective was to determine loblolly pine forest floor decomposition and release of nutrients in a simulated disturbance environment. Our hypothesis was that increased endogenous N and P from fertilization would not increase the rate of decomposition and nutrient release from the loblolly pine forest floor.
An experiment was conducted where litterbags containing loblolly pine Oi + Oe horizon material were incubated in an area with no overstory canopy simulating environmental conditions likely to occur following thinning or harvest.

Percent mass remaining and the decay rate constant did not vary among treatment levels (Table 5.2). The overall decay rate constant was determined for all the data ($k = 0.411$; $R^2=0.95$) and indicated a MRT of 12.2 months. Litterbags lost approximately 50% mass within 6 months and 80% within 1 year (Fig. 5.1). The proportion of C, N, P, and Ca released did not vary by treatment (Table 5.2). An irrigation effect was found for the proportion of K released and indicated a higher release in the control treatment for the first sampling interval. Treatment effects were observed for nutrient contents released (Table 5.2). At the end of the 1 year decomposition period, C content released from unfertilized and fertilized treatments was 7.1 and 13.0 Mg ha^{-1}, respectively (Fig. 5.2B). Nitrogen content released from unfertilized and fertilized treatments was 92 and 255 kg ha^{-1}, respectively (Fig. 5.3B). Phosphorus content released from unfertilized and fertilized treatments was 4.5 and 13.0 kg ha^{-1}, respectively (Fig. 5.4B). Potassium content released from unfertilized and fertilized treatments was 3.7 and 7.7 kg ha^{-1}, respectively (Fig.

5.5B). Calcium content released from unfertilized and fertilized treatments was 41.9 and 62.9 kg ha^{-1}, respectively (Fig. 5.6B).

The decomposition rate of the forest floor was not increased by higher endogenous N availability from fertilization supporting our hypothesis. That decomposition rates did not differ was most likely due to a lack of differences in soluble carbohydrates (Chapter 3). However, the decomposition rate was greatly increased over rates in similar studies where litterbags were incubated under the overstory canopy. Decay rate constants indicating mean residence times (MRT) for 99% mass loss of approximately 150 months was reported whereas incubation of litter under environmental conditions likely to occur following disturbance in this study indicated a MRT of 12.2 months. Different environmental conditions under the overstory canopy and in the open were likely the main factors driving the large differences in decomposition rates. Comparison of under-canopy and on-site weather station data from 1998 revealed that annual mean temperature and relative humidity differed by only 5%. However, annual mean photosynthetically active radiation and net radiation at the weather station were 17% and 58% greater than under the overstory canopy. This comparison of environmental data suggests that at SETRES, higher light levels rather than differences in temperature and moisture accelerated decomposition. Higher light levels increase photodegradation facilitating microbial decomposition and abiotic photochemical mineralization of C.

Under the overstory canopy, decomposing loblolly pine Oi horizon litterbags were found to accumulate N and P and release K and Ca. Decomposition of loblolly pine litterbags in an open area without an overstory canopy in this study also detected a release of K and Ca that was greatly accelerated. Instead of an accumulation of N and P, approximately 80% of the N and P were released within one year. Results suggest that a disturbance that removes the overstory canopy will greatly accelerate forest floor decomposition and nutrient release. Our results suggest the potential for a mid-rotation thinning to result in increased nutrient availability at a time when trees demand is relatively high. However, this depends on the size of the canopy gap. Further studies are warranted that investigate the influence of different thinning regimes on increases in nutrient availability from forest floor decomposition and the consequent influences on tree growth.

Results suggest that the forest floor N pool that has accumulated over the life of the stand will be quickly released following harvest. On sandy mineral soils, this can potentially result in a loss of a substantial amount of site N capital due to an inability of these soils to retain mineral-N. The 255 kg N ha^{-1} released within 1 year of harvest represents a substantial amount of N particularly for nutrient poor sites creating the need for site preparation techniques that minimize loss of this N.

6.3. Conclusions

Fertilization of loblolly pine and sweetgum growing on acidic, sandy mineral soils with N will not result in a long-term increase in site N availability and N supply to subsequent rotations will not be increased through higher mineral soil N. Increased mineral soil N from fertilization is temporary and not sustained after application of fertilizer N ends. Long-term increases in mineral soil N are not likely due to an inability of acidic, sandy mineral soils to retain NH_4-N and NO_3-N supplied from both fertilizer and forest floor decomposition. Nitrogen release from the decomposing forest floor proceeds slowly due to sequestration of 80-90% of litter N in a residual fraction. The forest floor accumulates N due to slow release of N during decomposition. Fertilization with N results in only temporary increases in mineral soil N availability that occur during fertilizer application and from forest floor decomposition. However, residual fertilization effects from forest floor decomposition diminish quickly and disappear approximately 3-4 years after cessation of fertilization. Future changes in N availability are primarily determined by decomposition of the forest floor following a disturbance that accelerates decomposition and release of N. A thinning disturbance would have beneficial effects by increasing N availability at a time when tree N demand is relatively high. At harvest, this increased N availability could result in loss of the forest floor N pool due to the inability of acidic, sandy mineral soils to retain mineral-N and to low N demand of seedling in the newly planted next rotation. At harvest, site preparation techniques that inhibit forest floor decomposition to minimize loss of N are necessary. In contrast to N, fertilization of loblolly pine and sweetgum with P results in a long-term increase in site P availability. Fertilization with P has lasting effects by increasing mineral soil P in stable forms that can be made available for plant uptake over time suggesting increased supply of P to trees in the next rotation. Retention of P in the mineral soil is likely due to the tendency of acidic, sandy mineral soils to accumulate P in Al- and Fe-phosphates. Accumulation in the mineral soil is also aided by P being readily released from litter due to sequestration of P in soluble fractions. The forest floor is a net source for P. In fertilized loblolly pine and sweetgum plantation growing on sandy mineral soils, N cycling proceeds slowly relative to P cycling and does not involve accumulations of stable N forms in the mineral soil. Conversely, P cycling proceeds quickly relative to N. Phosphorus is readily released from litter and accumulates in the mineral soil in a stable but plant available form that supplies P to trees over time.

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