SOIL-SITE PRODUCTIVITY INDICES AND TREE GROWTH IN LOBLOLLY PINE (*PINUS TAEDA* L.) PLANTATIONS

by

FABIO SARTORI

(Under the direction of Daniel Markewitz)

ABSTRACT

This research investigates soil variables relevant to growth in loblolly pine (*Pinus taeda* L.) plantations in the Piedmont of Georgia, USA. Under conditions of complete competing vegetation control, soil-site productivity relationships were investigated to test whether nitrogen (N) availability indexes and descriptive soil-site variables were correlated to stand growth. In addition, relationships between soil N availability and the soil organic matter light fraction (LF) were investigated.

I hypothesized that: 1) under simplified forest conditions due to complete competition control, indices of soil available N will be strong predictors of pine growth and 2) indices of soil available N will be positively correlated to the amount of LF in the soil.

In plots on Udults, and Udifluvents that received herbicide (H) and herbicide plus fertilization (HF) treatment resin extractable N, and potentially mineralizable N in long-term laboratory incubations were estimated. Resin extractable and laboratory estimates of N mineralization were correlated to stand growth and to the quantity of LF. In addition, plots (N=66) under experimental treatment combinations of Control (C), Fertilization (F), H, and HF were evaluated for changes in soil nutrients and carbon.

Resin extractable N was found well correlated (R^2=0.7) to mean annual increment (MAI) for 13 out of 17 sampling dates. Based on four sampling dates, resin core estimates of N availability were also significantly correlated (R^2=0.5) to the corresponding laboratory estimates of soil potentially mineralizable N. Another significant relationship was found between the LF pool and resin core estimates of N mineralization (r=0.63, n=25). Finally, the factorial analysis indicates that H, F, and HF treatments can alter the quantity of nutrients other than N with a clear effect of H treatments on soil carbon and exchange capacity.

On a regional scale, when controlling for understory competing vegetation on N limited soils, it is possible to improve on soil-site productivity relationships. The significant correlation between soil potentially mineralizable nitrogen and resin core estimates of N availability suggests that both techniques are valuable tools for the prediction of growth on N limited soils and that the LF is an important variable affecting N availability.

INDEX WORDS: soil-site relationships, available soil nitrogen, potentially mineralizable nitrogen, limiting factor, herbicide, fertilizer, light organic matter fraction.

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B.S., The University of Padua, Italy 1997

M.S., The University of Georgia, 2004

A Dissertation Submitted to the Graduate Faculty of The University of Georgia in Partial Fulfillment of the Requirements for the Degree

DOCTOR OF PHILOSOPHY

ATHENS, GEORGIA

2004

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DEDICATION

In memoria dei miei genitori, Ida e Carlo.

ACKNOWLEDGEMENTS

This research was supported through an assistantship provided by the D.B. Warnell School of Forest Resources (WSFR), The University of Georgia, and the Traditional Industries in Pulp and Paper Processing program of the State of Georgia. It was also made possible through the use of the WSFR facilities, and the permanent research plots and inventory data of the ongoing Consortium for Accelerated Pine Productivity Studies (CAPPS), and the Plantation Management Research Cooperative (PMRC).

I would like to thank my major advisor Dr. Daniel Markewitz for the opportunity to pursue my interests in studying our natural environment and career goals, providing all the tools and advice I needed at any time during these past years.

I thank all my other committee members Drs. Bruce E. Borders, Miguel L. Cabrera, Daniel B. Hall, and Lawrence A. Morris for their suggestions and reviewing this dissertation.

I would also like to thank Mr. Jay Brown for his assistance during my field and laboratory activities, Mr. Patrick Bussel, Mr. Mike Marsh, and Ms. Norma Rainwater of the Georgia Forestry Commission.

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CHAPTER 1

INTRODUCTION

For more than thirty years researchers have investigated the relationship between soil-site variables and tree growth in forest and pine stands, using different approaches to estimate site quality related to forest productivity as summarized by Carmean (1975). This author reports studies by Coile (1948; 1952) in the Piedmont under loblolly pine (*Pinus taeda* L.) stands regarding the relationship between site index, based on age and height measurements from dominant trees, and soil-site variables. In the same physiographic region, the prediction of biomass production from soil variables in both natural and managed (i.e., plantation) forests has focused largely on the estimation of nitrogen (N) and phosphorus (P) concentrations, contents, or fluxes since forest growth is often limited by available N and P (Mckee, 1977; Hart et al.1986). These limitations to growth often exist despite a high level of litter and soil total N or soil total P indicating that low cycling rates in forest soils can create inadequate N or P availability (Wells, 1977).

Unfortunately, many of these previous studies have not found strong relationships between soil variables and stand productivity. Among those few that found significant relationships there is one by Maimone et al. (1991), using the soil potentially mineralizable N method (N_0), proposed by Stanford and Smith (1972). These authors found that N_0 was significantly correlated with tree volume growth under fertilized 14-year-old loblolly pine plantation in the Coastal Plain of North Carolina.

One difficulty with virtually all of these previous studies, however, was the lack of control for competing vegetation, which in many cases can create a large but unmeasured covariate (Tiarks and Haywood, 1986; Schabenberger and Zedaker, 1999). For more than 20 years researchers have investigated the effect of the understory competing vegetation on tree growth in loblolly pine stands. A regional study in the southern U.S. based on results of the Competition Omission Monitoring Project (Miller et al. 1991; 1995) underscores the effects of different continuous treatments (i.e., no control, woody, herbaceous, and total control of the competing vegetation) during the first four years of stand development on tree growth. Many other studies (e.g. Powers and Jackson, 1978; Swindeel et al., 1988; Bacon and Zedaker, 1987; Haywood and Tiarks, 1990) report on the significance of competition control treatments on growth response in young loblolly pine plantations.

In this research I have attempted to provide a re-assessment of soil-site variability relationships on sites with complete and continuous control of competing vegetation. This simplification of the forest stand improves the quantification of forest stand productivity and may clarify the relationships between soil variables and stand growth. The major hypothesis of my work is that under conditions of complete competing vegetation control it is possible to improve on previous soil-site productivity studies based on N availability indices. I also hypothesize that indices of soil N availability will be positively correlated to the amount of soil organic matter light fraction in the soil.

To address my objectives in Chapter 2 I follow a twofold approach: 1) in the first component of this work I consider a broad survey approach by endeavoring to sample a relatively large number of plots but for a limited number of soil and site variables. In this study I try to identify major drivers of growth in loblolly pine (*Pinus taeda* L.) stands under conditions of complete and continuous competing vegetation control as well as useful covariates to be included in growth and yield models; 2) In the second component I consider intensive measures of soil available nitrogen (N) but on a more limited number of research plots to better explain the mechanism of the soil-site relationships. This second component was conducted within the Consortium for Accelerated Pine Productivity Studies (CAPPS) that includes continuous and careful control of the understory competing vegetation and fertilization treatments applied throughout the entire life of each stand.

In Chapter 3 I analyze decadal scale changes in the soil pools of the essential nutrients in three locations that are part of CAPPS. These plots (N=66) include the full factorial experimental treatment combinations of Control (C), Fertilization (F), Herbicide (H), and Herbicide plus Fertilizer (HF). I attempt to define the major growth responses in these systems, describe the decadal effects of the long term herbicide and fertilizer application on soil nutrient status, and evaluate the herbicide and fertilizer effects on soil C retention whose presence directly influences the capacity of the soil to retain the essential elements for tree growth.

In Chapter 4 I make use of soils from some of the same H and HF plots as above to: i) investigate the relationship between indices of N availability estimated via long term laboratory incubations and the in situ mixed bead resin core technique, ii) identify the effects of a continuous annual N fertilization on the quality of the light fraction soil organic matter (SOM), and iii) define possible relationships between estimates of N availability and the quality of the light fraction organic matter.

Finally in Chapter 5 I highlight what were the most relevant findings of this work.

CHAPTER 2

SOIL-SITE RELATIONSHIPS IN UNDERSTORY-VEGETATION-FREE LOBLOLLY PINE (*PINUS TAEDA* L.) PLANTATIONS[1].

[1] Sartori, F., Markewitz D., Borders E. B., and Shiver B.D. To be submitted to Forest Ecology and Management

Abstract

In intensively managed loblolly pine (*Pinus taeda* L.) plantations on Kanhapludults, Kandiudults, and Udifluvents, in the Piedmont of Georgia, the relationship between soil-site variables and tree growth was investigated in 19 plots under conditions of complete competition control (H). In these stands, the following variables were measured: percent slope and slope position, available water holding capacity, total C, total N, and C:N ratio for the 0-50 cm depth increment soil, as well as for the O horizon, and for foliage, soil extractable P and mineralizable N for the 0-10 cm depth, and potentially mineralizable N for the 0-15 cm depth soil. These variables were correlated to mean annual increment using multiple regression and simple linear correlation methods. In addition, twelve plots (two Herbicide (H) and two Herbicide plus Fertilizer (HF) treatments in three different locations) were intensively measured for soil available N using a mixed bead resin core technique in the 0-10 cm depth. Tree height inventory data consisting of individual tree repeated measures were used to fit a multilevel nonlinear mixed effects model to test whether soil available N can be a useful covariate under simplified conditions rather than only considering the categorical distinction between H and HF plots.

When considering soil site relationships across sites and studies from all 19 plots it was not possible to find any specific variable directly correlated to growth. Under more simplified conditions soil available N, as measured by the resin extraction, was found to be a good predictor of mean annual increment (R^2=0.7) for the cumulative resin extractable N data. On a regional scale, on N limited soils, when controlling for important confounding factors (i.e. understory competing vegetation) it is possible to improve on previous soil-site productivity studies.

Introduction

Site index, based on age and height measurements from dominant trees, and soil properties have been considered by many authors for estimating site productivity as reported by Carmean (1975). Of particular relevance to the current work in Georgia is the early soil-site relationship work of Coile (1948; 1952) in the Piedmont of North Carolina. Unfortunately, many of these previous studies have not found strong relationships between soil variables and stand productivity. One difficulty with virtually all of these previous works, however, was the lack of control for competing vegetation, which in many cases can create a large but unmeasured covariate (Schabenberger and Zedaker, 1999). In other words, previous productivity estimates were based only on crop trees while other non-crop trees, shrubs, and grasses remained unquantified.

Recent developments in the theory of nonlinear mixed-effects models (NLMMs) (Lindstrom and Bates, 1990) have been applied to tree growth, specifically growth and yield models, using different approaches and modeling different response variables of interest based on such repeated measures data (Hall and Bailey, 2001; Hall and Clutter, 2004; Zhang and Borders, 2004). In NLMMs the fixed model parameters can be expressed as a function of meaningful biological covariates or of specific treatment effects as in the traditional nonlinear regression model. The random parameters instead allow for the accounting of different autocorrelation and/or heteroskedasticity sources present in the data by imposing specific model errors and/or random effects correlation structures as presented by Pinheiro and Bates (2000, pp. 337-414).

The prediction of biomass production from soil variables in both natural and managed (i.e., plantation) forests has focused largely on the estimation of nitrogen (N) and phosphorus (P) concentrations, contents, or fluxes since forest growth is often limited by available N and P (Mckee, 1977; Hart et al.1986). These limitations to growth often exist despite a high level of litter and soil total N or soil total P indicating that low cycling rates in forest soils can create inadequate N or P availability (Wells, 1977).

In the lower Coastal Plain of South Carolina, McKee (1977), working on Hapludults, Ochraquults, and Albaaqualfs under loblolly pine stands aged 42 to 76, found that site index was well predicted using a multiple regression with soil total N, pH, and clay content as predictor variables. Within the same soil series, he found the highest correlation coefficient for N to be r=0.55. Also working in the Coastal Plain and Piedmont of the Southeast, Russ and Ballard (1982) found that site index was significantly and directly correlated to foliar N concentrations (r=0.4) and inversely correlated to the surface soil C to N ratio (r=-0.37) under fertilized, young (aged between 3 and 9 year old) and semimature (aged between 10 and 22 year old) loblolly pine stands. Hart et al. (1986), working across the Coastal Plain of North Carolina on Udults and Psamments under unfertilized loblolly pine plantations aged 5 to 11, found no significant relationships between soil N or other soil variables, and volume growth. In the corresponding fertilized plots, however, Hart et al. (1986) did demonstrate that N- and P-fertilizer growth response were correlated to soil Al (r=0.79) and NH_4-N (r=0.73). Maimone et al. (1991) working on Albaquults under fertilized 14-year-old loblolly pine plantation in the Coastal Plain of North Carolina found that N_0 (i.e., potential mineralizable N) correlated with 2-year tree volume growth (r=0.81 for April sampling date). Similarly, the recent work of Zhou and Dean (2003) in 45-55-year old, naturally regenerated loblolly pine stands on Paleudalfs in Louisiana found a poor although significant relationship (R^2=0.24, n=23) between site index and soil available N estimated through laboratory incubations.

Outside the Southeast, Powers (1980) working in California and southern Oregon, sampled well-formed dominant trees under natural and planted stands of ponderosa pine (*Pinus ponderosa* Laws.) on Ultisols, Alfisols, and Inceptisols and found that mean annual increment was well correlated to laboratory incubations used to estimate soil available N (R^2=0.59) when N was within 0 and 12 ppm.

None of the above studies, however, have accounted for the influence of competing vegetation on tree growth, which can be an important confounding factor (Tiarks and Haywood, 1986). For more than 20 years researchers have investigated the effect of the understory competing vegetation on tree volume growth in loblolly pine stands. A regional study in the southern U.S. based on results of the Competition Omission Monitoring Project (Miller et al. 1991; 1995) underscores the effects of different continuous treatments (i.e., no control, woody, herbaceous, and total control of the competing vegetation) during the first four years of stand development on tree growth. For example, on flatwoods in north-central Florida on a Haplaquod, the competition control treatment (mechanical plus chemical) under 4-year old loblolly and slash pine had tree biomass greater than in the corresponding control by ~11 and ~10 m^3 ha^{-1}, respectively. Many other studies (e.g. Powers and Jackson, 1978; Swindeel et al., 1988; Bacon and Zedaker, 1987; Haywood and Tiarks, 1990) report on the significance of competition control treatments on growth response in young loblolly pine plantations.

The above studies that found a significant relationship between soil available N and tree growth clearly indicate that there is potential predictive value in measures of soil nutrient availability. In addition, the work that demonstrates the effect of competition on tree growth highlights the extent of unexplained variability in these earlier studies that may have limited the use of soil predictors for plantation management. The presence of understory vegetation increases ecosystem complexity and thus the prediction of growth response based on N availability becomes more difficult. In the absence of the understory and competing tree vegetation it becomes easier to obtain a precise estimation of above ground biomass and annual growth increment through the simple measures of tree height and diameter.

The main aims of this study are to try to improve on some of the results obtained in previous studies, using plots that had continuous and careful control of competing vegetation and in some cases an annual fertilization treatment that has extended throughout the entire life of the stand.

To make use of these fertilized treatments, rather than simply considering the qualitative distinction between fertilized and non-fertilized plots that both included complete competition control, I tested, through the use of data-based models, whether soil available N is a useful covariate for predicting individual tree height growth.

The specific objectives of the study were met through a twofold approach: 1) in the first component of this work I considered a broad survey by endeavoring to sample a relatively large number of plots but for a limited number of soil and site variables. From this study I tried to identify major drivers of growth in loblolly pine (*Pinus taeda* L.) stands under conditions of complete and continuous competing vegetation control as well as useful covariates to be included in growth and yield models; 2) In the second component I considered intensive measures of soil available nitrogen (N) but on a more limited number of research plots to better explain the mechanism of the soil-site relationships. This second component was conducted within the Consortium for Accelerated Pine Production Studies (CAPPS, Borders and Bailey, 2001) that includes continuous and careful control of the understory competing vegetation and fertilization treatments applied throughout the entire life of each stand.

Materials and Methods

In this study I have attempted to provide a re-assessment of soil-site variability relationships on sites with complete and continuous control of competing vegetation. This simplification of the forest stand clearly improves the quantification of forest stand productivity and may improve the relationships between soil variables and stand growth.

In addition, I have used some statistical and modeling approaches (Lindstrom and Bates, 1990; Pinheiro and Bates 2000) new to this type of study to improve modeling growth based on a large, repeated, tree inventory dataset.

Research sites

All research sites are located in the Piedmont of Georgia. Three study sites are part of the long-term Consortium for Accelerated Pine Productivity Studies (CAPPS) at UGA (Borders and Bailey, 2001). These sites are located at the B.F. Grant Forest near Eatonton (33° 20' N, 83° 23' W), at Whitehall Forest in Athens (33° 57' N, 83° 19' W), and near Dawsonville at the Georgia Forestry Commission (34° 21' N, 84° 08' W). Two other sites are part of the Plantation Management Research Cooperative (PMRC) at UGA (Shiver et al., 1990) with one site located north of Athens, in Jackson County, GA (34°05'N, 83°46'W) and the other being an old spacing study (Pienaar and Shiver, 1993) also in the B.F. Grant Forest.

Sites are part of eco-region section 231A as described by McNab and Avers (1994). This section is identified as Southern Appalachian Piedmont (SAP) and consists of an intensely metamorphosed, moderately dissected plain consisting of thick saprolite, continental sediments, and accreted terrains. Elevation ranges from 100 to 400 m. The potential vegetation is oak-hickory-pine forest and southern mixed forest (Kuchler, 1964). The predominant vegetation form is evergreen forest. The loblolly (*Pinus taeda* L.) -shortleaf pine (*Pinus echinata* Mill.) cover type is common on disturbed areas and usually has an understory component of dogwood (*Cornus sanguinea* L.) and sourwood (*Oxydendrum arboreum* (L.) DC.). The growing season lasts about 205 to 235 days.

Athens is characterized by warm to hot summers, with mean July high temperature of 32 ºC, and moderately cold but highly variable winters, with mean January low temperature of -0.1 ºC and mean annual precipitation of about 126 cm (1961 to 1990, Univ. of Georgia, http://climate.engr.uga.edu/info.html). Similarly, Dawsonville has mean July high temperature of 30.7 ºC, and mean January low temperature of –0.1 ºC, with a mean annual precipitation of about 140 cm (1911 to 2003, Univ. of Georgia, http://www.griffin.peachnet.edu/bae/). The climate of Eatonton has a mean July high temperature of 32.5 ºC, mean January low temperature of 1.1 ºC, and mean annual precipitation of about 124 cm (1911 to 2003, Univ. of Georgia, http://www.griffin.peachnet.edu/bae/). In all locations precipitation has a maximum in early March and minimum in October.

Soils in the Athens and Eatonton sites presented an A or Ap horizon that ranged from sandy to sandy loam but was absent in the most eroded sites. The most common soil series at these sites (United States Department of Agriculture-Soil Conservation Service, 1968) are Cecil (fine, kaolinitic, thermic Typic Kanhapludults), Pacolet (fine, kaolinitic, thermic Typic Kanhapludults), Appling (fine, kaolinitic, thermic Typic Kanhapludults), and Davidson (fine, kaolinitic, thermic Rhodic Kandiudults). The Jackson County study plots are exclusively on Davidson series soils while the Dawsonville site is in the Etowah river floodplain on Congaree (fine-loamy, mixed, active, nonacid, thermic Typic Udifluvent) soils.

For all studies, tree volume data were available from inventories conducted by CAPPS or the PMRC of the D.B. Warnell School of Forest Resources (Pienaar and Shiver, 1993; Borders and Bailey, 2001; Zhang et al., 2002). Tree biomass was calculated based on annual individual tree repeated measures of tree diameter and height (each tree is identified by a unique number) and the corresponding volume was computed using an allometric equation (Harrison and Borders, 1996). Plot level estimates were computed by adding up individual tree volumes on a per hectare basis.

In CAPPS (Borders and Bailey, 2001) four treatments were applied to randomly chosen plots planted with a 1660 trees ha^{-1} density (Borders et. al, 2004): a Control (C), Herbicide (H), Fertilizer (F), and Herbicide plus Fertilizer (HF) treatments. The herbicide treatment consisted of a broadcast of sulfomethuron methyl during early spring of the first three growing seasons following planting and subsequent application of glyphosphate as needed in mid-summer each year thereafter. The fertilizer treatment consisted of 280 $kg \cdot ha^{-1}$ DAP plus 112 $kg \cdot ha^{-1}$ KCl in the spring and 56 $kg \cdot ha^{-1}$ of NH_4NO_3 mid-summer during the first two growing season. In subsequent growing seasons: 168 $kg \cdot ha^{-1}$ NH_4NO_3 early to mid-spring. At age 11 336 $kg \cdot ha^{-1}$ NH_4NO_3+ 140 $kg \cdot ha^{-1}$ triple super phosphate. At age 12, 560 $kg \cdot ha^{-1}$ super rainbow ® with micronutrients +168 kg ha^{-1} NH_4NO_3 early spring. At age 13 forward 336 $kg \cdot ha^{-1}$ NH_4NO_3 early spring.

The four PMRC study plots considered in this work were planted with an initial density of 1730-1853 trees ha^{-1} and received a herbicide treatment consisting of the use of glyphosphate prior to site preparation and subsequent application of glyphosphate, triclopyr, and, occasionally, diesel fuel to eliminate all competing vegetation (Shiver et al., 1990). One of the main objectives of this PMRC study was the comparison of the stand structure and yields of genetically improved and unimproved plantations, with and without the understory competing vegetation (Shiver and Rheney, 1991). Four other plots from the PMRC spacing study were used in this research: two with planting density of 998 trees ha^{-1} and two with 1483 trees ha^{-1} were sprayed before and one year after installation with Oust ® to control for the competing vegetation (Pienaar and Shiver, 1993). Due to the previous agricultural history of this site woody competition was extremely limited.

Field Investigation I

Study sites

For the first component of this study loblolly pine stands (4 in Athens, 11 near Eatonton, and 4 in Jackson County) of varying ages that received herbicide treatments were sampled in July and August 2000. The four plots in the Athens site had two 8-yr-old and two age 12-yr-old plots. In the Eatonton site there was one 6-year-old, two 11-year-old, five 13-year-old, and four (spacing study) 18-year-old plots. The four plots in Jackson County were all 14 years old.

Field methods

In each plot two 1m-depth soil profiles were randomly selected and dug using a stainless steel bucket auger (cylinder: 16.5 cm length and 10 cm diameter). If there was a lithic or paralithic contact, the depth was noted and the profile truncated at that depth. Landscape position and slope were also estimated at each sampling location. Color, texture, and thickness of each horizon were described in the field (United States Department of Agriculture-Soil Conservation Service, 1968). Texture was estimated using the “feel method” through a profile description to 1 m depth (Thein, 1979). A composite soil sample from the 0-50 cm depth was collected, stored cold, and returned to the laboratory.

On the basis of the observed texture and thickness of each soil horizon up to the 1 m depth, available water holding capacity was estimated. The following empirical parameters were utilized: 0.05 cm water cm^{-1} soil for sand and loamy sand textures; 0.1 cm water cm^{-1} soil for loamy very fine sand and coarse sandy loam; 0.15 cm water·cm^{-1} soil for sandy loam, sandy clay loam, clay loam, loam, silty clay, sandy clay and clay; and 0.2 cm water·cm^{-1} soil silt loam, silty clay loam and silt (Brady, 1974).

Tree foliage of the first flush of 1999 was also sampled by detaching one or two small branches from the high part of the crown using a shotgun and composited by plot. The thickness of the litter layer (i.e., the soil O horizon) was measured and a sample collected at each of two sampling points using a 15-cm diameter by 10-cm high brass core.

During June 2001 an additional survey was conducted on the same sites, using a stainless steel probe (diameter 2.5 cm, length 60 cm) with removable internal liners and a slide hammer: ten randomly selected 0-10 cm depth sub-samples within each plot were composited into one plastic bag, stored cold, and returned to the laboratory. In addition, the same 19 plots as above, plus an additional plot near Eatonton (N=20) that also received a complete competition control treatment (H), were sampled, using a stainless steel probe (diameter 2.5 cm, length 60 cm) with removable internal liners and a slide hammer. The O horizon was completely removed to avoid inclusion into the soil sample of any organic material derived from the litter layer during sample collection. A sample of 15 cores per plot at 0-15 cm depth was collected during the same time and composited in the same bag, maintained on ice, and immediately returned to the laboratory.

Laboratory methods

Composited 0-50 soil samples for chemical analysis were air dried and passed through a 2-mm sieve prior to analysis. Total nitrogen was measured on an Alpkem auto-analyzer (OI Analytical, College Station, TX) using the phenate method after digestion with H_2SO_4-H_2O_2 and H_2SeO_2 as a catalyst (Kuo, 1996). Total C was measured after pulverization with a dry combustion technique using a CE Elantech (Lakewood, NJ, USA) CNS analyzer (Bremmer and Mulvaney, 1986). Soil C, N, and the corresponding soil C:N ratios were considered as an index to be correlated to mean annual increment.

Litter layer and foliar samples were stored cold, returned to the laboratory, dried at 105°C to constant weight and weighed. Litter and foliar total C and N were measured after pulverization with a dry combustion technique using a CE Elantech CNS analyzer. Litter and foliar C, N, and the corresponding C:N ratios were considered as an index to be correlated to mean annual increment.

The 0-10 cm depth field moist soil samples were analyzed for total N measured on an Alpkem auto-analyzer (OI Analytical, College Station, TX) using the indophenol-blue method after TKN digestion ((U.S. Environmental protection Agency, 1983b). Available soil nitrogen (NH_4-N and NO_3-N), using a 2M-KCl extraction was also performed, using a 1:10 soil/solution (w/w) ratio. The extract solution was analyzed for NH_4-N and NO_3-N concentrations colorimetrically on a Alpkhem auto-analyzer (OI Analytical, College Station, Texas) using, respectively, the alkaline phennate and cadmium reduction method (U.S. Environmental protection Agency, 1983a; 1983b). Finally, for N, a 30-day incubation at 27 º C was performed to assess N mineralization potential (Hart et. al., 1996). Extractable P was estimated on these same samples using the double-acid (H_2SO_4-HCl) extract (Kuo, 1996).

The fresh 0-15 cm-depth soil samples were passed through a 4-mm sieve and stored at 4°C until the incubation began based on the procedure described by Maimone et al. (1991) who refer to Stanford and Smith (1972). Each soil sample was placed inside a 3.5-cm i.d. polyvinyl chloride (PVC) plastic pipe and incubated at 27 °C and relative humidity of 99%. Each sample was leached with an extracting salt solution at cumulative intervals of 7, 30, 57, 105, and 158 d. To facilitate the flow of the solution through the soil 40 g of 0.85-mm acid washed ignited silica sand was mixed with 40g of soil sample and a 1-cm plug of glass wool was placed on the top of the soil-sand mixture to prevent disturbance of the soil with the addition of leaching solutions. A constant 0.003 MPa pressure was maintained using a compressor to collect leachates.

During each extraction, 42 sample tubes (two replicate tubes and two laboratory blanks) were leached with three 50-mL volumes of 0.01 M $CaCl_2 \cdot 2H_2O$ extract solution to remove NH_4-N and NO_3-N. Tubes were then leached with one 25-mL volume of a nutrient solution lacking N to restore nutrient status in the soil and to account for the possible influence of low P availability on N mineralization after each extraction. The composition of this solution was: 0.002 M $CaSO_4 \cdot 2H_2O$, 0.002 M $MgSO_4 \cdot 7H_2O$, 0.01 M KH_2PO_4, and 0.0025 M K_2SO_4.

Extraction was carried out until leachate was no longer being removed. During the first extraction the volume of leachate obtained was measured to verify complete removal of added solutions and to adjust for minor variation in moisture retention. Sample moisture content after the first extraction was assumed to be uniform and approximately equal across samples because of the previous moisture removal under pressure. The extract solution was analyzed for NH_4-N and NO_3-N concentrations colorimetrically on a Alpkhem auto-analyzer.

Statistical analyses

Pearson correlation coefficients as well as multiple regression diagnostics associated with several model selection criteria (R^2, Cp, AIC, SBC and PRESS, SAS Institute Inc., 1990) were used for model selection, having the 2000 and 2001 measures of soil and site characteristics, as predictors, and mean annual increment, as response variable.

The N_0 value was determined by fitting cumulative data considering the one-pool equation (Cabrera et al., 1994) based on first order kinetics: $N_t=N_0\ (1-e^{kt})$, where N_t (ug g^{-1}) is cumulative nitrogen mineralized at time t (days), N_0 (ug g^{-1}) is a starting estimate of potentially mineralizable nitrogen, and k (d^{-1}) is its corresponding rate constant of mineralizazion. To estimate N_0 and k a multilevel nonlinear mixed-effects model was fitted with a banded and diagonal correlation structure of the random effects specific for plot and for site, respectively (Pinheiro and Bates, 2000, pp. 337-414). In this analysis, the CAPPS plots near Eatonton named the "Powerline" and the "Monitor" were considered as separate locations. Site was considered as a categorical, grouping variable to fit this model to soil potentially mineralizable N cumulative data.

Field Investigation II

Study sites

The second component of this study used twelve plots of the CAPPS study, only four of which overlapped with those used in field study I. Two H-HF plot pairs located in three different locations were employed. A total of 12 H-HF plots (2 H+2 HF in three different locations) were considered in this more intense field investigation from July 2002 through September 2003.

Field methods

To obtain an index of soil available N, the mixed bead resin core technique (Binkley and Mattson, 1983) was used to capture NO_3-N and NH_4-N in the soil solution that had been transported throughout a 10 cm depth soil core contained by a PVC tube (20 cm length and 3.5 cm inside diameter). Four or six bag/tube assemblies per plot were systematically assigned to each plot considering a square grid to account for local variability.

For resin bag placement, soil O horizon was removed and a 3.5 cm inside diameter PVC tube of 20 cm length was driven into the ground to a 10 cm depth. The PVC tube with soil was then removed from the ground maintaining the soil core internally and a bag was placed at the bottom and made flat. The PVC tube was put back in place and gently pressured at the surface to seal the tube. PVC tubes were left open to the atmosphere. Four or six PVC-tubes (six for the majority of plots and collection dates) were placed on an approximate square grid in each plot on a monthly or bi-weekly basis. This procedure was repeated 17 times, with 10 monthly samplings starting July 2002 through May 2003 and 7 bi-weekly samplings from June to September 2003.

Laboratory methods

Multiple 4 × 4 cm size resin bags were constructed using Nitex ® 03-80/37 nylon material (SEFAR AMERICA, Depew, NY) and nylon thread, and 4 g of 20-50 mesh ion exchange resin from BIO-RAD (Hercules, CA) using 2 g of AG® 1-X8 chloride form and 2 g of AG® 50W-X8 hydrogen form. Prior to placement in the field, each resin bag was placed in a separate centrifuge tube in 40 ml of 1 M HCl solution and recharged by shaking it on a reciprocating shaker at 200 rpm for at least 24 hours and rinsed with deionized water afterwards.

Statistical analyses

The different collection dates were correlated across all 12 locations with stand level mean annual increment measured in 2001 after testing the normality assumption of the mean annual increment data. In 2001 plots aged 13, 12-14, and 13-15 year old in Athens, Eatonton, and Dawsonville, respectively, were utilized.

To test whether soil available N can be used as a useful covariate to model individual tree height for these herbicide treated stands, a multilevel non-linear mixed effects-model (NLMM) was developed. Non-linear mixed effects models (Lindstrom and Bates, 1990) applied to growth data can allow for the incorporation of biologically meaningful covariates that can help explain part of the variability in growth and, thus, reduce the number of estimated model parameters (Pinheiro and Bates, 2000). Futhermore, multilevel NLMMs help account for the presence of autocorrelation and heteroskedasticity that arise due to grouping and repeated measurements through time by including random group effects and other assumptions regarding the variance-covariance structure of the data (Hall and Bailey, 2001).

In the current study, NLMMs were applied considering the Chapman-Richards three-parameter model function to fit repeated measures of individual tree heights grouped by plot and individual tree. The data set being used for this modeling effort is a large (N=4551, e.g. 12 plots×30 trees×13 repeated measurements) subset of the inventory data of the CAPPS study (Borders et. al., 2004). It consists of repeated measures corresponding to a specific tree within a certain plot since stand establishment. The number of tree repeated measures vary slightly depending on stand age. In a 0.05 ha plot, measurement trees have been marked with a specific number and remeasured each year. For each of the 12 H and HF plots of Field Investigation II, 30 individual trees, out of approximately 81 contained in the measurement plot, were randomly selected and used to fit a NLMM with plot and tree specific random effects.

The model parameters of the Chapman-Richards equation were expressed as a function of location (using the following dummy variables: Z1=Z2=0, for Athens; Z1=0 and Z2=1, for Eatoton; Z1=1 and Z2=0, for Dawsonville), treatment (Fert=0, H; Fert=1, HF), and soil available N (continuos, log-scale). In relation to y_{ijk}, the j_{th} tree in the i_{th} plot at the k_{th} measurement year the following model is fitted (M1):

$$y_{ijk} = \gamma_{1ij}\{1 - \exp(-\gamma_{2i} age_{ijk})\}^{\gamma_{3ij}} + \varepsilon_{ijk} ,$$

$$\gamma_{1ij} = \beta_1 + \beta_2 \cdot Fert_i + \beta_3 \cdot Z1_i + \beta_4 \cdot Z2_i + \beta_5 \cdot LOGN_i + b_{0,ij}^{(1)} + b_{1,i}^{(1)} ,$$

$$\gamma_{2ij} = \beta_6 + \beta_7 \cdot Fert_i + \beta_8 \cdot Z1_i + \beta_9 \cdot Z2_i + \beta_{10} \cdot LOGN_i + b_{0,ij}^{(2)} + b_{1,i}^{(2)} ,$$

$$\gamma_{3ij} = \beta_{11} + \beta_{12} \cdot Fert_i + \beta_{13} \cdot Z1_i + \beta_{14} \cdot Z2_i + \beta_{15} \cdot LOGN_i + b_{0,ij}^{(3)} + b_{1,i}^{(3)} ,$$

where β_1, β_2,…,β_{14}, and β_{15} are fixed parameters. All three parameters (γ_1, γ_2, and γ_3) are allowed to vary from plot to plot and from tree to tree through dependences of random tree effects, $b_{0,ij}^{(1)}, b_{0,ij}^{(2)}, b_{0,ij}^{(3)}$, and random plot effects, $b_{1,i}^{(1)}, b_{1,i}^{(2)}, b_{1,i}^{(3)}$. In addition to normal errors, a normal distribution for both the tree-specific and plot-specific random effects is assumed as follows:

$$\begin{bmatrix} b_{0,ij}^{(1)} \\ b_{0,ij}^{(2)} \\ b_{0,ij}^{(3)} \end{bmatrix} \overset{iid}{\sim} N_3(\mathbf{0}, \phi\boldsymbol{\Sigma}_0) , \qquad \begin{bmatrix} b_{1,i}^{(1)} \\ b_{1,i}^{(2)} \\ b_{1,i}^{(3)} \end{bmatrix} \overset{iid}{\sim} N_3(\mathbf{0}, \phi\boldsymbol{\Sigma}_1) , \qquad \boldsymbol{\varepsilon}_{ij} \overset{iid}{\sim} N(\mathbf{0}, \phi\boldsymbol{\Sigma}_2)$$

The M1 model was fitted using the S-PLUS nlme function (with uncorrelated random effects (i.e., $\boldsymbol{\Sigma}_0 = \boldsymbol{\Sigma}_1$=I) for each Chapman-Richards parameter (γ) at each level, considering a continuous AR(1) model error autocorrelation structure, $\boldsymbol{\Sigma}_2$, as defined in Pinheiro and Bates (2000).

The first model (M1) was compared to a corresponding nested model (M2) without soil available N as a covariate in the model. The major difference between M1 and M2 is that parameters of the Chapman-Richards equation vary only as a function of treatment and location in M2, assuming:

$$\gamma_{1ij} = \beta_1 + \beta_2 \cdot \mathrm{Fert}_i + \beta_3 \cdot \mathrm{Z1}_i + \beta_4 \cdot \mathrm{Z2}_i + b_{0,ij}^{(1)} + b_{1,i}^{(1)},$$

$$\gamma_{2ij} = \beta_5 + \beta_6 \cdot \mathrm{Fert}_i + \beta_7 \cdot \mathrm{Z1}_i + \beta_8 \cdot \mathrm{Z2}_i + b_{0,ij}^{(2)} + b_{1,i}^{(2)},$$

$$\gamma_{3ij} = \beta_9 + \beta_{10} \cdot \mathrm{Fert}_i + \beta_{11} \cdot \mathrm{Z1}_i + \beta_{12} \cdot \mathrm{Z2}_i + b_{0,ij}^{(3)} + b_{1,i}^{(3)},$$

A similar specification of the Chapman-Richards parameters has already been discussed in more detail by Hall and Bailey (2001).

To select the best model between M1 and M2 I used an approximate F test (Pinheiro and Bates, 2000, p.323). This test addresses the major inquiry of interest whether or not the inclusion of soil available N as a covariate offers a better model fit.

Results and Discussion

Information collected during the 2000 site survey are presented in Table 2.1. Plots were largely located in midslope positions. Slopes ranged nine-fold in slope from 2 to 18 %. The litter layer bulk density ranged five-fold, from 0.12 to 0.58 g cm^{-3}, while litter layer thickness had the least range, from 4 to about 6 cm. Site characteristics as well as other soil descriptive data such as color or texture (data not shown) were not well correlated to mean annual increment across the plots.

The C and N data for the 0 to 50 cm depth soil increment, the litter layer and foliage are presented in Table 2.2 along with the available water holding capacity data (AWHC). Nitrogen concentration in this upper portion of the soil ranged by two-fold, litter layer N by three-fold, and foliage less than one-fold. Based on the correlation matrix (Table 2.3), soil available water holding capacity was found to be significantly correlated ($r=0.6$, $p<0.01$, $n=19$) to mean annual increment. The other significant relationship found based on the 2000 survey is between mean annual increment and the litter C to N ratio ($r=-0.866$). Similar results underlying the significance of available water holding capacity were obtained using a multivariate regression approach.

In the Piedmont, water availability is generally not thought to be the most important limiting factor (Jokela et al., 2004) as reported also by Albaugh et al. (2004) who underscore the important role of nutrients rather than the water resource in accelerating growth. In the current study, however, because available water holding capacity is estimated as a function of soil depth it may represent an indirect estimate of rooting depth especially on highly eroded hill shoulders where tree growth is often limited by the presence of a thick saprolitic or clayey layer. An example of such an occurance is provided by Fralish (1994) in mature stands of mixed community species in the Shawnee Hills of Illinois. He found that basal area growth was significantly correlated to available water holding capacity and effective soil depth ($r=0.81$ and $r=0.82$, respectively).

The other significant relationship between MAI and litter soil C to N may be explained considering that a higher C to N ratio corresponds to a lower rate of N mineralization, which would limit N supply and growth.

The soil values measured in the 0 to 10 cm layer in 2001 (Table 2.4) were generally higher in N than the measures for the 0 to 50 cm soil samples. Higher N values in the 0 to 10 cm soils relative to the 0 to 50 cm soils are to be expected since soil organic matter concentrations generally decrease with depth. Aerobic incubations in the 0 to 10 cm soils varied broadly over the plots ranging from 0.7 to 20 ug-N g^{-1} soil 28-$days^{-1}$.

This range of values is consistent with similar incubations for Piedmont soils comparable to those reported by Cabrera (1993) for Cecil soils under a pasture with values for the N pool released of 4.39-10.4 mg N kg^{-1} and the corresponding k of 0.0457-0.041 h^{-1}. The estimated soil available N via the 28-day incubation, however, had a very high variability between laboratory replicates making inference among plots difficult.

Values for soil extractable P on these plots had a limited variation across most of the plots but were highly elevated in the PMRC spacing study plots. The concentrations for extractable P are typical for the region and are consistent with the known agricultural history of the spacing study site.

Finally, values for N_0 (Table 2.5) were comparable to those reported by Cabrera (1993) whereas the k mineralization rate constant found in this study was always lower than the variation range reported by the same author. A lower k may indicate differences in the soil organic matter quality or may be related to the different incubation time used. Other factors that can influence the N_0 and k estimation are summarized by Cabrera et al. (1994).

When correlating mean annual increment to the 2001 measured variables there was no overall significant relationship across sites with potentially mineralizable N (N_0), (Table 2.6) or extractable P. If these relationships were considered by location, mean annual increment was found significantly correlated to soil potentially mineralizable N (R^2=0.33) but only for the Eatonton plots (Figure 2.1) and even this relationship was not very strong. Similar difficulties finding relationships between soil N and P availability and growth have been found in other previous studies in the Coastal Plain and Piedmont regions (Hart et al., 1986; Lea and Ballard, 1982). The relationship found between N_0 and tree growth is in agreement with the findings of Maimone et al. (1991, r=0.81 or R^2=0.65 for April sampling date) and underscores the potential value of this technique to reflect field N availability in forest soils.

Based on the findings of Field Investigation I it appeared that in the presence of more homogenous conditions under the H treatment it would be possible to find direct relationships between growth and estimates of N availability. For this reason Field Investigation II considered a more homogeneous group of plots with respect to age and planting density to better investigate the relationship between soil available N and growth. In Field Investigation II, resin N rates over the course of the sampling period (Figure 2.2) were found to be an order of magnitude higher in the HF plots relative to the H plots. Peaks in resin N were observed during September-October and May-June. The May-June peak likely reflects only the annual fertilizer application in April.

In comparing the observed growth responses in the CAPPS plots to the corresponding field estimates of resin N, there is a clear correspondence between growth rates and resin N for the H and HF plots. Mean annual increment was significantly and strongly correlated to resin N (log scale) across sites for almost all collection dates (Figure 2.3). Thirteen collection dates out of seventeen dates had $0.5<R^2<0.8$ and four had a poor relationship with two separate clusters representing the H and HF plots, respectively. A log transformation was used to stabilize variance because the HF plots had a much higher variability and higher values, in many cases 100-fold higher compared to the corresponding H plots. To obtain an overall site productivity index the cumulative amount of available N over the 17 collection dates was considered. In this case of the cumulative values the different plots maintain a relative high-low rank and are well correlated to mean annual increment (Figure 2.4, R^2=0.7).

The resin extractable values for the HF plots across locations ranged from ~ 9 to >1700 μg $N \cdot g^{-1}$ while H values ranged from ~4 to <860 μg $N \cdot g^{-1}$. Season patterns in resin extractable N are similar to those described by Vitousek and Mattson (1985) in loblolly pine stands shortly after harvest. The strong relationships between the cumulative resin extractable N and MAI is similar to the relationship found by Powers (1980) in ponderosa pine stands using laboratory incubations. On the current plots it appears soil available N is a major driver of pine tree growth and in the absence of competing vegetation a relatively strong predictor.

The pine stands measured in this study are all <17 years-old and thus in a phase of rapid growth. The youngest stands in this study (aged 12 in 2002) are probably still building a large mass of foliage, indicating that demand for soil N in uptake is probably the greatest. Once the crown has closed, an increasing portion of annual N demand is met through internal cycling of N from old to new foliage. Considering these retranslocation processes it is likely that the age of sampling of these stands provided the opportunity to show a relationship in terms of cause and effect for soil N and growth. Internal foliage and root recycling in older stands likely diminishes the strength of this relationship.

The above relationships for resin extractable N and MAI is an average response across sites. This response may be confounded, however, by a location effect. For example, locations may differ in soil moisture conditions or previous treatment histories. In the current study, the Dawsonville site differs from the Athens and Eatonton locations based on precipitation, soil type, and applied treatments. The other two locations are more homogeneous with respect to these variables. Given these site differences the effect of location or treatment on this relationship have been addressed by using NLMMs to model tree height and test whether soil available N is a significant factor for modeling growth within site and treatment. Based on this modeling approach soil available N on a log scale had a significant effect (α=0.05) for all three fixed parameters of the Chapman-Richards model form (Table 2.7). Including soil available N (M1) as a covariate in the NLMM fixed parameters significantly improved (p-value<0.0001) the model fit based on an approximate F test via the S-plus anova statement. M1 had a homoskedastik residual pattern distribution and presence of significant autocorrelation (Figure 2.5).

The major goal of my modeling effort was to test by comparison via an approximate F-test the significance of soil available N as a covariate to model tree height. To my knowledge no other author has ever used this modeling approach including soil-site variables in a growth and yield data-based model. A similar approach was used by Hall and Bailey (2001) to model tree height in loblolly pine stands of different densities using the number of trees per ha as a covariate. Furthermore, in CAPPS the same approach likely cannot be adopted given all stands have the same planting densities.

Conclusions

The mixed bead resin core technique could detect major differences between fertilized and non-fertilized plots. Continuous fertilization extended during the entire life cycle of each stand caused soil available N to be on average 10- to 100-fold greater in the fertilized plots than in the corresponding plots without any fertilizer treatment. In these simplified pine ecosystems soil available N, as estimated by monthly or biweekly measures of resin extractable N is a useful quantitative covariate to be included in growth and yield models. The significant direct correlation found between mean annual increment and soil available N (R^2=0.7) likely results partly from the age of the considered stands since soil nutrient demand is great and N is allocated primarily to stem biomass during this period.

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Table 2.1. Landscape position, slope, litter bulk density, and litter average height estimated in August 2000 for loblolly pine stands in Athens, Eatonton, and Jackson County, Georgia.

Location[1]	Age[2]	Position[3]	Slope[4]	BD[5]	Ht[6]
	yrs		%	g cm^{-3}	cm
Athens (CAPPS)	8	su	4	0.34	4.5
	8	ms	8	0.22	4.9
	12	ms	12	0.58	4.0
	12	ms	15	0.54	4.4
Eatonton (CAPPS)	6	su	2	0.12	3.5
	11	ms	4	0.35	4.6
	11	ms	13	0.37	4.4
	13	ms	5	0.26	5.9
	13	fs	7	0.30	5.3
	13	ms	4	0.30	5.3
	13	ms	4	0.31	5.4
Eatonton (PMRC)	18	su	0	0.26	6.3
	18	ms	2	0.17	6.3
	18	ms	3	0.21	6.8
	18	ms	5	0.18	6.3
Jackson (PMRC)	14	ms	18	0.32	4.0
	14	ms	15	0.56	5.3
	14	ms	15	0.56	5.3
	14	ms	16	0.40	4.6

[1] CAPPS is the Consortium for Accelerated Pine Productivity Studies, and PMRC is the Plantation Management Research Cooperative both within the Warnell School of Forest Resources at the University of Georgia (Age refers to 2000).
[2] Landscape position: su= summit, ms= mid-slope, fs= foot-slope.
[3] Slope was estimated as the average of two measurements in each plot using a Suunto clinometer.
[4], [5] Two sampling points per plots were collected using a 15-cm diameter by 10-cm high brass core to estimate bulk density.
[6] Litter height.

Table 2.2. Available water holding capacity, total carbon, total nitrogen, and C:N ratio for 0 to 50 cm soil, forest litter, and pine foliage measured in August 2000 for loblolly pine stands in the Athens, Eatonton, and Jackson County sites, Georgia.

Location[1]	Age[2]	AWHC[3]	C[4]	N	C:N	C	N	C:N	C	N	C:N
			Soil			Litter			Foliage		
	yr	cm m^{-1}	%	%		%	%		%	%	
Athens (CAPPS)	8	10.5	1.0	0.05	21	16.4	0.32	51	52.5	1.38	38
	8	15	0.8	0.04	22	33.1	0.65	51	52.1	1.15	45
	12	7.5	0.8	0.04	22	19.2	0.46	42	53.5	1.25	43
	12	15	0.7	0.03	22	18.7	0.46	40	52.3	1.29	41
Eatonton (CAPPS)	6	4.5	0.9	0.05	19	24.2	0.43	57	50.0	1.27	39
	11	19	0.8	0.04	22	26.1	0.57	46	50.4	1.09	46
	11	3.8	1.5	0.06	27	22.2	0.45	49	48.9	1.17	42
	13	15	0.6	0.03	19	30.8	0.68	45	51.8	1.05	49
	13	6	0.6	0.03	21	31.0	0.69	45	52.7	1.01	52
	13	3.8	0.7	0.04	18	28.4	0.64	45	51.7	1.32	39
	13	7.5	0.9	0.05	18	26.9	0.64	42	50.6	1.27	40
Eatonton (PMRC)	18	18	0.8	0.05	15	22.0	0.58	38	49.6	1.2	42
	18	18.8	0.8	0.04	20	31.2	0.77	41	50.6	1.16	44
	18	15	0.6	0.03	19	23.0	0.55	42	51.4	1.13	46
	18	16.5	0.7	0.04	16	41.6	0.96	43	51.3	1.18	43
Jackson (PMRC)	14	15	0.8	0.04	18	24.6	0.62	40	54.6	1.22	45
	14	15	0.8	0.04	17	25.4	0.55	47	52.3	1.07	49
	14	15	0.8	0.04	17	25.4	0.55	47	52.7	1.16	46
	14	15	0.9	0.05	19	35.3	0.78	45	52.4	1.19	44

[1] CAPPS is the Consortium for Accelerated Pine Productivity Studies, and PMRC is the Plantation Management Research Cooperative both within the Warnell School of Forest Resources at the University of Georgia.
[2]Age is relative to measurements made in the year 2000.
[3] Available water holding capacity estimated for 0-1 m depth.
[4] Soil, litter, and foliar C and N were measured after pulverization with a dry combustion technique using a CE Elantech CNS analyzer.

Table 2.3. Correlation matrix for soil and stand variables measured during August 2000 and stand mean annual increment for loblolly pine stands in the Athens, Eatonton, and Jackson County sites, Georgia (N=19).

	Foliage C:N[1]	Litter C:N[2]	Soil C:N[3]	AWHC[4]	SLOPE[5]
MAI	0.18†	-0.87	-0.51	0.62	-0.13
	0.47‡	<.0001	0.02	0.005	0.60
Foliage C:N		-0.11	0.11	0.39	0.07
		0.65	0.64	0.09	0.79
Litter C:N			0.34	-0.44	0.06
			0.15	0.056	0.81
Soil C:N				-0.28	0.30
				0.24	0.21
AWHC					-0.13
					0.59

[1] Foliage C:N ratio.
[2] Litter C:N ratio.
[3] Soil C:N ratio.
[4] Available water holding capacity to 1m depth.
[5] Percent slope.
† Linear Pearson Correlation coefficient (r) between the considered variables.
‡ Corresponding p-value.

Table 2.4. Soil extractable P, total N, and available N estimated through a 30-day laboratory incubation on 0-10 cm depth samples collected in June 2001 from loblolly pine stands in Athens, Eatonton, and Jackson County, Georgia (N=19).

Location	Age	N	Av-N[1]	SE_N[1]	Ext-P[2]
	yr	ug g^{-1}			
Athens (CAPPS)	8	815	10.0	0.2	0.6
	8	625	0.7	4.5	0.5
	11	729	20.8	2.9	0.6
	11	627	12.5	4.8	0.8
Eatonton (CAPPS)	5	520	8.3	0.9	0.5
	6	754	18.7	12.2	0.9
	10	748	11.2	6.4	0.6
	10	364	1.8	1.7	0.6
	12	656	19.1	1.7	0.5
	12	845	16.5	2.2	0.5
	12	611	13.6	5.3	0.4
	12	716	3.4	0.6	0.4
Eatonton (PMRC)	18	923	19.1	3.3	3.5
	18	732	1.8	5.1	6.8
	18	770	14.9	8.6	10.2
	18	878	9.8	6.3	7.3
Jackson (PMRC)	12	1107	0.7	3.9	0.6
	12	970	14.6	1.4	0.4
	12	950	11.1	0.6	0.6
	12	901	16.7	0.8	0.4

[1] Av N is mineralized N after a 30 day-incubation at 27 ˚C. Two laboratory replicates per sample were averaged to estimated mean standard error.
[2] Extractable P was determined colorimetrically on a Alpkem after extraction with double acid.

Table 2.5. Soil potentially mineralizable N (N_0) through a 158-day incubation was estimated using samples collected at 0-15 cm depth during June 2001 from loblolly pine stands in Athens (A), Eatonton (E), or Jackson County (J), Georgia (N=19). Only plots that received the herbicide treatment were used for this incubation and are represented in rows grouped by site. The corresponding values for each day are the sum 0.01 M CaCl2 extracted NO3-N plus NH4-N.

		Days since incubation start						Estimated[2]	
Site	Plot	7	30	57	105	158	N_t[1]	$\hat{N}_0$	$\hat{k}$
		$\mu g\ g^{-1}$					ug g^{-1}	ug g^{-1}	d^{-1}
Athens (CAPPS)	1	3.7	6.8	4.4	1.8	1.9	18.6	19.9	0.021
	2	3.6	4.6	6.7	1.5	2.3	18.7	19.3	0.021
	3	3.1	5.5	11.0	1.9	2.4	23.8	24.8	0.020
	4	4.2	5.3	9.0	2.6	0.6	21.7	24.8	0.020
Eatonton (CAPPS) Monitor	1	1.9	6.2	3.6	3.7	1.0	16.2	18.9	0.015
	2	3.8	6.3	4.7	5.0	2.7	22.3	25.6	0.014
	3	1.2	2.1	2.2	1.6	0.5	7.5	8.6	0.017
	4	0.8	4.7	2.2	2.2	1.6	11.5	12.6	0.016
Eatoton (CAPPS) Powerline	1	2.5	6.5	5.7	4.6	2.9	22.2	24.0	0.016
	2	1.4	5.8	5.6	5.4	2.2	20.4	21.7	0.016
	3	2.1	8.1	7.4	5.6	9.9	33.1	32.8	0.014
	4	1.5	4.0	7.8	3.8	2.5	19.6	20.6	0.016
Eatoton (PMRC)	1	4.0	6.0	5.6	3.5	3.5	22.4	22.9	0.019
	2	0.9	4.3	6.1	3.3	3.6	18.1	17.0	0.020
	3	1.4	4.8	2.7	2.8	2.6	14.3	13.9	0.021
	4	4.5	6.7	6.8	4.0	2.1	24.1	25.9	0.019

Table 2.5 (Continued).

Jackson (PMRC)	1	4.1	5.6	9.5	2.2	2.7	24.1	26.4	0.017
	2	1.2	6.1	7.8	3.9	3.2	22.2	22.8	0.018
	3	2.7	7.4	6.7	4.3	2.2	23.2	25.2	0.017
	4	1.5	13.3	8.1	6.0	3.9	32.7	36.1	0.015

[1] Cumulative amount of N extracted over the 158 day incubation.
[2], Corresponding estimated parameters using a multilevel nonlinear mixed effects model. The one-pool model form based on first order kinetics was used to fit the corresponding cumulative data.

Table 2.6. Correlation matrix for mean annual increment, mineralizable nitrogen by 30-day-incubation, potentially mineralizable N (N_0), and extractable P estimated in 2001 for loblolly pine stands in the Athens, Eatonton, and Dawsonville sites, GA.

	Mineralizable N[1]	N_0[2]	Extractable P[3]
MAI[1]	0.17†	0.44	0.39
	0.476‡	0.0618	0.0901
	20§	19	20
Mineralizable N		0.03	-0.02
		0.9161	0.9232
		19	20
N_0			-0.01
			0.9807
			19

[1] Mean Annual Increment.
[2] Soil mineralizable N estimated through a 30-day incubation.
[3] Potentially mineralizable nitrogen.
† Correlation coefficient r between the considered variables.
‡ Corresponding p-value.
§ Sample size.

Table 2.7. Results of multilevel nonlinear mixed effects model (M1) using the Chapman-Richards equation similarly to Hall and Bailey (2001) to test the significance of soil available N (log scale) as a covariate within location and treatment for modeling pine tree height under simplified conditions of complete competition control. All analyses were based on N=4551. The Chapman-Richards equation parameters are expressed as a simple linear function of soil available N (log scale, continuous), fertilization treatment, and location (dummy variables, 0 or 1).

Model fixed parameters		Value	p-value
Upper asymptote (Intercept)	β_1	16.7	<.0001
Upper asymptote (Z1)	β_2	-2.1	<.0001
Upper asymptote (Z2)	β_3	3.9	<.0001
Upper asymptote (TRT)	β_4	2.8	<.0001
Upper asymptote (LOGN)	β_5	1.9	0.0002
Rate.(Intercept)	β_6	0.196	<.0001
Rate (Z1)	β_7	0.040	<.0001
Rate (Z2)	β_8	-0.044	<.0001
Rate (TRT)	β_9	-0.026	0.0006
Rate (LOGN)	β_{10}	-0.024	0.0002
Shape (Intercept)	β_{11}	2.282	<.0001
Shape (Z1)	β_{12}	0.249	<.0001
Shape (Z2)	β_{13}	-0.016	0.772
Shape (TRT)	β_{14}	-0.193	0.0093
Shape (LOGN)	β_{15}	-0.183	0.004

Figure legend

Figure 2.1. Mean annual increment versus potentially mineralizable N (N_0) estimated in 2001 for plots near Eatonton, GA that received the herbicide treatment (H).

Figure 2.2. Temporal resin extracted N (ug $N \cdot g^{-1}$ resin) for all considered plots (H plus HF) and H only estimated using the mixed bead resin core technique. Six polyvinyl chloride (PVC) tubes per plot were placed monthly to 10 cm depth during 2002 and 2003 soil depth with a mixed-bead resin bag at the bottom of each tube to capture available N in the leaching soil solution.

Figure 2.3. Mean annual increment versus soil available N on a log-scale across locations and by collection date (January, April, and July). In 2001 plots aged 13, 12-14, and 13-15 year old in Athens , Eatonton, and Dawsonville, respectively.

Figure 2.4. Mean annual increment versus cumulative over 17 collection dates soil available N on a log scale across location. In 2001 plots aged 13, 12-14, and 13-15 year old in Athens , Eatonton, and Dawsonville, respectively.

Figure 2.5. Diagnostic plots of a nonlinear mixed effects model (M1), using the Chapman-Richards three parameter equation. The residual plot shows a homoskedastic pattern, while the autocorrelation function plot shows presence of significant autocorrelation at lag 1, 5, 6, 8, and 9. There are 12 possible lags since sample trees were measured yearly until age 13 at most.

Figure 2.1. Mean annual increment versus potentially mineralizable N (N_0) estimated in 2001 for only plots near Eatonton that received the herbicide treatment (H).

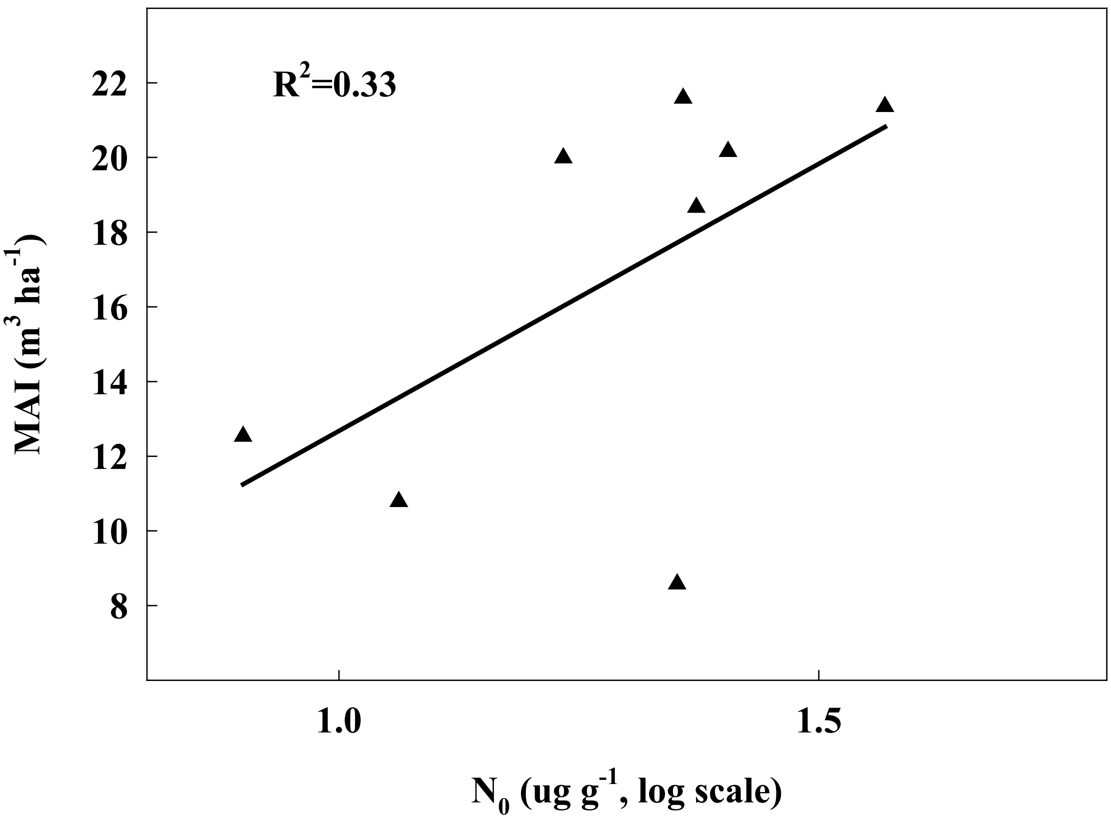


Figure 2.2. Temporal resin-extractable N (ug N·g^{-1} resin) for all considered plots (H plus HF) and H. Six polyvinyl chloride (PVC) tubes per plot were placed monthly to 10 cm depth during 2002 and 2003 soil depth with a mixed-bead resin bag at the bottom of each tube to capture available N in the leaching soil solution.

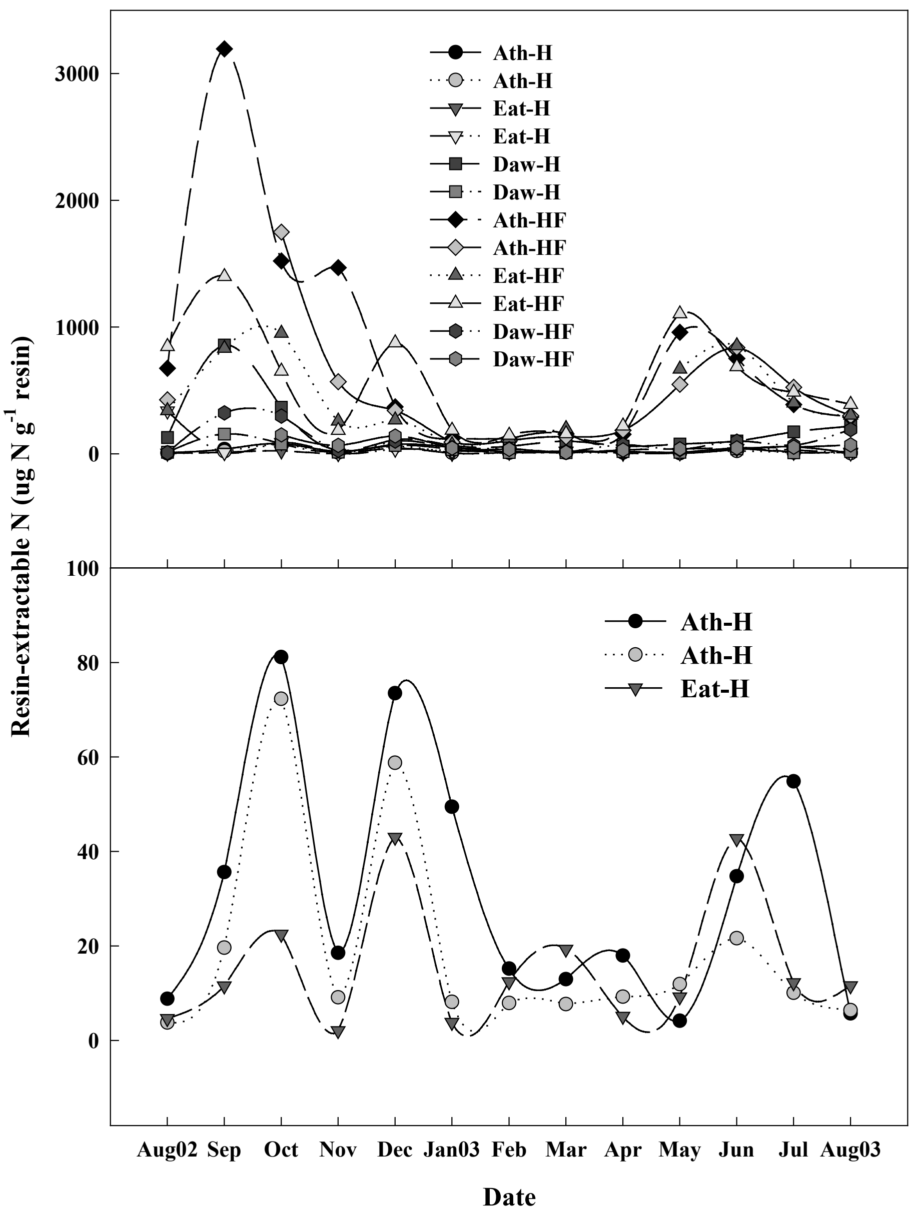


Figure 2.3. Mean annual increment versus resin-extractable N on a log-scale across locations and by collection date (January, April, and July). In 2001 plots aged 13, 12-14, and 13-15 year old in the Athens, Eatonton, and Dawsonville sites, respectively.

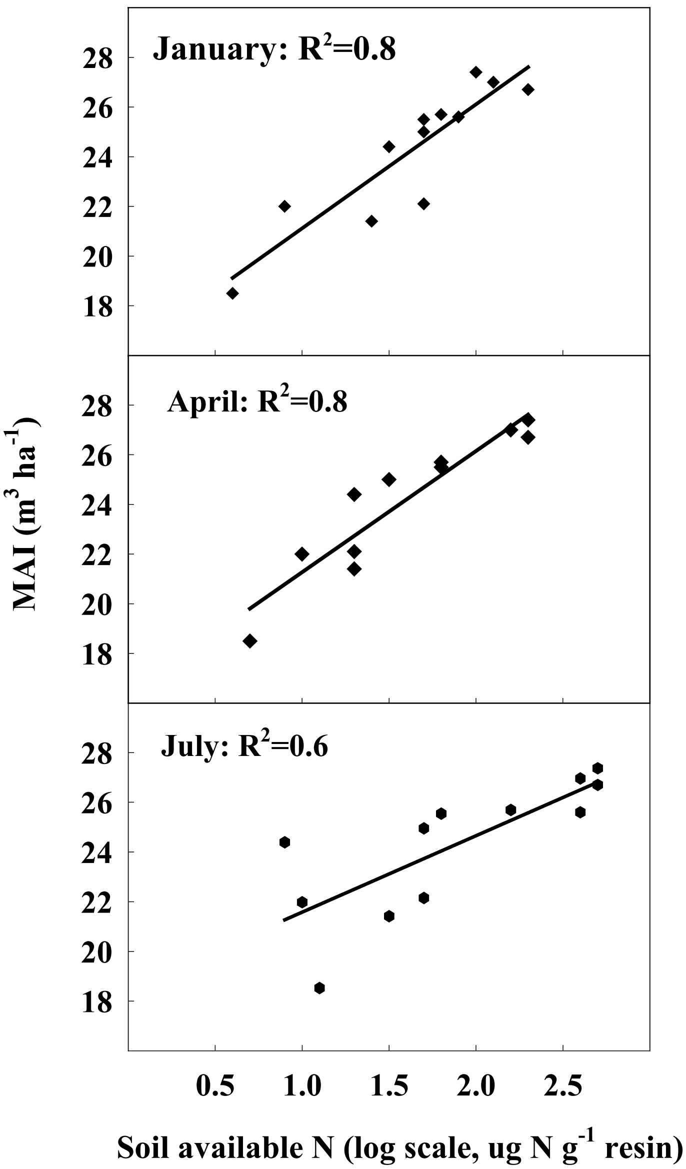


Figure 2.4. Mean annual increment versus cumulative over 17 collection dates resin-extractable N on a log scale across location. In 2001 plots aged 13, 12-14, and 13-15 year old in the Athens, Eatonton, and Dawsonville sites, respectively.

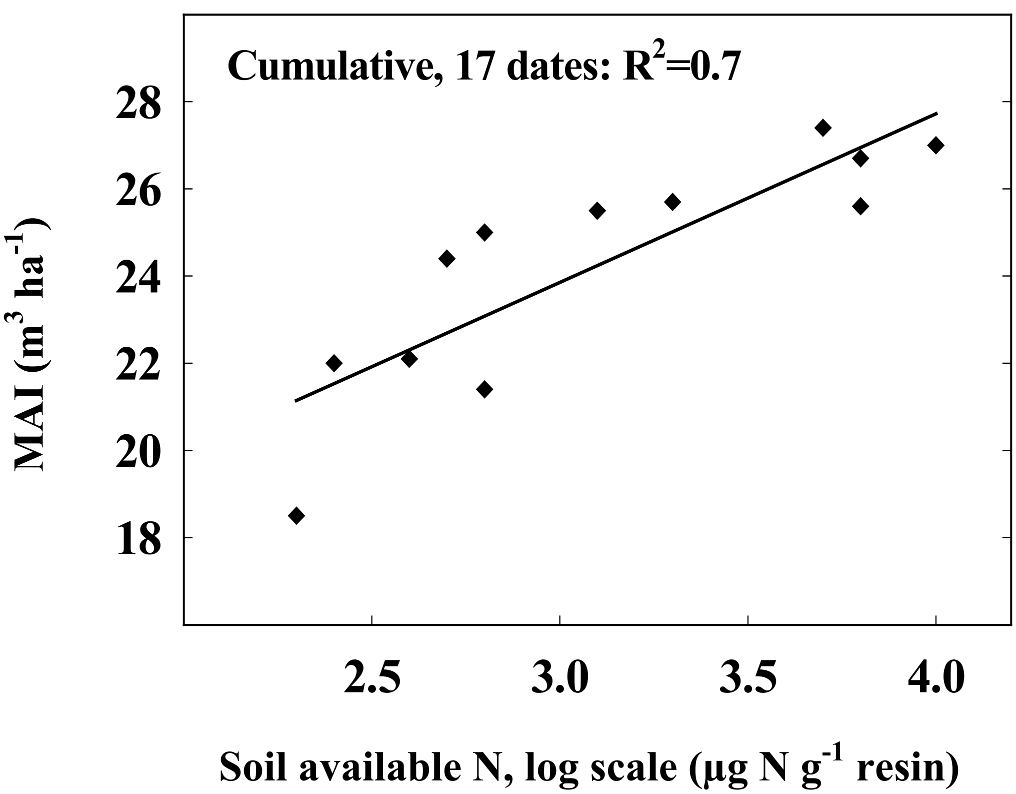
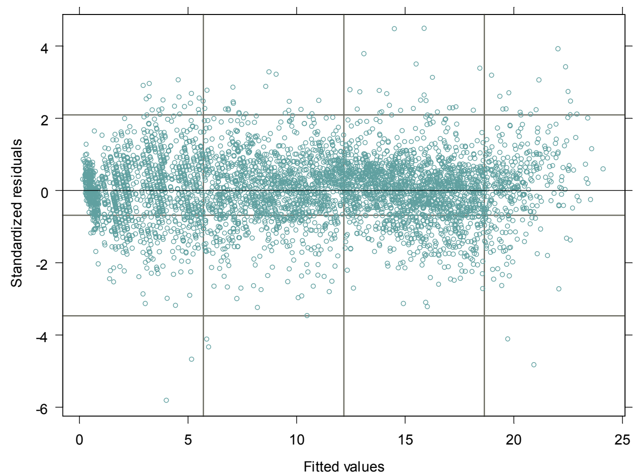
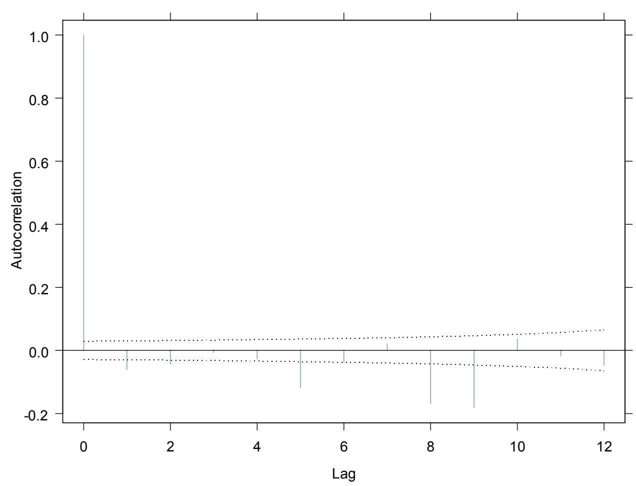


Figure 2.5. Diagnostic plots of a nonlinear mixed effects model (M1), using the Chapman-Richards three parameter equation. The residual plot shows a homoskedastic pattern, while the autocorrelation function plot shows presence of significant autocorrelation at lag 1, 2, 5, 6, 8, and 9. There are 12 possible lags since sample trees were measured yearly until age 13 at most.





CHAPTER 3

SOIL NUTRIENT CHANGES IN INTENSIVELY MANAGED LOBLOLLY PINE (PINUS TAEDA L.) PLANTATIONS[2]

[2] Sartori, F., Markewitz D., and Borders E. B. To be submitted to Canadian Journal of Forest Research.

Abstract

The impact of herbicide (H) and fertilizer (F) treatments in intensively managed loblolly pine (*Pinus taeda* L.) plantations of the Piedmont region of Georgia has been analyzed in stands of varying ages (6 through 14) to explain the relevant growth responses corresponding to these treatments. Soil total C, N, P, extractable P, pH_w, pH_s, and exchangeable chemistry (acidity, Ca, Mg, and K) were measured in 66 stands in three different locations comparing H, F, and their combination HF to the control plot: during December 1999 and January 2000 in Eatonton (n=40), in July and August 2001 in Athens (n=12), and in March 2003 in Dawsonville (n=14). In addition, twelve plots (two H and two HF at each location) were repeatedly measured on 17 dates, with 10 monthly (starting July 2002 through May 2003) and 7 bi-weekly (June -September 2003) collections using the mixed bead resin core technique to estimate soil N availability. On the same dates wet-only deposition samples were collected and analyzed for total mineral N. At all three study locations the herbicide treatment reduced soil C and ECEC while, overall, the fertilizer treatment did not determine any significant soil C increase. The largest C reduction due to herbicide treatment was ~7 Mg C ha^{-1} between the F and HF in Dawsonville. The HF plots had much higher resin extractable N than the corresponding H plots in all months of the year. At a plot level, HF values ranged from ~9 to ~3195 µg N g^{-1} resin while H values ranged from ~4 to ~858 µg N g^{-1} resin. The corresponding annual cumulative resin extractable N ranged from ~13 kg N ha^{-1} in the H to 372 kg N ha^{-1} in the HF. Wet-only inorganic N deposition was similar across sites being ~12.6 kg ha^{-1} for Dawsonville, 9.9 kg ha^{-1} for Athens, and 11.4 kg ha^{-1} for Eatonton. Nitrogen and P were the two nutrients associated with higher growth rates for the stands in Athens and

Eatonton on relatively nutrient poor Ultisols. Based on the experimental design of this study it was not possible to define whether N or P was the major driver of growth in these stands. Changes in soil nutrients were closely related to reductions in soil C that were observed with herbicide treatments. The fertilization treatment did not have a corresponding consistent increasing effect on soil C content suggesting that such treatments would likely not buffer and counter the negative effects of the herbicide treatment on soil C. Fertilization and herbicide treatments did simulate C sequestration but mainly in the above ground biomass.

Introduction

The availability of the essential plant nutrients in loblolly pine production systems is key to understanding both the short and long-term productivity of these ecosystems (Wells, 1977; Wells et al., 1986; Jokela et al., 2004). Understanding the impacts of fertilizer applications of macro- and micro-nutrients on soil nutrient availabilities to enhance productivity and to compensate for nutrient losses due to timber removal is essential for sustainable production of pine plantations (Fox, 2000). Similarly the impacts of herbicide use to increase current stand productivity may have impacts on long-term soil productivity that have to be better understood (Echeverria et al., 2004). Finally, at present, there is an increasing interest in quantifying the effects of fertilizer and herbicide use in forest management in relation to soil C retention and using forest ecosystems as an offset for CO_2 emission (Huntington, 1995; Lal, 2004).

Nitrogen and P are typically considered the limiting nutrients in southern pine plantations (Binkley and Hart, 1989). It has been clearly demonstrated in different physiographic regions of the Southeast that loblolly pine growth responds primarily to N and P fertilization (Vose and Allen, 1988; Maimone et al., 1991). Vose and Allen (1988), for example, found that in loblolly pine stands in the Lower Coastal Plain of North Carolina leaf area index, and thus growth, was positively correlated to increasing levels of N fertilization. These results are in agreement with the findings of Polglase et al. (1992) for loblolly pine stands of northern Florida. Phosphorus, in some cases, has been cited as the primary limiting element particularly in Lower Coastal Plain sites (Morris and Campbell, 1991). Other studies in the Coastal Plain, however, have found no significant response to P fertilization highlighting important variations in this region (Vose and Allen, 1988). In the Piedmont, P alone has rarely been found limiting (Wells at al., 1986).

Borders and Bailey (2001) have described the relevant growth responses that can be achieved with continuous fertilization and herbicide treatment for stands in the Piedmont and Coastal Plain of Georgia on nutrient limited sites. Extremely high growth responses, in particular, to the combined fertilization plus herbicide treatment have been reported (Borders and Bailey 2001; Borders et al. 2004). For example, the combination of such treatments in the lower Coastal Plain of Georgia, as reported by the same authors, has more than doubled the stem biomass production by age 15 compared to the corresponding control treatment.

These relevant above ground biomass growth responses may have important implications for soil C sequestration as well as soil organic matter processes related to nutrient cation exchange capacity or N mineralization. Typically, the sequestered C in these regrowing forests is allocated mainly to the above ground biomass rather than to the soil organic pools (Richter et al., 1995; Shan et. al., 2001; Markewitz et al., 2002). None-the-less effects of these growth enhancing herbicide and fertilizer treatments on soil C have been observed. A number of studies suggest that competition control through herbicide treatments reduce soil C and N content in forest ecosystems (Aust and Lea, 1991; Polglase et al., 1992; Carlyle, 1993; Munson et al., 1993). For example, Aust and Lea (1991) reported significant decreases (29%) in soil Carbon content after two years of post-harvest competition control in a study examining ecosystem recovery in a water tupelo-bald cypress wetland in southwest Alabama. The recent work of Echeverria et al. (2004) also demonstrates about a 20% decline in soil C content with herbicide treatments in loblolly pine stands. Conversely, soil C response to fertilization has generally been positive (Johnson and Curtis, 2001) although decreases have also been observed (Shan et al. 2001).

In addition, I also address specific N fluxes that are believed to be major drivers of growth in southeastern pine ecosystems. The pine stands in this study are in the early stages of their growth curve and thus nutrient demand is great. It is during this period of rapid early growth that nutrient limitations can be most apparent. The main objectives of this study are: i) define the soil limiting nutrients in these pine ecosystems based on differences among treatments, ii) describe the decadal effects of the long-term herbicide and fertilizer application on soil nutrient status, and iii) evaluate the herbicide and fertilizer effects on soil C retention.

Materials and Methods

Research locations

Three research sites were located in the Piedmont of Georgia and were part of the Consortium for Accelerated Pine Productivity Studies (CAPPS) (Borders and Bailey, 2001). One site was located at Whitehall Forest in Athens (33° 57' N, 83° 19' W), a second at the B.F. Grant Forest near Eatonton (33° 20' N, 83° 23' W), and a third near Dawsonville (34° 21' N, 84° 08' W). The location of these study sites, based on the eco-region classification developed by McNab and Avers (1994), are in the Southern Appalachian Piedmont (SAP), consisting of an intensely metamorphosed, moderately dissected plain formed of thick saprolite, continental sediments, and accreted terrains. Elevation ranges from 100 to 400 m. The potential vegetation is oak-hickory-pine forest and southern mixed forest (Kuchler, 1964). Predominant vegetation form is evergreen forest. The loblolly (*Pinus taeda* L.) -shortleaf pine (*Pinus echinata* Mill.) cover type is common on disturbed areas and usually has an understory component of dogwood (*Cornus sanguinea* L.) and sourwood (*Oxydendrum arboreum* (L.) DC.). The growing season lasts about 205 to 235 days.

The Athens site is characterized by warm to hot summers, with mean July high temperature of 32 ºC and moderately cold but highly variable winters, with mean January low temperature of -0.1 ºC and mean annual precipitation of about 126 cm (1961 to 1990, Univ. of Georgia, http://climate.engr.uga.edu/info.html). The climate of Eatonton has a mean July high temperature of 32.5 ºC, mean January low temperature of 1.1 ºC, and mean annual precipitation is about 124 cm (1911 to 2003, Univ. of Georgia, http://www.griffin.peachnet.edu/bae/). Similarly, Dawsonville has mean July high temperature of 30.7 ºC, mean January low temperature of –0.1 ºC, and mean annual precipitation of about 140 cm (1911 to 2003, Univ. of Georgia, http://www.griffin.peachnet.edu/bae/). In all three locations precipitation has a maximum in early March and minimum in October.

Soils in Athens and Eatonton had an A or Ap horizon that ranged from sandy to sandy loam in texture and was absent in the most eroded sites. The most common soil series (United States Department of Agriculture-Soil Conservation Service, 1968) at these sites are Cecil (fine, kaolinitic, thermic Typic Kanhapludults), Pacolet (fine, kaolinitic, thermic Typic Kanhapludults), Appling (fine, kaolinitic, thermic Typic Kanhapludults), and Davidson (fine, kaolinitic, thermic Rhodic Kandiudults) and reflect in some cases severe erosion related to the cotton farming era. The Dawsonville site is on the floodplain of the Etowah River and as such has younger soils of the Congaree series (fine-loamy, mixed, active, nonacid, thermic Typic Udifluvent, United States Department of Agriculture-Soil Conservation Service, 1968). Slopes ranged between 6-13 % at Whitehall, 3-11 % at the Eatonton site, and are minimal for the Dawsonville floodplain site, < 2 %.

For all studies tree volume data were available from the inventories conducted by CAPPS at the D.B. Warnell School of Forest Resources (Borders and Bailey, 2001). Tree biomass was calculated based on annual individual tree repeated measures of tree diameter and height. Each tree was identified by a unique tag number and repeatedly measured since stand establishment. The corresponding volume was computed using an allometric equation (Harrison and Borders, 1996). Plot level estimates were computed by adding up individual tree volumes and expressed on a per hectare basis.

In CAPPS, four treatments were applied to randomly chosen plots (Borders et. al, 2004): a Control (C), Herbicide (H), Fertilizer (F), and Herbicide plus Fertilizer (HF) treatment. The herbicide consisted of a broadcast of sulfomethuron methyl during early spring of the first three growing seasons following planting and subsequent application of glyphosphate as needed in mid-summer each year thereafter. The fertilizer treatment consisted of 280 $kg{\cdot}ha^{-1}$ DAP plus 112 $kg{\cdot}ha^{-1}$ KCl in the spring and 56 $kg{\cdot}ha^{-1}$ of NH_4NO_3 mid-summer during the first two growing season. In subsequent growing seasons: 168 $kg{\cdot}ha^{-1}$ NH_4NO_3 was applied in early to mid-spring. At age eleven 336 $kg{\cdot}ha^{-1}$ NH_4NO_3 + 140 $kg{\cdot}ha^{-1}$ triple super phosphate was applied. At age twelve 560 $kg{\cdot}ha^{-1}$ Super Rainbow® with micronutrients +168 kg ha^{-1} NH_4NO_3 was applied in early spring. From age thirteen forward 336 $kg{\cdot}ha^{-1}$ NH_4NO_3 was applied in early spring. In Dawsonville the fertilization treatment was interrupted in 1996 in all plots because strong competition from understory vegetation had compromised seedling growth in some of the F plots.

Sets of these four treatments were replicated in time and space in experimental blocks. In total, N=66 stands were sampled in December 1999 and January 2000 in Eatonton (n=40, 10 blocks), in July and August 2001 in Athens (n=12, 2 complete and 2 incomplete blocks with only H and C), and in March 2003 in Dawsonville (n=14, 4 blocks). In Dawsonville, two F plots were not sampled because seedlings were suppressed by the vigorous understory vegetation competition. In Eatonton four blocks were planted in 1988, four in 1990 and two in 1995. In Athens two blocks were planted in 1989 and two in 1993. In Dawsonville two blocks were planted in 1987 and two in 1989.

Soil samples

Mineral soil samples were collected from each stand using a stainless steel probe (diameter: 2.5 cm; length 60 cm) with removable internal liners and a slide hammer, after removal of the O horizon. Fourteen soil cores in each stand were divided into three different depth increments from the surface: 0-10, 10-30, and 30-50 cm and composited by depth within a plot. Samples for physical and chemical analysis were returned to the laboratory, air dried, and passed through a 2-mm sieve prior to analysis. Soil total C and total N were measured after pulverization with a dry combustion technique using a CE Elantech (NJ) CNS analyzer (Bremmer and Mulvaney, 1982). Total P was measured on an Alpkem auto-analyzer (OI Analytical, College Station, TX) using the Murphy-Riley chemistry after digestion with H_2SO_4-H_2O_2 and H_2SeO_2 as a catalyst (Kuo 1996). Extractable P was estimated via a double-acid (H_2SO_4-HCl) extraction. NO_3 and NH_4 were measured on an Alpkem auto-analyzer (OI Analytical, College Station, TX) using the cadmium reduction method and the indophenol blue method, respectively, after extraction in 2M KCl (Mulvaney, 1996). Potassium, Ca, and Mg were measured by atomic absorption in the presence of $LaCl_3$ after extraction with double acid (Sumner and Miller, 1996). Soil pH in water and 0.01 M $CaCl_2$ was determined in a 1:2 weight to volume ratio (Thomas, 1996). The effective cation exchange capacity (ECEC) was determined by the sum of cations method (Sumner and Miller, 1996).

For bulk density collections, a 7.5-cm diameter by 7.5-cm high brass slide-hammer driven core sampler was used. After collection samples were returned to the laboratory, dried at

105°C for 48 hours, and weighed. In the Eatonton sites bulk density was measured within each plot for the 0 to 7.5 cm depth (N=40). In the 10-30, and 30-50 cm depths bulk density values were estimated for the blocks (N=10) by collecting cores from 15 to 22.5 cm and from 35 to 42.5 cm. In the Athens site bulk density was measured in the 0 to 7.5 cm horizon of the C and H plots (N=4). Soil bulk density was not measured in the Dawsonville site so a bulk density of 1.4 g cm^{-3} was assumed based on the United States Department of Agriculture-Soil Conservation Service for the Congaree soil series of Newton and Rockadale County, GA (United States Department of Agriculture-Soil Conservation Service, 1999).

Temporal Soil N Measurements

Twelve plots (two H and two HF at each location) were repeatedly sampled 17 times, with 10 monthly (starting July 2002 through May 2003) and 7 bi-weekly (June -September 2003) collections. Collections were made using six polyvinyl chloride tubes with a 3.5 cm internal diameter following approximately a regular grid. Tubes were driven to a 12 cm soil depth, removed with soil intact, a small portion of soil at the base of the tube was removed and replaced with a mixed-bead resin bag (Binkley and Matson, 1983) to capture available N in the soil solution. Two sets of ~70 bags were available for this study: at each collection date one set of bags would be collected from the field and replaced with the freshly recharged set of bags. The 4 × 4 cm resin bags were constructed using Nitex ® 03-80/37 nylon material and sewed using nylon thread. Four grams of 20-50 mesh ion exchange resin from BIO-RAD (2 g of AG® 1-X8 chloride form and 2 g of AG® 50W-X8 hydrogen form) was placed within each bag. Each resin bag collected from the field was placed in separate centrifuge tubes with 40 ml of 1 M HCl solution and recharged by shaking it on a reciprocating shaker at 200 RPM for at least 24 hours. NO_3-N and NH_4–N were measured on an Alpkem auto-analyzer (OI Analytical, College Station, TX) and on a Technicon segmented flow analyzer (Nicholson, 1984; U.S. Environmental Protection Agency, 1979), using the cadmium reduction column and the indophenol blue method for NO_3-N and NH_4–N, respectively, after a 1 M KCl extraction using a reciprocating shaker. Because the resins were initially recharged with HCl the KCl extracts had low pH (i.e.~1-2) that could cause interference for the analysis of NH_4-N, therefore, samples were neutralized by addition of a drop of a 5 N NaOH solution prior to analysis.

During each collection the soil within the PVC tube was collected and composited by plot in a plastic bag, returned to the laboratory, air dried, and passed through a 2-mm sieve for further analysis. The mixed bead resin core technique as used in this study demonstrated some of the limitations discussed by Hanselman et al. (2004). During many collection dates standing water was found on the top of the soil core internal to the PVC tubes indicating that water dynamics did not reflect that in the field and such prolonged water saturated conditions may induce denitrification. Such conditions may produce biased estimates of soil N availability (Hanselman et al., 2004). Nonetheless, the major objective of this study was a comparison among treatments within the same site. For this reason, other limitations as discussed by Adams et al. (1989) were not considered to be confounding factors.

Atmospheric N deposition

At each location one automated solar powered dry fall-wet fall collector (Aerochem Metrics, Kissimee, FL) was placed in an opening to collect wet-fall only precipitation samples. Intervals between collection dates were the same as those of the resin bag collections. The rain samples were taken to the laboratory and a subsample was stored frozen in a scintillation vial until analysis. Nitrate and ammonia were measured on a Dionex DX 500 Ion Chromatograph (Dionex, Sunnyvale CA).

Statistical Analyses

Statistical analyses on the 2001 and 2003 single-time measured soil variables (i.e. exchangeable chemistry, soil total C, N, etc.) for the comparison of all treatments were most concerned with soil differences among plot treatments by depth. The experimental design for these samples followed a completely randomized block design. I conducted a three-way MANOVA test (Wilk's Lambda) to test for an overall treatment effect using the SAS GLM procedure (SAS Institute Inc., 1990). High collinearity between the soil variables can cause the determinant of the within treatments sums of squares and cross product matrix to be zero, making Wilk's test inapplicable (Johnson and Wichern, 1998). To avoid this problem and be able to carry out a uniform comparison across sites based on the measured variables, I included in my model total C, N, extractable P, exchangeable acidity, and effective cation exchange capacity (ECEC).

If treatment was significant ($p < 0.05$) in the MANOVA, I conducted an F-test (univariate ANOVAs) for each individual variable by depth testing the effect of treatment, age, and block nested within age. If the univariate analysis was significant ($p < 0.05$), I then conducted Fisher's protected LSD ($p < 0.05$) to test the following orthogonal contrasts: 1) herbicide (H and HF) versus non-herbicide (C and F), 2) H vs. HF, and 3) C vs. F. In this study it was of interest to address specific questions in comparing H, HF, F, and C plots with respect to the different soil nutrients using specific orthogonal contrasts for pre-planned comparisons as suggested by Dean and Voss (1999). The questions of interest addressed using the selected orthogonal contrasts were: 1) are the plots that received the herbicide treatment significantly different from the non-herbicide treated plots?, 2) In the presence of herbicide application does fertilization have a significant effect?, and 3) In the absence of herbicide application does fertilization have a significant effect?

For the repeated soil N measurements the effect of treatment, block, collection time, and the interaction between treatment and time was tested using SAS PROC MIXED (Wolfinger and Chang, 1995). I used this SAS procedure considering a split-plot design for repeated measures having treatment, block, time, and the interaction of treatment x time as predictors of the log transformed available N response variable. Various different variance-covariance structures for the repeated measures through time were considered, but the split-plot in each case was found to be the most appropriate and therefore selected for analysis. The log was used as a variance stabilizing transformation because for many collection dates the HF had values 100-fold higher than the corresponding H treatment. The analysis was carried out by location considering that the study plots in Dawsonville had been treated differently over the years.

Results

Treatment effects on soil properties based on the MANOVA analysis are reported in Table 3.1. For the 0-10 and 30-50 cm depth in Athens and 0-10 cm in Eatonton there was an overall significant treatment effect (α=0.05). To the contrary in Dawsonville treatment had no overall significant effect at any depth. For descriptive purposes of comparison among sites results for the different variables have also been reported as average values by depth and treatment for each location (Tables 3.2 and 3.3).

C, N, and P

At the Athens site (Table 3.2) there was no statistically significant difference at any depth for total C concentrations between herbicide and non-herbicide plots, although herbicide treated plots always had lower concentrations than the other treatments. There was a general decreasing pattern in C concentration with depth having a maximum concentration of 2 % in the surface and a minimum of 0.3 % at 30 to 50 cm. Total N showed a similar decreasing pattern with depth ranging from a maximum of 0.11 % to a minimum of 0.01%. Total P did not demonstrate a similar pattern with depth. Total P was low, however, in the 0-10 cm depth for the Control, ~485 $ug \cdot g^{-1}$, but was not significantly different compared to the other treatments. Conversely, in the 0-10 cm depth, extractable P was significantly lower (α=0.05) in the Control, 1.7 $ug \cdot g^{-1}$, compared to the F treatment, 8.6 $ug \cdot g^{-1}$, and tenfold lower (α=0.1) in the H, 1.4 $ug \cdot g^{-1}$, compared to the HF, 14 $ug \cdot g^{-1}$. Fertilized plots retained higher extractable P concentrations in the lower depths relative to the other treatments although these differences were not statistically significant.

At the Eatonton site (Table 3.2), there was a decreasing pattern with depth for total C concentration with values ranging from 1.53 to 0.29 %. In the 0-10 cm depth there were no significant treatment effects on total C but the H and HF plots had the lowest values. In the 10-30 cm depth the same H and HF plots did have a significantly lower total C concentration relative to the corresponding plots without the herbicide treatment. There were no significant differences in total C at 30-50 cm. Total N had a similar decreasing pattern with depth declining from 0.08 to 0.02 % N. There were no significant differences within treatment. Total P did not decline regularly with depth but in 0-10 cm depth was lower (not significantly) in the Control, ~339 $ug \cdot g^{-1}$, compared to all the other treatments. Extractable P in 0-10 cm depth was significantly lower in the Control, ~1.2 $ug \cdot g^{-1}$, compared to the F, ~4.8 $ug \cdot g^{-1}$, and in the H, ~0.8 $ug \cdot g^{-1}$, compared to the HF, ~4 $ug \cdot g^{-1}$. The same pattern of elevated extractable P in the F and

HF was maintained at lower depth, although again no statistically significant differences were apparent.

Although the overall MANOVA test for Dawsonville was not significant the univariate ANOVAS were carried out considering this initial overall result. Total C concentration again declined with depth from a high of 2.1 % to a low of 0.7 % (Table 3.2). On average total C was higher in plots that did not receive any herbicide treatments but such differences were again not statistically significant. Total N had a general decreasing pattern ranging from a high of 0.13% at the surface to a low of 0.04 % at 30-50 cm depth but no significant effects of treatment on concentration. Total P was not measured in these plots. Extractable P instead, based on the univariate ANOVAS by depth, was significantly lower (α=0.05) at 0-10 cm in the Control (34 $\mu g\ g^{-1}$) than in the F treatment (87 $\mu g\ g^{-1}$). Differences in extractable P between these treatments were still significant at 10-30 cm but were non-significant at 30-50 cm.

pH, Cations, and Exchange chemistry

In Athens, the fertilization treatment significantly reduced soil pH_w and pH_s at 0-10 cm depth both in the presence and in the absence of the herbicide treatment (Table 3.3). At deeper depths this effect was still significant for pH_w in the F treatment relative to the control. Exchangeable Ca concentration at 0-10 cm depth was higher (not significantly) in the F (1.9 $cmol_c\ kg^{-1}$) relative to the C treatment (1.4 $cmol_c\ kg^{-1}$) and maintained a similar but non-significant pattern at lower depths.

Exchangeable K concentration was relatively constant through the profile and the only significant difference was between C and F at 30-50 cm depth. Exchangeable acidity at 0-10 cm was significantly higher (α=0.05) in the HF, 1.3 $cmol_c\ kg^{-1}$, compared to the H, 0.5 $cmol_c\ kg^{-1}$, and in the F, 1.2 $cmol_c\ kg^{-1}$, compared to C, 0.3 $cmol_c\ kg^{-1}$. ECEC in both 0 to 10 cm and 10 to 30 cm was significantly lower (α=0.05) in plots that received the herbicide treatment compared to non-herbicided plots, and was significantly lower (α=0.1) in the C, 2.6 $cmol_c\ kg^{-1}$, compared to the F treatment, 4.1 $cmol_c\ kg^{-1}$, in the 30 to 50 cm depth.

In the Eatonton location pH_w at all three depths (α=0.05) was significantly higher in the C compared to the F treatment and in the H compared to the HF treatment (Table 3.3). Similarly, at 0-10 cm depth, pH_s was significantly lower (α=0.05) for the herbicide treated plots compared to non-herbicide treated plots, as well as being significantly lower in the F, 4.4, compared to the Control, 4.8, and the HF, 4.2, compared to the H, 4.7. At 10-30 cm only F was significantly lower (α=0.1) than the Control. Exchangeable Ca did not show any significant changes due to treatments. Exchangeable Mg at 0-10 cm depth was significantly lower (α=0.05) for plots that received the herbicide treatment and significantly lower (α=0.1) in the HF compared to the H treatment. Exchangeable K was significantly lower (α=0.05) in the 0 to10 cm depth for plots that received the herbicide treatment. Exchangeable acidity at 0 to 10 cm was significantly higher for the herbicide treated plots, and lower in the C and H compared to the F and HF, respectively. These differences in exchangeable acidity decreased with depth. No significant difference was found for effective cation exchange capacity at any depth although herbicide treated plots always tended to have lower values.

In Dawsonville significant differences in relation to treatments were only apparent in the 0 to 10 cm depth (Table 3.3). Exchangeable Ca and Mg in this depth were lower in herbicide treatment plots. Similarly, ECEC was significantly lower (α=0.1) for those plots that received the herbicide treatment.

Temporal patterns in soil available N

A season pattern in soil available N was observed for both the H and HF plots in all locations. In general, the highest mineralization rates, for all three locations, were observed during September and October (Table 3.4). The HF plots had much higher N availabilities than the corresponding H plots in all months of the year. HF plot averages across locations ranged from ~ 9 to >1700 $\mu g\ N \cdot g^{-1}$ while H values ranged from 4 to <860 $\mu g\ N \cdot g^{-1}$.

In the Athens location, based on the overall test using the split-plot model design for repeated measures, all three categorical variables (treatment, time, and treatment x time) were significant (i.e., $p<0.0001$). On average (Table 3.4) HF resin extractable N ranged from ~105 $\mu g\ N \cdot g^{-1}$ in January to ~3195 $\mu g\ N \cdot g^{-1}$ in September while H rates ranged from ~8 $\mu g\ N \cdot g^{-1}$ in May to ~77 $\mu g\ N \cdot g^{-1}$ in October.

In the Eatonton location the only significant variables were treatment ($p<0.05$) and time ($p<0.001$). On average HF resin extractable N rates ranged from ~128 $\mu g\ N \cdot g^{-1}$ (January) to ~1116 $\mu g\ N \cdot g^{-1}$ (September) while H rates ranged from ~4 $\mu g\ N \cdot g^{-1}$ (November) to ~171 $\mu g\ N \cdot g^{-1}$ (August).

In Dawsonville, treatment and the treatment x time interaction were not significant ($p=0.95$ and $p=0.77$, respectively), while time was significant ($p< 0.0001$). HF resin extractable N rates ranged from ~12 $\mu g\ N \cdot g^{-1}$ (March) to ~322 $\mu g\ N \cdot g^{-1}$ (September) while H rates ranged from ~25 $\mu g\ N \cdot g^{-1}$ (November) to ~506 $\mu g\ N \cdot g^{-1}$ (September).

Atmospheric mineral nitrogen inputs

Athens and Dawsonville had the same volume weighted mean annual concentration of total inorganic nitrogen in wet-only deposition (0.7 $mg\ L^{-1}$) that was only slightly greater than inputs to Eatonton (0.6 mg L^{-1}). Cumulative amounts of precipitation (Table 3.5) differed, however, with Dawsonville (209.2 cm) exceeding Athens (165.4 cm) and Eatonton (173.4 cm). As a result, Dawsonville also had a higher total inorganic N deposition (12.6 kg ha^{-1}) during the period of collection compared to Athens (9.9 kg ha^{-1}), and Eatonton (11.4 kg ha^{-1}).

Discussion

The results from these samplings, in terms of soil nutrient availability for the different treatments, are consistent with the findings regarding stand growth in that plots with higher soil available N and exchangeable P also have relatively higher growth rates. Resin N was, in fact, linearly correlated to mean annual increment (R^2=0.7) across these sites (Sartori, 2004).

Based on the observed tree growth response data (Borders and Bailey 2001), stands in Athens and Eatonton showed a clear response to fertilization (F), herbicide (H), and particularly their combination, fertilizer plus herbicide treatment (HF). For example, at age 12 in Athens mean annual increment was ~6 m^3 ha^{-1} and ~11 m^3 ha^{-1} greater than the corresponding Control plot for the H and HF, respectively (Table 3.6). In Eatonton at age 13 mean annual increment was ~6 m^3 ha^{-1} and ~13 m^3 ha^{-1} greater than the corresponding Control plot for the H and HF, respectively (Table 3.7). In Dawsonville (Table 3.8) the response to herbicide treatments was clear but that to fertilization was not so apparent likely due to the abandonment of annual fertilization. For example, at age 12 mean annual increment was ~6 m^3 ha^{-1} greater than the corresponding Control plot for both the H and HF.

In these stands, available N has been found well correlated to mean annual increment under the herbicide and herbicide plus fertilizer treatments (Sartori, 2004). This N to MAI relationship demonstrates that when P is not limiting N becomes the predominant predictor of pine growth. This growth prediction is best related to soil nutrient availability in the H and HF plots where growth is clearly defined by pine tree biomass estimates. In the C and F plots, other components of biomass production such as competing hardwood trees, understory shrubs, or herbaceous ground cover were not quantified and are thus confounding to these soil-site relationships.

There was not a clear distinction between treatments based on other nitrogen components. In general, surficial soil total nitrogen in Athens (Figure 3.1) and Eatonton (Figure 3.3) is slightly higher (not significantly) for plots that received the fertilization treatment whereas in Dawsonville (Figure 3.5) there is no clear pattern. These results reflect what was found using the mixed bead resin core technique (high and low N values for the H and HF, respectively, for Athens and Eatonton) and confirm previous studies that found total N correlates well to soil N availability indices (Binkley and Hart, 1989). At deeper depths these differences in N among treatments tended to disappear.

In Athens, based only on the resin extractable N estimates at 0-10 cm depth, the corresponding yearly cumulative N extracted was on average ~370 kg ha^{-1} yr^{-1} for the HF plots and 13.5 kg ha^{-1} yr^{-1} for the corresponding H plots. The corresponding soil available N as a percent of soil total N (~ 606 kg ha^{-1}, Figure 3.1) that is annually mineralized is on average ~ 2 % for the H plots. Similarly, in Eatonton total cumulative extracted N was on average ~18 kg ha^{-1} yr^{-1} for the H plots and ~270 kg ha^{-1} yr^{-1} for the HF. In the H plots the corresponding soil available N as a percent of soil total N (i.e. ~ 845 kg·ha^{-1}, Figure 3.3) was again ~ 2 %. In Dawsonville total cumulative extracted N was on average ~53 kg ha^{-1} yr^{-1} for the H plots and ~43 kg ha^{-1} yr^{-1} for the HF. In the H plots the corresponding soil available N as a percent of soil total N (i.e. ~1358 kg·ha^{-1}, Figure 3.5) was ~ 4 %.

The above estimates for annual resin N extraction in the H plots are within the range of previously reported data. For example, Piatek and Allen (1999) and Vitousek and Matson (1985) report that in the 0-15 cm layer net N mineralization under loblolly pine in the Piedmont of North Carolina decreased from 80-90 kg ha^{-1} yr^{-1} at age 1 to 40-50 kg ha^{-1} yr^{-1} at age 5, and had a highest overall value of 100 kg ha^{-1} yr^{-1} under a herbicide treatment. The estimated annual mineralization of the total soil N pool in the H plots are, however, relatively high compared to those of Polglase et al. (1992). Polglase et al. (1992) found, via periodic extractions of *in situ* soil cores on poorly drained sandy soils in northern Florida, % mineralization values of 0.007 % for control plots and 0.77 % for the combined fertilizer-weed control treatment in loblolly and slash pine stands.

The same percent mineralization estimates for the HF plots are difficult to compare with previous work because fertilizer is applied regularly each year in the HF plots of this study and, thus, available N is highly elevated, as are the mineralization estimates, relative to total soil N. To my knowledge, other authors have not reported similar findings in forest plots with annual fertilization. The annual fertilizer application of ~59 kg-N·ha^{-1} yr^{-1} (after the second growing season) has likely created the elevated spikes represented in Table 3.4 that are interpreted as high N mineralization estimates. Such spikes indicate that the mixed bead resin core technique may provide a more useful tool than laboratory incubation for quantifying the peak availability of N in mineral soils after fertilization. Laboratory incubations after fertilization often present difficulties (Polglase et al., 1992) and in some studies have indicated large immobilizations of N (Echeverria et al., 2004).

Temporal patterns in N availability for this study were observed in both H and HF plots (Table 3.4). In fact, relative peak N availability estimates were observed in both treatments in October despite fertilizer applications in HF plots in April. A similar sinusoidal pattern in N availability was reported by Vitousek and Matson (1985) for their harvested plots in North Carolina with a peak early in the growing season (April through June).

Atmospheric N inputs due to wet-only deposition were the same across locations (i.e., ~10 $kg \cdot ha^{-1} \cdot yr^{-1}$) (Table 3.5). Atmospheric deposition estimates are in agreement with those reported by other authors in the literature for this region (Richter and Markewitz, 1996). The similarity in N deposition across these sites simply demonstrates that atmospheric N inputs are not responsible for differences in growth across the sites and should not be considered as confounding for the comparison among treatments.

Although available N is likely a major driver of growth among the treatments at all sites, the presence of readily available P is essential for sustained tree growth. In Athens, P fertilization has significantly increased extractable P contents in the F and HF treatment plots at 0-10 cm depth (i.e., ~10 and ~18 kg ha^{-1}, respectively) and a similar significant increase was detected in the lower 30-50 cm depth. In all cases, however, available P only represents a small percentage of the corresponding soil total P that ranged between 563 and 926 kg ha^{-1} (Figure 3.2). Results in Eatonton were similar to Athens (Figure 3.4) where the surface extractable P was approximately six-fold higher in plots that received the fertilizer treatment (i.e., ~6 and 5 $kg \cdot ha^{-1}$ for F and HF plots, respectively) and represents only a small percentage of the corresponding soil total P that ranged from ~458 to 518 $kg \cdot ha^{-1}$. In Dawsonville (Figure 3.5) extractable P was always higher in the fertilized compared to the non fertilized plots in presence (~122 kg ha^{-1} vs. ~48 kg ha-^{1}) and in absence (~80 kg ha^{-1} vs. ~48 kg ha-^{1}) of the herbicide treatment. The P fertilization in this study caused a long-term effect on the soil nutrient status since P was not applied annually like N. The retention of P fertilizer over decade or even centuries time scale has been observed by other authors (e.g. Fransson and Bergkvist, 2000). The long-term recycling of P fertilizer inputs through organic residues was demonstrated in a N. Florida pine plantation and would be important at these sites for the sustained availability of P and the subsequent effect on growth (Polglase et al., 1992).

The use of readily available ammonium nitrate and diammonium phosphate (DAP) fertilizers, containing ammonium forms, and of KCl during the first two growing seasons lowered soil pH and increased exchangeable acidity significantly in the fertilized plots compared to the non-fertilized ones. Such a fertilizer effect is typically found in forest and agro-forest systems subject to the use of acidifying fertilizers (Jokela et al., 1991), but may also result from other mechanisms such as the presence of organic acid derived from litter decomposition (Binkley et al., 1989).

There are some other indirect consequences of soil acidification, for example, the availability of micro-nutrients such as iron may be increased (Pritchett and Fisher, 1987). Another possible indirect effect of soil acidification is the reduction of nitrification when pH is below 4.5-5.0 (Morris and Campbell, 1991). Soil acidification may also lower negative charge generated through variable charge processes (Thomas, 1996). In general, however, soil acidification in these ecosystems does not affect the growth of pine species that are well suited to acidic soils (Jokela et al., 1991).

Acidification is also a consequence of the relatively low buffering capacity of Ultisols with low base saturation and mineral weathering release. The Hapludult soils in Eatonton and Athens had a much lower ECEC (Table 3.3), on the order of one third, compared to the Udifluvent soils in Dawsonville. ECEC appears to follow the same pattern as for soil C for all three locations being lower in presence of the herbicide treatment and as a consequence exchangeable cations follow a similar pattern.

Exchangeable Ca (Table 3.3) considered the next most important nutrient for possible deficiencies after N, P, and K (Jorgensen and Wells, 1986; Huntington et al., 2000) was also lower in presence of the herbicide treatment at 0-10 cm depth in Athens. The herbicide treatment effect on Ca was relatively less detectable in Eatonton at 0-10 cm depth but elicited a clear and significant decline in H plots for Dawsonville. In the absence of the herbicide treatment, Ca was generally higher in the fertilized plots both at the surface and at depth. These results suggest that any loss of organic matter due to herbicide treatments or exchange complex acidification due to fertilization may well limit the availability of Ca for growth. Conversely, fertilization inputs appear able to counter these declines.

Exchangeable Mg, in Athens, followed a similar and significant pattern as for Ca being lower under the herbicide treatment with differences among treatments decreasing with depth. In Eatonton, there was a significant decline in exchangeable Mg associated with the herbicide treatment at 0-10 cm but not at deeper depths. Similarly, in Dawsonville there was a significant pattern due to the herbicide treatment but declining with depth. Such patterns resulted again from the combined fertilization and herbicide treatment effects that can increase soil exchangeable Mg concentrations but can also reduce the presence of negatively charged sites available on soil organic matter to bind these plant essential cations.

Results relating to total C are in agreement with those reported by Echeverria et al. (2004) who conducted a study using some of the same stands and found total C was significantly lower in the herbicide plots compared to the other treatments. In addition to this pattern in Athens at 0-10 cm depth (Figure 3.1) plots that received the F treatment in presence or in absence of the herbicide appeared to have a higher (but not significantly) surficial soil C content, being ~17.3, 23.5, 15.4, and 17.2 Mg C ha^{-1} for the C, F, H, and HF treatment, respectively. This result would suggest that fertilization had a beneficial effect on soil C although not significant. Results in the other two locations confirmed what was found on a more limited number of plots by Echeverria et al (2004): in Eatonton (Figure 3.3) plots under the herbicide treatment had lower surficial C, being 19.8, 20.3, 18.5, and 18.5 Mg C ha^{-1} for the Control, F, H, and HF, respectively. Similarly, in Dawsonville (Figure 3.5) this effect was observed although it was not statistically significant. Here the surface C was 28.6, 30.0, 23.5, and 23.1 Mg C ha^{-1} for the Control, F, H, and HF, respectively. These results appear to indicate that the statistically significant declines observed in response to herbicide treatments were not balanced by increases under fertilization. In other words, HF plots typically had lower surface soil C contents relative to control plots.

The above-ground biomass growth rates in these stands were significantly higher under both the herbicide and fertilizer treatment (Borders and Bailey, 2001). These increased growth rates in the oldest stands translate into increased C inputs through litterfall on the order of 3 to 4 Mg-C ha^{-1} yr^{-1}. These increased annual inputs, however, do not correspond to increased mineral soil organic C. This result is in agreement with other previous studies underscoring that C sequestration occurs primarily in the above ground biomass (Richter et al., 1995; Shan et. al., 2001; Markewitz et al., 2002). Much of the increased litterfall input was retained, however, in the soil O horizon (Borders et al., 2004).

Conclusions

Based on the fertilization regime and experimental design of this study it is not possible to define whether N or P is the major driver of growth in these stands. None-the-less, on the relatively nutrient poor Ultisols in Athens and Eatonton high values of soil available N and P were associated with high growth rates for the stands. Other soil nutrients did not demonstrate this relationship with growth. On the nutrient richer Udifluvents in Dawsonville it was not possible to find such a clear correspondence between the soil N and P concentrations and the response to fertilization and herbicide treatment in the above ground biomass.

In all sites, however, the use of the mixed bead resin core technique could clearly detect large differences between N availability in the H and HF treatments, with the latter being about 100 fold higher or more under favorable temperature and moisture conditions. Mineralization rates in the fertilized plots were extremely high in response to the annual N fertilizer application in early spring. Rainfall N inputs were very uniform across the three locations and not relevant to observed difference in soil N availability or tree growth.

In all three locations the treatments also affected soil exchange chemistry. These differences were most apparent in the surfaces soil for pH, Ca, and ECEC but were not consistent across all sites. These changes in the exchange complex were closely related to reductions in soil C that were observed with herbicide treatments. The fertilization treatment did not have a corresponding consistent increasing effect on soil C content suggesting that such treatments would likely not buffer and counter the negative effects of the herbicide treatment on soil C.

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Table 3.1. Three-way MANOVA Wilk's lambda p-values by location, depth, and model categorical variable. Total C, N, extractable P, exchangeable acidity, and ECEC were considered as predictors of the overall MANOVA test for treatment effect to account for possible correlation between variables measured on the same soil sample. All locations are in the Piedmont of Georgia. Soil variables were measured in 2003 when stands ranged in age from 12 to 15 years old.

Depth		Athens	Eatonton	Dawsonville
cm			p value	
0-10	TRT[1]	0.019	<0.0001	0.516
	Block	0.219	0.123	0.326
10-30	TRT	0.170	0.2008	0.717
	Block	0.182	0.1255	0.127
30-50	TRT	0.016	0.3661	0.984
	Block	0.017	0.0047	0.925

[1] Treatment (Control, Herbicide, Fertilizer, and their combination HF).

Table 3.2. Total C, N, P, and extractable P (mean±1SE) measured for replicate loblolly pine stands within the Consortium for Accelerated Pine Productivity Studies (CAPPS) in Georgia. The control (C), fertilizer (F), herbicide (H), and HF treatments were sampled in December 1999 and January 2000 in Eatonton (n=40), in July and August 2001 in Athens (n=12), and in March 2003 in Dawsonville (n=14).

Location	Depth	Treatment	C	N	P	Ext-P
	cm		%	%	ug·g^{-1}	
Athens	0-10	C	1.5±0.2	0.05±0.01	485±71	1.7±0.1
		F	2.0±0.6	0.11±0.04	566±85	8.6±1.9
		H	1.1±0.1	0.04±0.00	502±29	1.4±0.2
		HF	1.3±0.3	0.06±0.01	691±55	14.0±5.7
						B,c[1]
	10-30	C	0.6±0.1	0.03±0.01	455±85	0.8±0.1
		F	0.5±0.1	0.03±0.01	642±82	2.0±0.5
		H	0.5±0.0	0.03±0.00	451±13	1.2±0.5
		HF	0.6±0.1	0.03±0.01	576±19	1.8±0.1
	30-50	C	0.3±0.1	0.02±0.00	446±88	0.6±0.1
		F	0.3±0.0	0.01±0.00	585±74	0.9±0.2
		H	0.3±0.0	0.01±0.00	516±20	0.8±0.2
		HF	0.3±0.0	0.01±0.00	435±41	1.7±0.0
Eatonton	0-10	C	1.5±0.2	0.07±0.01	339±48	1.2±0.5
		F	1.5±0.2	0.08±0.01	389±37	4.8±1.2
		H	1.3±0.1	0.06±0.00	341±30	0.8±0.2
		HF	1.4±0.1	0.07±0.01	402±36	3.9±1.1
						B,C
	10-30	C	0.81±0.18	0.041±0.01	319±39	0.4±0.2
		F	0.72±0.08	0.04±0.00	336±22	1.3±0.8
		H	0.58±0.05	0.032±0.00	361±28	0.3±0.1
		HF	0.55±0.03	0.034±0.00	340±25	0.4±0.1
			A			
	30-50	C	0.32±0.05	0.021±0.00	345±36	0.3±0.1
		F	0.35±0.02	0.025±0.00	335±33	0.3±0.1
		H	0.31±0.02	0.021±0.00	402±40	0.3±0.1
		HF	0.29±0.01	0.023±0.00	330±29	0.3±0.1
Dawsonville	0-10	C	2.0±0.0	0.12±0.00	NA	34.0±7.4
		F	2.1±0.4	0.13±0.03	NA	87.0±37.0
		H	1.7±0.1	0.10±0.01	NA	51.0±18.0
		HF	1.6±0.0	0.09±0.00	NA	58.0±13.0

Table 3.2 (Continued)

					C
10-30	C	1.4±0.1	0.08±0.00	NA	19.0±5.7
	F	1.4±0.3	0.07±0.02	NA	39.0±18.0
	H	1.2±0.2	0.07±0.01	NA	25.0±8.3
	HF	1.3±0.1	0.07±0.00	NA	18.0±4.2
					C
30-50	C	1.1±0.2	0.06±0.01	NA	2.8±0.3
	F	0.7±0.1	0.04±0.01	NA	6.5±4.5
	H	0.9±0.2	0.05±0.01	NA	5.0±0.6
	HF	1.0±0.2	0.06±0.01	NA	4.3±0.8

[1] Orthogonal contrasts at each depth: letters beneath a column indicate significance at $p < 0.05$ (capitals) or 0.10 (lower case) for contrasts: A) Herbicide plots (H and HF) vs. non-herbicide ones (C and F), B) Herbicide (H) vs. Herbicide plus Fertilizer (HF), C) Fertilizer (F) vs. Control (C).

Table 3.3. pHw, pHs, and exchangeable cations, exchangeable acidity, and effective cation exchange capacity (mean±1SE) for Accelerated Pine Productivity Studies measured for replicate loblolly pine stands (N=66) that received the four different treatments and were sampled in December 1999 and January 2000 in Eatonton (n=40), in July and August 2001 in Athens (n=12), and in March 2003 in Dawsonville (n=14).

	Depth	Treat.[1]	pHw	pHs	Ca	Mg	K	Exc.-Ac.	ECEC
					——— $cmol_c\ kg^{-1}$ ———				
Athens	0-10	C	5.6±0.1	4.7±0.1	1.4±0.2	0.8±0.1	0.2±0.0	0.3±0.1	2.6±0.3
		F	4.4±0.0	3.9±0.1	1.9±1.2	0.8±0.1	0.2±0.0	1.2±0.3	4.1±1.0
		H	5.3±0.1	4.4±0.1	0.9±0.2	0.5±0.1	0.2±0.1	0.5±0.1	2.1±0.4
		HF	4.5±0.0	3.9±0.1	0.4±0.1	0.2±0.1	0.1±0.0	1.3±0.1	2.1±0.2
			B,C	B,C		A,B		B,C	A,c
	10-30	C	5.4±0.1	4.5±0.1	0.7±0.1	0.8±0.2	0.2±0.1	0.4±0.1	2.1±0.3
		F	4.9±0.2	4.5±0.1	1.5±0.5	1.0±0.1	0.3±0.0	0.4±0.1	3.1±0.3
		H	5.4±0.1	4.5±0.1	0.7±0.1	0.8±0.1	0.1±0.0	0.4±0.1	2.1±0.2
		HF	5.2±0.0	4.6±0.1	1.2±0.2	0.6±0.2	0.2±0.1	0.4±0.1	2.5±0.2
			C						A,C
	30-50	C	5.5±0.0	4.5±0.1	0.5±0.1	0.9±0.2	0.1±0.1	0.4±0.1	2.1±0.3
		F	5.2±0.2	4.9±0.3	1.4±0.6	1.4±0.1	0.3±0.0	0.1±0.1	3.2±0.4
		H	5.4±0.1	4.6±0.1	0.6±0.1	1.0±0.1	0.2±0.0	0.3±0.0	2.1±0.2
		HF	5.4±0.0	5.1±0.2	1.3±0.4	0.9±0.2	0.3±0.1	0.1±0.1	2.6±0.1
							c		C

Table 3.3 (Continued)

Eatonton	0-10	C	5.3±0.0	4.8±0.1	2.4±0.6	0.8±0.1	0.2±0.0	0.3±0.0	3.7±0.7
		F	4.9±0.1	4.4±0.1	1.6±0.2	0.6±0.1	0.2±0.0	0.8±0.1	3.2±0.3
		H	5.3±0.0	4.7±0.1	2.0±0.3	0.6±0.1	0.1±0.0	0.4±0.1	3.0±0.3
		HF	4.8±0.1	4.2±0.1	1.3±0.2	0.4±0.1	0.1±0.0	1.0±0.1	2.8±0.2
			B,C	A,B,C		A,b	A	A,B,C	
	10-30	C	5.4±0.1	4.8±0.1	1.6±0.6	0.9±0.1	0.1±0.0	0.4±0.1	3.0±0.6
		F	5.0±0.1	4.5±0.1	1.4±0.2	0.9±0.2	0.1±0.0	0.7±0.2	3.2±0.4
		H	5.3±0.1	4.6±0.1	1.5±0.3	0.8±0.1	0.1±0.0	0.4±0.1	2.8±0.3
		HF	5.0±0.1	4.6±0.1	1.5±0.2	0.7±0.1	0.1±0.0	0.4±0.1	2.8±0.2
			B,C	c				c	
	30-50	C	5.5±0.0	4.7±0.1	1.1±0.2	1.1±0.1	0.1±0.0	0.5±0.1	2.8±0.2
		F	5.1±0.1	4.7±0.1	1.6±0.3	1.2±0.2	0.1±0.0	0.6±0.3	3.5±0.5
		H	5.5±0.0	4.8±0.1	1.4±0.3	1.1±0.1	0.1±0.0	0.2±0.1	2.8±0.3
		HF	5.2±0.0	4.7±0.1	1.4±0.2	1.1±0.1	0.1±0.0	0.3±0.1	2.8±0.2
			B,C					a	
Dawsonv.	0-10	C	NA	NA	11.3±1.7	2.7±0.3	0.7±0.1	0.5±0.2	15.2±2.0
		F	NA	NA	11.4±2.7	2.7±0.8	0.6±0.2	0.2±0.0	15.0±3.7
		H	NA	NA	7.6±1.3	1.9±0.3	0.5±0.1	0.7±0.2	10.65±1.6
		HF	NA	NA	6.6±1.6	1.6±0.3	0.5±0.1	1.2±0.2	9.9±1.7
					a	a			a
	10-30	C	NA	NA	10±1.1	2.4±0.2	0.5±0.1	0.4±0.2	13.3±1.3
		F	NA	NA	10.3±3.1	2.2±0.8	0.5±0.2	0.4±0.3	13.4±4.3
		H	NA	NA	8.2±1.2	2.4±0.3	0.4±0.1	0.5±0.2	11.6±1.6
		HF	NA	NA	8.5±0.8	2.1±0.3	0.5±0.1	0.4±0.1	11.5±1.0

Table 3.3 (Continued)

30-50	C	NA	NA	6.2±0.4	2.2±0.2	0.3±0.1	0.4±0.2	9.3±0.4
	F	NA	NA	5.5±1.6	1.7±0.5	0.3±0.0	0.6±0.5	8.1±2.5
	H	NA	NA	5.2±0.6	1.8±0.2	0.4±0.1	0.5±0.2	8.0±0.7
	HF	NA	NA	5.7±0.1	1.9±0.2	0.4±0.0	0.3±0.2	8.4±0.2

[1] Testing orthogonal contrasts at each depth: letters beneath a column indicate significance at $p < 0.05$ (capitals) or 0.10 (lower case) for contrasts: A) Herbicide plots (H and HF) vs. non-herbicide ones (C and F), B) Herbicide (H) vs. Herbicide plus Fertilizer (HF), C) Fertilizer (F) vs. Control (C). In Dawsonville the herbicide and fertilization treatments were applied only until 1996.

Table 3.4. Resin-extractable N (NO_3-N plus NH_4-N) concentration (mean ± (1SE), ug N·g^{-1} resin, n=2 plots) and corresponding annual cumulative content (kg N ha^{-1}), estimated using the mixed bead resin core technique in the 0-10 cm depth on a monthly basis during 2002-2003 for all HF- and H-plots located in Athens (A), Dawsonville (D), and Eatonton (E), Georgia.

Month	Herbicide			Herbicide plus Fertilizer		
	A	E	D	A	E	D
August	6.3 (2.5)	171.1 (166.5)[1]	69.9 (57.7)	550.6 (124.5)	593.3 (253.2)	8.8 NA
September	27.6 (8.0)	10.5 (1.1)	506.3 (351.3)	3195.0 NA	1115.5 (283.7)	322.0 NA
October	76.7 (4.4)	88.5 (1.4)	222.3 (144.5)	1197.9 (323.4)	1286.0 (18.5)	223.3 (74.1)
November	15.3 (3.2)	4.1 (0.1)	25.9 (12.7)	1113.9 (355.2)	252.0 (6.6)	46.8 (19.1)
December	66.1 (7.4)	39.3 (3.7)	87.8 (21.8)	355.8 (14.8)	571.7 (304.4)	119.8 (20.1)
January	28.8 (20.6)	13.4 (9.5)	47.8 (14.7)	105.2 (14.3)	128.6 (57.6)	45.9 (1.1)
February	11.5 (3.6)	15.5 (3.1)	34.6 (24.2)	109.4 (7.8)	133.4 (12.6)	37.6 (2.4)
March	10.3 (2.6)	19.4 (0.1)	54.0 (44.2)	144.1 (11.0)	178.5 (21.1)	12.3 (2.0)
April	13.6 (4.3)	11.8 (6.7)	39.1 (20.7)	168.9 (16.4)	219.3 -	49.9 (20.3)
May	8.0 (3.9)	8.9 (0.4)	44.2 (32.5)	753.1 (205.1)	886.4 (217.7)	35.8 (2.5)
June	28.2 (6.6)	41.4 (1.3)	69.8 (30.8)	794.7 (43.1)	770.0 (82.5)	72.3 (26.4)
July	32.5 (22.4)	20.5 (8.3)	90.6 (82.0)	456.9 (68.7)	443.7 (42.7)	59.7 (7.9)

Table 3.4 (Continued)

	—kg N ha^{-1} yr^{-1}—			—kg N ha^{-1} yr^{-1}—		
Cumulative[2]	13.5	18.5	53.7	371.9	273.5	43.0

[1] Standard error of the mean by site, collection date, and treatment.
[2] The corresponding cumulative contents were computed considering that each bag contained 4 g of resins and the soil core volume internal to the PVC tube was ~96.2 cm^3 (depth=10 cm).

Table 3.5. Monthly average total mineral nitrogen (NO3-N plus NH4-N) concentration, contents, and monthly precipitation and average temperature during 2002-2003 for the three study locations, Athens, Dawsonville, and Eatonton.

Month	Athens				Eatonton				Dawsonville			
	T[1]	P[2]	Conc.[3]	Cont.[4]	T	P	Conc.	Cont.	T	P	Conc.	Cont.
	ºC	cm	ug ml^{-1}	kg ha^{-1}	ºC	cm	ug ml^{-1}	kg ha^{-1}	ºC	cm	ug ml^{-1}	kg ha^{-1}
Jul	27.2	6.3	0.4	0.3	27.1	10	0.3	0.3	25.9	6.9	1	0.7
Aug	26.8	3.4	0.7	0.2	22.3	0.2	0.7	0	25.5	3.7	1.1	0.4
Sept	24.4	19.6	0.3	0.5	22.7	7	0.1	0.1	23	18.3	0.4	0.7
Oct	18.4	7.7	1.1	0.8	12.6	14.2	0.8	1.1	17	20.7	0.4	0.9
Nov	10.3	11.9	0.4	0.4	11.7	10.5	0.3	0.3	8.9	15.9	0.3	0.5
Dec	6.6	15.1	0.4	0.6	11.5	13.9	0.7	0.9	5.6	24.3	0.2	0.5
Jan	5.1	4.5	0.7	0.3	6.8	19.1	0.7	1.3	3.1	6.6	0.7	0.5
Feb	8.1	12	0.3	0.4	8.7	12.5	0.3	0.4	6.5	17.5	0.5	0.9
Mar	13.2	13.3	1.2	1.6	5.9	13.7	0.6	0.8	12.3	11.8	0.6	0.7
April	16.3	7.3	1.7	1.3	21.2	13.8	0.5	0.7	15.7	12.3	0.7	0.9
May	20.6	17.6	0.8	1.4	22.7	19.4	0.6	1.2	19.2	19	0.5	1
Jun	23.8	13.8	0.5	0.7	24.4	10.9	0.9	1	22.7	16.6	0.4	0.7
Jul	25.7	25.5	0.3	0.7	25.6	15.8	0.5	0.8	24.4	11.5	0.9	1
Aug	26	7.1	0.9	0.6	27.2	12	2	2.4	25.4	12	1.5	1.8
Sept	22.4	0.4	0.6	0	20.4	0.3	1.2	0	21	12.3	1.1	1.4

Table 3.5 (Continued)

18.3†	165.4‡	0.7†	9.9‡	18.0†	173.4‡	0.6†	11.4‡	17.1†	209.2‡	0.7†	12.6‡

[1] Monthly average temperature.
[2] Monthly precipitation in cm.
[3] Average rain sample concentration by location.
[4] Corresponding estimated total mineral N content on a per ha basis.
†, Simple average value for temperature (T) and volume weighted average for concentration.
‡, Cumulative amount.

Table 3.6. Total stem volume outside bark estimated using allometric equations (Harrison and Borders, 1996) and corresponding mean annual increment (MAI) measured in 2000 for loblolly pine stands at Whitehall Forest in Athens, GA.

BLOCK	TRT[1]	FGS[2]	Age	Density[3]	VOB[4]	MAI
		yr	yrs	Trees ha^{-1}	m^3 ha^{-1}	m^3 ha^{-1}
1	C	89	12	1443	187.5	15.6
	F	89	12	1206	177.5	14.8
	H[5]	89	**12**	**1483**	**257.8**	**21.5**
	HF	89	**12**	**1423**	**317.9**	**26.5**
2	C	89	12	1443	180.9	15.1
	F	89	12	988	189.3	15.8
	H	89	**12**	**1305**	**249.2**	**20.8**
	HF	89	**12**	**1344**	**306.2**	**25.5**
3	C	93	8	1404	54.4	6.8
	H	93	8	1601	142.1	17.8
4	C	93	8	1522	62.5	7.8
	H	93	8	1581	139.2	17.4

[1] C= control, F=fertilized, H=herbicide, and HF= combined H plus F treatment. Experimental plots were randomly assigned to the different treatments and blocks were considered to account for the local variability.
[2] First growing season since stand establishment.
[3] Initial stand density was 1660 trees ha^{-1} (Borders et al., 2004).
[4] Tree volume outside bark was estimated using an allometric equation on individual trees and computing the corresponding sum for the total volume per ha basis.
[5] Bold faced plots were repeatedly measured for soil available N using the mixed bead resin core technique.

Table 3.7. Stem volume outside bark (TVOB) estimated using allometric equations (Harrison and Borders, 1996) and corresponding mean annual increment (MAI) measured in 2000 for loblolly pine stands at the "Powerline" site in the B.F. Grant Forest near Eatonton, GA.

BLOCK[1]	TRT[2]	FGS[3]	Age	Density[4]	VOB[5]	MAI
		yr	yrs	Trees ha^{-1}	———$m^3\ ha^{-1}$———	
1	C	88	13	1542	167.9	12.9
	F	88	13	1305	243.7	18.7
	H	**88**	**13**	**1562**	**251.3**	**19.3**
	HF	**88**	**13**	**1463**	**319.7**	**24.6**
2	C	88	13	1581	153.5	11.8
	F	88	13	1206	223.8	17.2
	H	**88**	**13**	**1502**	**235.1**	**18.1**
	HF	**88**	**13**	**1285**	**341.0**	**26.2**
3	C	90	11	1423	93.3	8.5
	F	90	10	1483	132.8	13.3
	H	**90**	**11**	**1483**	**226.3**	**20.6**
	HF	90	11	1562	289.8	26.3
4	C	90	11	1522	107.3	9.8
	F	90	11	870	112.5	10.2
	H	90	11	890	120.0	10.9
	HF	90	11	890	147.6	13.4
5	C	95	6	1542	39.8	6.6
	F	95	6	1601	28.2	4.7
	H	95	6	1601	72.9	12.1
	HF	95	6	1601	88.4	14.7

[1] C= control, F=fertilized, H=herbicide, and HF= combined H plus F treatment. Experimental plots were randomly assigned to the different treatments and blocks were considered to account for the local variability.

[2] First growing season since stand establishment.

[3] Initial stand density was 1660 trees ha^{-1} (Borders et al., 2004).

[4] Tree volume outside bark was estimated using an allometric equation on individual trees and computing the corresponding sum for the total volume per ha basis.

5 Plots in bold face were repeatedly measured for soil available N using the mixed bead resin core technique.

Table 3.8. Tree volume outside bark (TVOB) estimated using allometric equations (Harrison and Borders, 1996) and corresponding mean annual increment (MAI) measured in 2000 for loblolly pine stands near Dawsonville, GA.

BLOCK	TRT[1]	FGS[2]	Age	Density[3]	VOB[4]	MAI
		yr	yrs	Trees ha^{-1}	m^3 ha^{-1}	
1	C	87	14	712	131.2	9.4
	F[5]	87	6	158	-	
	H[6]	**87**	**14**	**1265**	**346.1**	**24.7**
	HF	**87**	**14**	**1067**	**354.2**	**25.3**
2	C	87	14	1048	201.9	14.4
	F[5]	87	6	217	-	
	H	87	14	1226	352.5	25.2
	HF[5]	87	14	969	289.9	20.7
3	C	89	12	890	149.9	12.5
	F[5]	89	12	811	127.9	10.7
	H	89	12	1186	281.0	23.4
	HF	89	10	613	137.4	13.7
4	C	89	12	1147	201.1	16.8
	F[5]	89	12	554	85.1	7.1
	H	**89**	**12**	**1384**	**276.3**	**23.0**
	HF	**89**	**12**	**1067**	**278.5**	**23.2**

[1] C= control, F=fertilized, H=herbicide, and HF= combined H plus F treatment. Experimental plots were randomly assigned to the different treatments and blocks were considered to account for the local variability.
[2] First growing season since stand establishment.
[3] Initial stand density was 1660 trees ha^{-1} (Borders et al., 2004).
[4] Tree volume outside bark was estimated using an allometric equation on individual trees and computing the corresponding sum for the total volume per ha basis.
[5] Trees in some of the fertilized plots were suppressed by the understory vegetation and their treatment was abandoned.
6 Plots in bold face were repeatedly measured for soil available N using the mixed bead resin core technique.

Figure legend.

Figure 3.1. Soil total C and N by depth in the Athens location (mean ± SE). Samples were collected in July and August 2001 (n=12). Contents were estimated using an average bulk density, B.D.= 1.34 g cm^{-3} and B.D.= 1.16 g cm^{-3} for plots with and without the herbicide treatment, respectively.

Figure 3.2. Soil total and extractable P by depth in the Athens location (mean ± SE). Samples were collected in July and August 2001 (n=12). Contents were estimated using an average, B.D.= 1.34 g cm^{-3} and B.D.= 1.16 g cm^{-3} for plots with and without the herbicide treatment, respectively.

Figure 3.3. Soil total C and N by depth in the Eatonton location (mean ± SE). Samples were collected in December 1999 and January 2000 (n=40). Contents were estimated using an average soil bulk density of 1.35, 1.33, 1.42, and 1.36 g cm^{-3} for the C, F, H, and HF treatment, respectively.

Figure 3.4. Soil total P and extractable P by depth in the Eatonton location (mean ± SE). Samples were collected in December 1999 and January 2000 (n=40). Contents were estimated using an average soil bulk density of 1.35, 1.33, 1.42, and 1.36 g cm^{-3} for the C, F, H, and HF treatment, respectively.

Figure 3.5. Soil total C and N by depth in the Dawsonville location (mean ± SE). Samples were collected in March 2003 (n=14). Contents were estimated using an average B.D.= 1.4 g cm^{-3} for all different treatments (Newton and Rockadale County, GA USDA-SCS, 1999).

Figure 3.6. Soil extractable P by depth in the Dawsonville location (mean ± SE). Samples were collected in March 2003 (n=14). Contents were estimated using an average B.D.= 1.4 g cm^{-3} for all different treatments (Newton and Rockadale County, GA USDA-SCS, 1999). .

Figure 3.1. Soil total C and N by depth in the Athens location (mean ± SE). Samples were collected in July and August 2001 (n=12). Contents were estimated using an average bulk density, B.D.= 1.34 g cm^{-3} and B.D.= 1.16 g cm^{-3} for plots with and without the herbicide treatment, respectively.

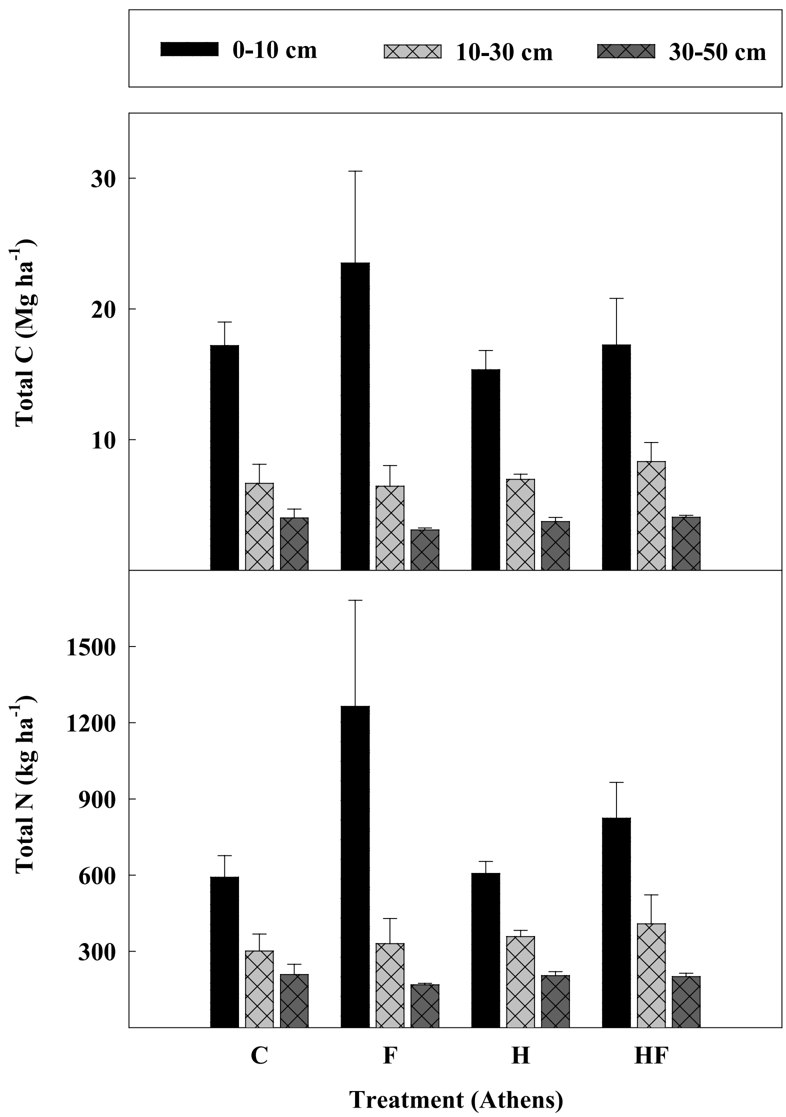


Figure 3.2. Soil total and extractable P by depth in the Athens location (mean ± SE). Samples were collected in July and August 2001 (n=12). Contents were estimated using an average, B.D.= 1.34 g cm^{-3} and B.D.= 1.16 g cm^{-3} for plots with and without the herbicide treatment, respectively.

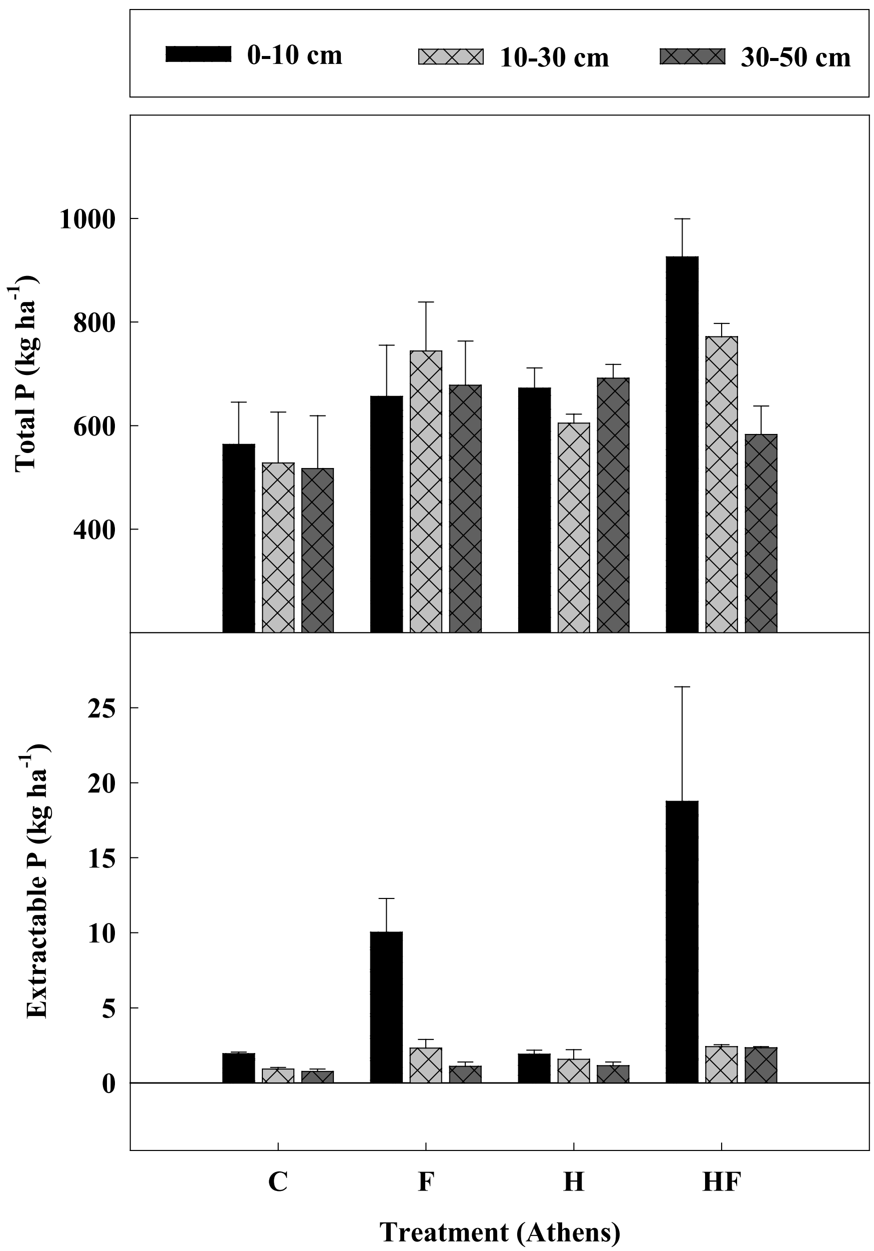


Figure 3.3. Soil total C and N by depth in the Eatonton location (mean ± SE). Samples were collected in December 1999 and January 2000 (n=40). Contents were estimated using an average soil bulk density of 1.35, 1.33, 1.42, and 1.36 g cm^{-3} for the C, F, H, and HF treatment, respectively.

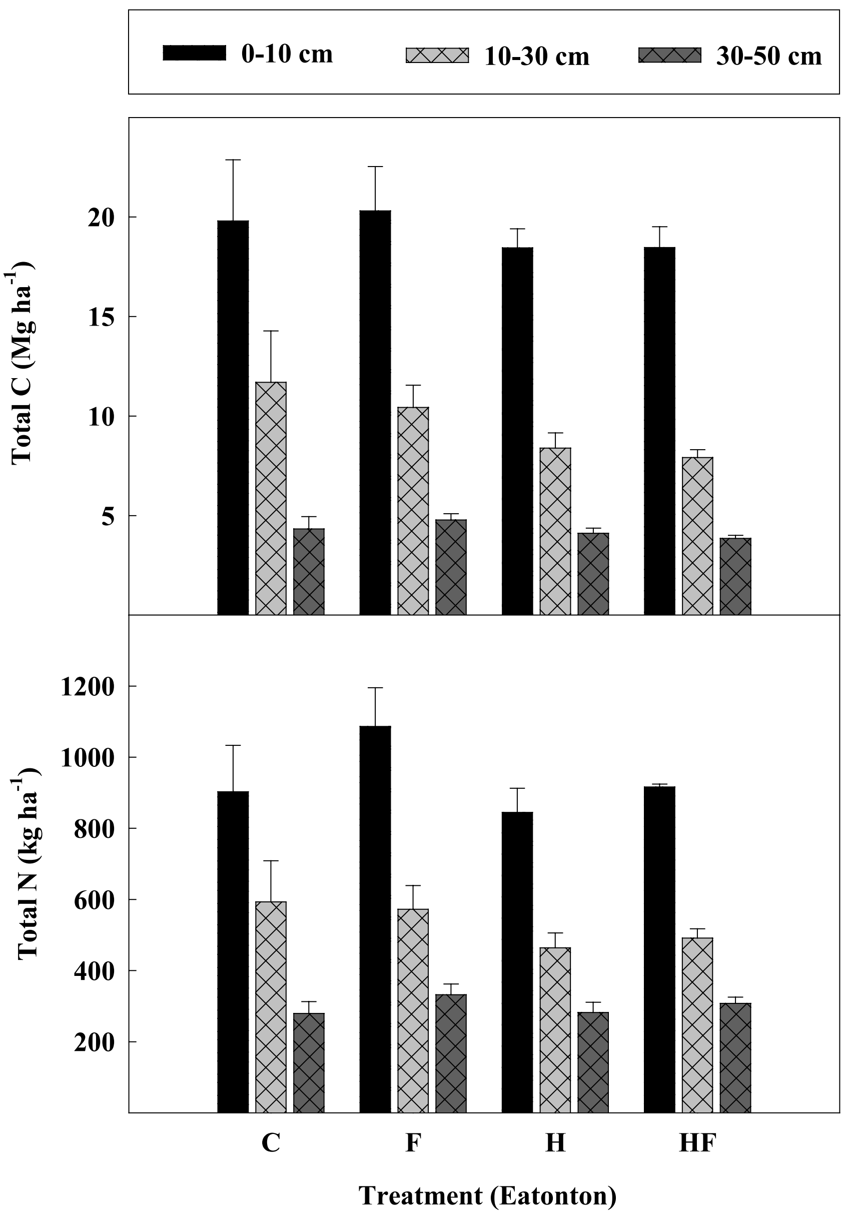


Figure 3.4. Soil total P and extractable P by depth in the Eatonton location (mean ± SE). Samples were collected in December 1999 and January 2000 (n=40). Contents were estimated using an average soil bulk density of 1.35, 1.33, 1.42, and 1.36 g cm^{-3} for the C, F, H, and HF treatment, respectively.

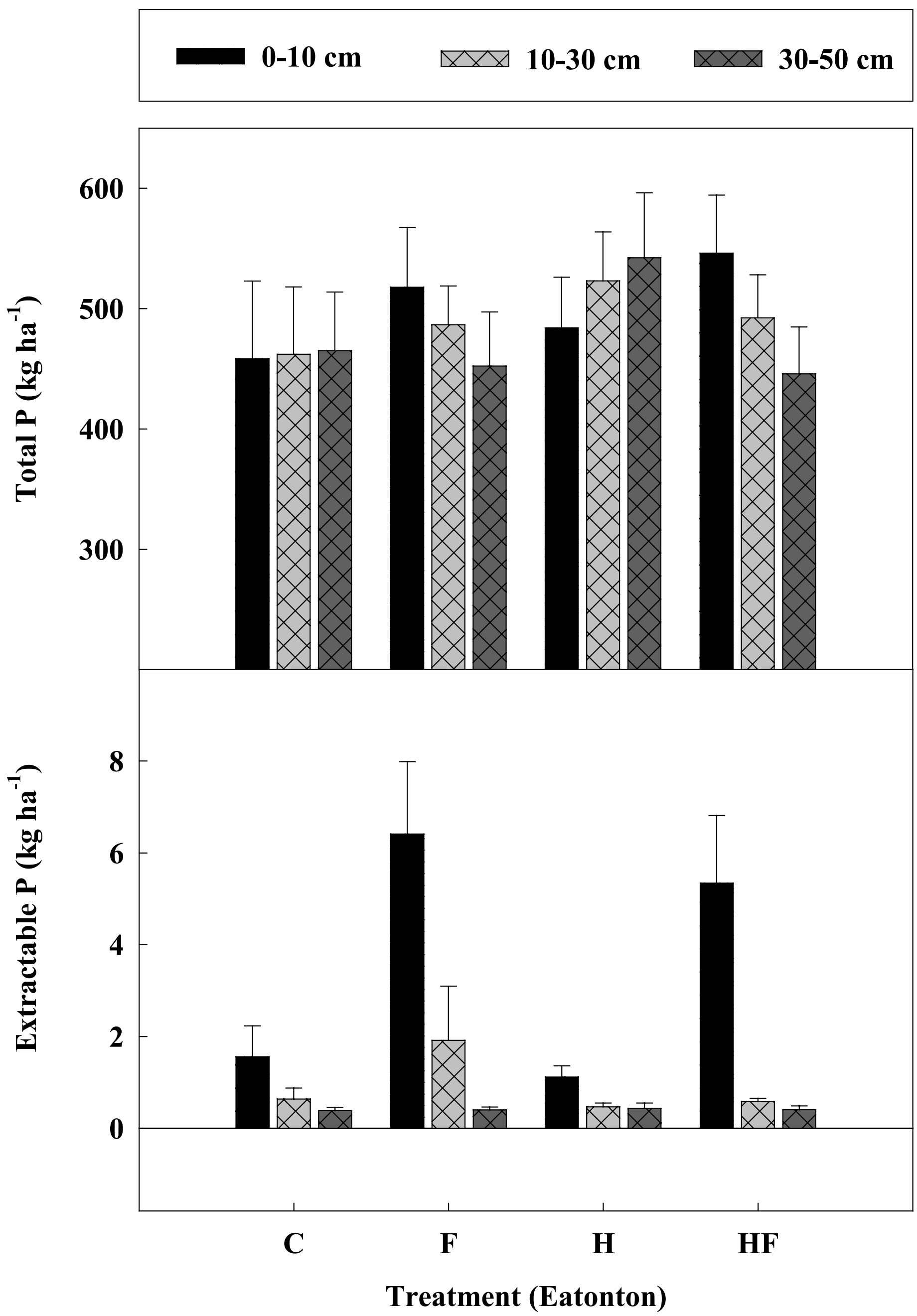


Figure 3.5. Soil total C and N by depth in the Dawsonville location (mean ± SE). Samples were collected in March 2003 (n=14). Contents were estimated using an average B.D.= 1.4 g cm^{-3} for all different treatments (Newton and Rockadale County, GA USDA-SCS, 1999).

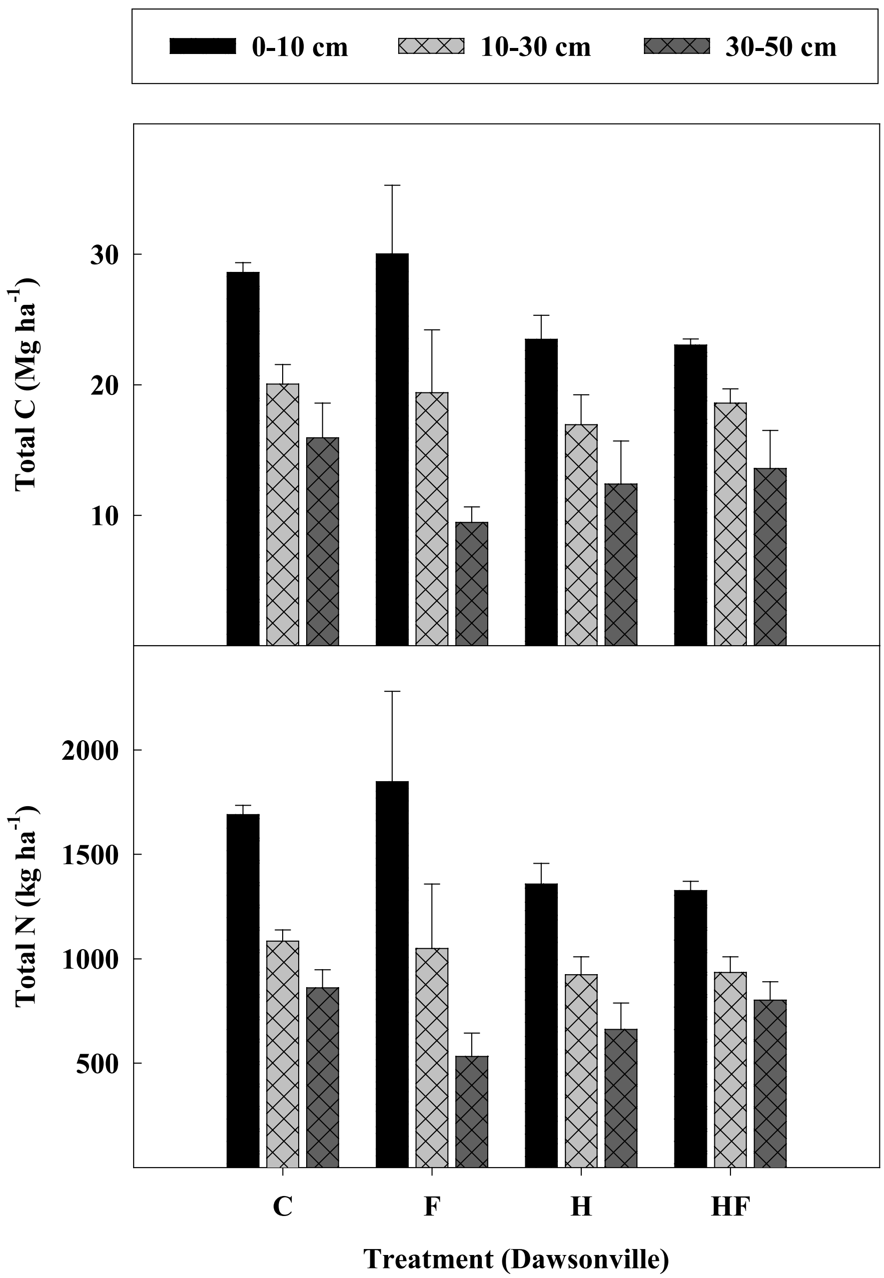
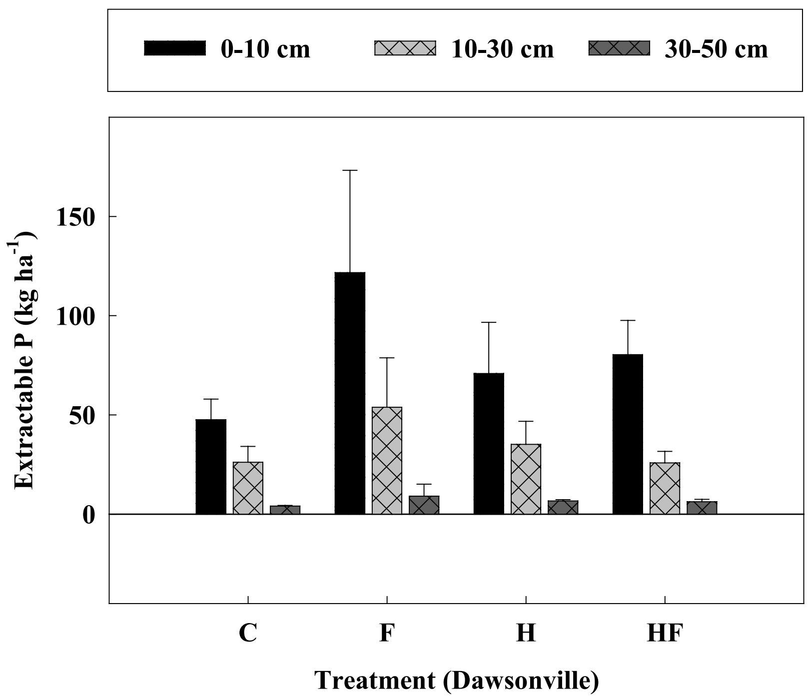


Figure 3.6. Soil extractable P by depth in the Dawsonville location (mean ± SE). Samples were collected in March 2003 (n=14). Contents were estimated using an average B.D.= 1.4 g cm^{-3} for all different treatments (Newton and Rockadale County, GA USDA-SCS, 1999).



CHAPTER 4

RELATIONSHIP BETWEEN INDICES OF SOIL N AVAILABILITY AND THE LIGHT ORGANIC MATTER FRACTION IN INTENSIVELY MANAGED LOBLOLLY PINE (*PINUS TAEDA* L.) STANDS[3]

[3] Sartori, F., and Markewitz D. To be submitted to Soil Biology and Biochemistry.

Abstract

Estimates of soil N availability are important both for predicting pine tree growth and for making environmentally and economically sound fertilizer recommendations. The objectives of this study were to: i) investigate the relationship between indices of N availability estimated via long term laboratory incubations and the *in situ* mixed bead resin core technique, ii) identify the effects of a continuous annual N fertilization on the quality of the light fraction soil organic matter (SOM), and iii) define possible relationships between estimates of N availability and the quality of the light organic fraction matter. The effect of fertilization (F) under conditions of complete competing vegetation control (H) was investigated using twelve paired plots, 2 H plus 2 HF (Herbicide plus Fertilizer) in three different locations in the Piedmont of Georgia on Hapludults, Kandiudults, and Udifluvents under loblolly pine (*Pinus taeda* L) stands. Surficial (0-10 cm depth) soil available N after three monthly (November, February, and March) and one bi-weekly (August) *in situ* incubations using the mixed bead resin core technique was correlated to the light fraction organic matter (LF) N, soil extractable N, and soil mineralazable N_t. Total N concentration in the LF was significantly higher (α=0.05) for the HF compared to the H plots. Nitrogen in the LF ranged from a minimum of 0.12 mg kg^{-1} for Eatonton under H during March to 0.29 mg kg^{-1} for Dawsonville under HF during August. There was no significant difference for LF total C ranging from 8.2-13 mg kg^{-1}. Resin extractable N was correlated to LF total N ($r=0.63$, $p<0.001$, $n=25$) but only under H treatment plots. Across treatment and by collection date there was a strong and significant correlation between field N and LF N for only one collection date (i.e. November, R^2=0.87). Continuous fertilization since stand establishment

significantly increased total N in the LF that is considered a short term sink for soil available N. Soil mineralizable N was found well correlated for all four collection dates ($0.5<R^2<0.6$) to field estimates of N availability underscoring that this laboratory technique is valuable for estimating *in situ* rates of readily available N.

Introduction

The estimation of soil nitrogen availability plays an important role in southern pine plantation management. Estimates of soil N availability are important both for predicting pine tree growth and for making environmentally and economically sound fertilizer recommendations. Soil N availability is dependent on the cycling of soil organic matter (SOM) and researchers have attempted to represent pools of SOM based on rates of decomposition to develop mechanistic models describing this relationship. (Parton et al., 1987; Hassink and Whitemore, 1997). Empirical techniques for quantifying the size of these SOM pools and the differing rates of decomposition have also been investigated to parameterize and validate these models (Hassink, 1995).

Recent studies using physical density fractionation of SOM have identified different organic pools with specific turn over rates that were validated by measures with C14 (Trumbone and Zheng, 1996). Under laboratory controlled conditions the N contained in light SOM (i.e., that SOM with a density < 2 g cm^{-3}) was found correlated to rates of N mineralization via laboratory incubations (Hassink, 1995). Other workers have also found this light SOM fraction to be related to soil N dynamics. For example, under continuous cultivation and cereal cropping Dalal and Mayer (1987) found that an observed loss of soil total N was correlated to the loss of light fraction organic matter. Similarly in a long term agricultural crop rotation experiment in Saskatchewan, Canada, the amount of SOM light fraction, and its N and C contents, were found positively correlated to microbial biomass N and potentially mineralizable N (Janzen et al., 1992).

Potentially mineralizable N (Stanford and Smith, 1972) is typically referred as the amount of nitrogen released from the active soil N fraction due to the activity of the bacterial population (Cabrera et al., 1994). This laboratory measured quantity differs from *in situ* studies of soil N availability because it does not account for the turnover of relevant organic pools such as the fine root biomass and is not influenced by the daily and seasonal patterns of soil moisture and temperature. Finding relationships between SOM, N availability, and growth, as sometime demonstrated for agricultural crops (e.g. Walley et al., 2002), is more difficult in forest ecosystems because the N cycle is more complex (Keeney, 1980). Long lived forest tree crops may recycle N internally obscuring relationships with soil available N and extensive root systems may access N from deep soil layers (i.e., >50 cm) again making relationships with surface soil N availability difficult to detect.

The aim of this work is to integrate previous results related to *in situ* N mineralization studies (Sartori, 2004) with laboratory measurements. One of the earlier findings indicated that continuous annual fertilization since stand establishment had the effect of increasing field estimated N mineralization rates by about 100-fold in fertilized plots compared to non-fertilized plots during relatively warm and wet months when conditions for mineralization were most favorable. Such high levels of exchangeable NO_3-N and NH_4-N were mainly a consequence of the large amount of readily available fertilizer applied in early spring since stand establishment fifteen or thirteen years ago. Given these previous results the specific objectives of this study were: i) investigate the relationship between indices of N availability estimated via long term laboratory incubations and the *in situ* mixed bead resin core technique, ii) identify the effects of a continuous annual N fertilization on the quality of the light fraction soil organic matter (SOM), and iii) define possible relationships between estimates of N availability and the quality of the light organic fraction matter.

Materials and Methods

This work considered six paired plots comprised of herbicide treatments and herbicide plus fertilizer treatments in three different locations throughout Georgia. These plots are part of a broader set of experimental treatments in the Consortium for Accelerated Pine Productivity Studies (CAPPS) arranged in a randomized complete block design that also include control and fertilizer treatments (Borders and Bailey, 2001). The influence of herbicide and continuous fertilization treatments was analyzed based on the corresponding soil potentially mineralizable nitrogen, *in situ* N mineralization rates using the mixed bead resin core technique, soil extractable N, and the quantity and quality of the soil organic matter light fraction.

Plot locations included Whitehall Forest in Athens (33° 57' N, 83° 19' W), the B.F. Grant Forest near Eatonton (33° 20' N, 83° 23' W), and plots in Dawsonville (34° 21' N, 84° 08' W). The stands in Dawsonville were treated somewhat differently than Athens or Eatonton because tree growth and survival in some of the fertilized plots was compromised by the strong understory competing vegetation. These fertilized plots were abandoned in year six and fertilizer applications were ceased. This cessation also affected the HF plots measured in this study.

The stands in Athens and near Eatonton were similar in soil type containing Cecil (fine, kaolinitic, thermic Typic Kanhapludults), Pacolet (fine, kaolinitic, thermic Typic Kanhapludults), or Madison (fine, kaolinitic, thermic Typic Kanhapludults) series (USDA-SCS, 1968). These soils also reflect in many cases, severe erosion related to the cotton farming era. The site near Dawsonville is located on a floodplain with Congaree soil (fine-loamy, mixed, active, nonacid, thermic Oxyaquic Udifluvents, USDA-SCS, 1968). Textural data by location and treatment as described below are presented in Table 4.1.

The following two treatments were applied to randomly chosen plots: the herbicide treatment (H) consists of a continuous control of all herbaceous and woody competing vegetation during the entire life of each stand, using sulfomethuron metyl during early spring of the first three growing seasons following planting and application of glyphosphate thereafter as needed (Borders at al., 2004). The herbicide treatment consisted of a broadcast of sulfomethuron methyl during early spring of the first three growing seasons following planting and subsequent application of glyphosphate as needed in mid-summer each year thereafter. The fertilizer treatment consisted of 280 $kg{\cdot}ha^{-1}$ DAP plus 112 $kg{\cdot}ha^{-1}$ KCl in the spring and 56 $kg{\cdot}ha^{-1}$ of NH_4NO_3 mid-summer during the first two growing season. In subsequent growing seasons: 168 $kg{\cdot}ha^{-1}$ NH_4NO_3 early to mid-spring. At age 11 336 $kg{\cdot}ha^{-1}$ NH_4NO_3+ 140 $kg{\cdot}ha^{-1}$ triple super phosphate. At age 12, 560 $kg{\cdot}ha^{-1}$ super rainbow (10-10-10) with micronutrients +168 kg ha^{-1} NH_4NO_3 early spring. At age 13 forward 336 $kg{\cdot}ha^{-1}$ NH_4NO_3 early spring.

Mixed bead resin core technique

Field mineralization data for this study have already been presented by Sartori (2004). Briefly these methods included 17 collections (10 monthly plus 7 bi-weekly) in 12 plots (two H and two HF at each location) between July 2002 and September 2003. Collections were made using 3.5 cm internal diameter PVC tubes placed to 10 cm depth with a resin bag at the bottom of each tube to capture the leaching solution. After collection, resin bags were extracted with 1 M KCl, which were later analyzed for NO_3-N and NH_4-N.

During each resin bag collection date the soil core internal to the PVC tube was also collected and composited by plot. These internal core samples were returned to the laboratory, air dried, and passed through a 2-mm sieve prior to further analysis. Analyses included extractable NO_3-N and NH_4-N in 2M KCl measured on an Alpkem auto-analyzer (OI Analytical, College Station, TX) using the cadmium reduction method and the indophenol blue method, respectively (Mulvaney, 1996). Soil total C and total N were also measured after pulverization with a dry combustion technique using a CE Elantech CNS analyzer (Bremmer and Mulvaney, 1982).

Soil organic matter fractionation

In this work the fractionation of soil core samples internal to the PVC tubes was performed on the six paired plots (six H plus six HF plots) and on one additional H plot from Dawsonville. Four dates of the composite soil core samples collected on November 26, 2002, February 26, 2003, March 2, 2003, and August 15, 2003 were analyzed. These soil samples (N=48) from the PVC cores were air-dried and fractionated, in replicate, into two density fractions using sodium polytungstate (SPT, Sometu-USA, CA) using a variation of the method proposed by Trumbore et al. (1996) and Golchin et al. (1994). Five grams of soil was placed in a 50 ml centrifuge tube with approximately 25 ml SPT mixed to $\rho = 2$ g cm^{-3}. The tubes containing soil-SPT mixture were shaken on a reciprocating shaker at 300 RPM for 15 minutes and then sonified for 5 minutes with 200 Watts power output and 20 kHz+500Hz frequency (Model 250, Branson Sonic Power Company, Danbury, CT, USA). Material that adhered to the centrifuge tube walls was moved back into suspension with a spatula. The suspensions were allowed to stand for 30 minutes prior to centrifugation to prevent mechanical occlusion of light fraction particles. Suspensions were then centrifuged at 2000 RCF for 60 minutes. The supernatant with the separated material was poured onto Millipore AP20 glass-fiber filters that were pre-dried for 48 hours at 65° C and pre-weighed, and filtered under vacuum. Particles that had adhered to the walls of the tubes were washed onto the filter paper. All material collected on the glass fiber filter paper was washed with at least 500 ml of deionized H_2O. The filter and the deposited material were then dried for 48 hours at 65° C, weighed, and the light fraction (LF) weight was computed by difference. The light fraction total C and N were measured on a CE Elantech NA2100 (Lakewood, NJ, USA) after grinding in a mortar and pestle.

Soil potentially mineralizable nitrogen

During May 2003 twenty 0-15 cm depth soil core samples were collected from each of 12 plots, 2 H and HF pairs in each of the three study locations. A stainless steel probe (diameter: 2.5 cm; length 60 cm) with removable internal liners and slide hammer was used to extract cores and all cores were combined in the same plastic bag during collection. Samples for chemical analysis were returned to the laboratory, air dried, and passed through a 2-mm sieve prior to analysis.

Soil potentially mineralizable nitrogen was estimated in the laboratory following the same procedure reported by Maimone et al. (1991) who modified Stanford's and Smith's method (1972). Incubation tubes were constructed using 3.5-cm i.d. polyvinyl chloride (PVC) plastic pipe. The filter tube had at the base a set of internal overlapping filters: one 5.0 µm pore size nylon filter membrane (bubble point pressure of 0.034MPa) supported by a porous polyethylene plastic disk; one 1.5-µm pore size glass-microfiber filter on the top of the nylon membrane; and a 5-mm layer of 0.85-mm acid washed ignited silica sand to act as a pre-filter. Each incubation tube was filled with a soil-sand mixture consisting of 40 g of sample soil combined with 40 g of 0.85-mm acid-washed ignited silica sand. Sand was added to provide better percolation of leaching solutions. After filling the tube a 1-cm thick plug of glass wool was placed on top of the soil-sand mixture to prevent disturbance of the soil during the addition of leaching solution.

Thirty-six samples (i.e., 12 plots x three replicates for each soil sample) were leached with three 50-ml volumes of 0.01 *M* $CaCl_2$-H_2O solution to extract NH_4-N and NO_3-N from the sand-soil mixture under low pressure. Tubes were then leached with one 25-ml volume of a nutrient solution lacking N to restore nutrient status in the soils. The composition of the ”minus N” nutrient solution was: 0.002 M $CaSO_4 \cdot 2H_2O$, 0.002 M $MgSO_4 \cdot 7H_2O$, 0.01 M KH_2PO_4, and 0.0025 M K_2SO_4. This solution is slightly different from the one used by Stanford and Smith (1972) in that 0.01 M KH_2PO_4 was used instead of 0.005 M $Ca(H_2PO_4)_2 \cdot H_2O$ because of solubility problems.

During the extraction, tubes were sealed with a rubber stopper and maintained under a constant positive pressure of 0.033 MPa, forcing the extractant or nutrient solutions through the soil-sand mix and out through the nylon membrane until no leachate was longer being removed. The volume of the leacheate being removed was measured only during the first extraction to verify complete removal of added solution and to adjust for minor variation in soil moisture retention. Leachates were analyzed for NH_4-N and NO_3-N concentrations colorimetrically on a segmented flow Technicon Autoanalyzer (U.S. Environmental Protection Agency, 1983a; 1983b). Tubes were extracted five times after 7, 21, 35, 63, and 98 days incubation start. In between extraction days tubes were incubated at 27 ºC and 99% relative humidity.

Statistical analyses

The effect of treatment, block, time, and the interaction between time and treatment were tested in SAS PROC MIXED (Wolfinger and Chang, 1995) using a split-plot design for repeated measures with four sampling dates (i.e., November, February, March, and August) on the soil organic light fraction C and N. The effect of fertilization on soil potentially mineralizable N curve parameters, N_0 and the corresponding mineralization rate constant k, was tested using a multilevel nonlinear mixed effects model based on the above experimental design. The S-Plus NLME function was used following the model building strategies and methods proposed by Pinheiro and Bates (2000) for repeated measured and clustered data. A cumulative first order kinetics model or the one-pool model form as described by Cabrera and Kissel (1994) had the fixed parameters N_0 and k set to vary as a function of location (Athens, Dawsonville, and Eatonton), block (two blocks at each location), and treatment (H or HF) to test their significance in modeling cumulative soil potentially mineralizable N. The NLME modeling approach is an alternative offered by recent software developments to model large data sets accounting for temporal autocorrelation and imposing more specific error and random effects covariance structures (Pinheiro and Bates, 2000).

Simple correlation using SAS PROC CORR (SAS Institute Inc., 1990) was used to test the relationships between variables measured on the air-dried soil core samples collected for this study (i.e., extractable nitrogen, soil total C and N, and C/N ratio, soil organic light fraction weight, total light fraction C and N, and C/N ratio). The total cumulative amount of soil mineralizable N was used as an estimate of soil potentially mineralazable nitrogen (N_0) and was regressed versus soil available N estimated using the mixed bead resin core technique for each collection date.

Results

Mixed bead resin core technique

These data are a subset of results already presented in previous work related to soil-site productivity studies in loblolly pine plantations in the Piedmont of Georgia (Sartori, 2004). Sartori (2004) found that monthly rates of N availability under the herbicide treatment were significantly higher in fertilized plots compared to the corresponding plots in absence of fertilization. In this study the same data are used for comparison to other laboratory techniques for the estimation of N availability or N pool sizes.

For the four dates estimated resin extractable soil available NO_3-N (Table 4.2), was on average the highest and the lowest overall in Athens during November (~1100 µg g^{-1} resin) within the HF plots and in August (~3 µg g^{-1} resin) within the H plots, respectively. Resin extractable soil available NH_4-N, was on average the highest in the HF during March (~97 µg g^{-1} resin) in Eatonton and the lowest in the H plots for November (~0.5 µg g^{-1} resin) in Dawsonville. The variability associated with the resin technique was relatively low for both the H and HF plots. The total cumulative amount of NO_3-N plus NH_4-N was on average the highest overall in Athens for the HF plots during November (~1113 µg g^{-1} resin) and lowest for the H plots during August (~3 µg g^{-1} resin). The HF plots were significantly different from the corresponding H plots using a split plot design for repeated measures considering 13 additional collection dates (Sartori, 2004).

Soil organic matter fractionation

The mass of soil organic matter light fraction did not show any specific pattern with sampling date, treatment or location (Table 4.3). A minimum value was observed in Dawsonville for the HF treatment during March (0.115 g) and a maximum value in the same location under the H treatment during February (0.188 g). Similarly total C was relatively constant across sites and treatment ranging between 8.2 and 13 mg C kg $soil^{-1}$. Values for all four collection dates were slightly lower in Dawsonville but not statistically significant. To the contrary total N in the light fraction differed significantly between the H and HF plots for all three locations (Table 4.4). Nitrogen ranged from a minimum of 0.12 mg N kg $soil^{-1}$ in the

Eatonton H during March to a maximum of 0.29 mg N kg soil^{-1} in the Dawsonville HF treatment during August. In general the light fraction N was always higher in the HF plots and as a consequence the corresponding C to N ratio was lower for the HF ranging between 33 in Dawsonville HF treatment during February to 67 in Eatonton H treatment during March.

The corresponding averages across treatment and sampling date as % C and % N in the soil organic matter light fraction are presented by location in Figure 4.1. Overall C was slightly lower in the H treatment in particular for the Dawsonville site (~31 %) and was the highest for Athens and Eatonton HF treatments (~36 %). Percent N had a larger difference between the H and HF in all three locations ranging from ~0.5 to 1%. As before the corresponding C to N ratio was higher under the H than the HF treatment.

Soil potentially mineralizable nitrogen

The range in mineralizable nitrogen (N_t) after 98-day incubation had similar variability in both the H and HF plots (data not shown) but with smaller difference than found for the *in situ* mixed bead technique, which had values of about 100-fold higher in the HF plots compared to the H (Sartori, 2004). N_t was found to be always higher in the fertilized plots (HF) compared to the non-fertilized (H) ones (Figure 4.2). Based on the multilevel nonlinear mixed effects model only treatment had a significant effect (α=0.05) on the mineralization rate k (Table 4.5). This result underscores that treatment is a useful covariate in explaining the variability in the data and should be included in modeling efforts. Based on the model residuals and corresponding autocorrelation function plot (Figure 4.3) this model appears to be acceptable in that it has a homoskedastik residuals pattern, but should be improved considering the presence of significant autocorrelation at lag 3 and 4.

Correlations among indices of soil available N and the SOM light fraction

The highest significant correlation with field available N was found for the light fraction total N concentration (Table 4.6, $r=0.63$, $p<0.001$, $N=25$) but only under the H treatment. No similar and significant relationship was found for the corresponding HF treatment. Other high and significant correlations were present but none of them regarding independently measured variables on different samples as above. For example, soil total C and N were correlated ($r=0.77$, $p<0.001$, $N=86$) as was soil total N and extractable NO_3-N ($r=0.346$, $p<0.01$, $N=84$).

When considering the correlation between field-available N and light fraction total N across treatment and collection dates (Figure 4.4) there was a strong and significant correlation for only one collection date (i.e., November, $R^2=0.87$, regression line not shown in the graph). A log transformation of the response variable soil N was used to obtain approximately equal variances because the HF plots had a much higher variability and values that in many cases were 100-fold higher compared to the corresponding H plots.

When considering only the relationship between soil available N and the light fraction total N within the H plots without any variable transformation there were poor but significant relationship for all collection dates (i.e., November had $R^2=0.4$; February $R^2=0.52$, March $R^2=0.12$, and August $R^2=0.82$). One constant result for all four collection dates within the H treatment plots, however, is the presence of a leverage point with both high soil available N and light fraction total N. This point always corresponds to one H plot in Dawsonville. Due to this leverage point the high and significant correlations are somewhat misleading, especially for August.

Soil mineralizable nitrogen (N_t) was found significantly correlated to soil available N estimated using the mixed bead resin core technique (Figure 4.5). The R^2 was the highest for the February collection date (R^2=0.6) and lowest (R^2=0.5) for November '02 and August '03. The use of a log scale for the November sampling date was necessary given that the HF plots, with respect to soil available N, have a much higher variance compared to the H plots. In general these regressions have specific model error residuals patterns (i.e. residuals are not independent and identically distributed) and were used only for an approximate comparison of these two techniques.

Discussion

A strong correlation between mineralizable soil N estimated in the field and total N in the light fraction across treatment and location occurred only for the November sampling date. This result suggests that there is likely interference to establishing a better relationship due to the large amount of readily available N applied with the fertilizer treatment in early spring of each year. Conversely, the consistent but weaker correlations under only the H treatment plots (e.g., r=0.63, n=25) and the corresponding regressions presented by date are similar to those found by other authors for net mineralization estimated via laboratory incubations and the soil organic matter light fraction (Hassink, 1995; Jansen, 1992; Sollins et al. 1984).

For example, Hassink (1995) working in grassland soils found that the correlation between mineralizable N and soil organic matter fractions decreased in the order light (r=0.77), intermediate (r=0.59), heavy (r=0.56) and non-macroorganic N (r=0.5). Similarly, Sollins et al. (1984) working in soils under different forest types in Oregon, Washington, and Costa Rica found that N mineralization from the light fraction, as a proportion of total N in the fraction, correlated significantly with the N content of that fraction (r=0.83). However, these same authors underscore that net nitrogen mineralization appeared to be largely driven by N not from the light fraction but from the soil heavy fraction.

To my knowledge no author has ever investigated or found significant relationships between *in situ* estimates of N availability using the mixed bead resin core technique and the quality of the soil organic matter light fraction. Moisture and temperature together with the quality of the soil organic matter are typically considered the primary drivers of soil net nitrogen mineralization (Goncalves and Carlyle, 1994). Clearly there are more moisture and temperature variations in field incubations than in the laboratory setting that may affect processes of N mineralization. For example, measures of the within plot variation amongst the PVC soil cores (Table 4.2) indicate that during the warm month of August variation relative to the standard error of the mean for both NH_4-N and NO_3-N under both the H and HF treatments was greatest. Conversely, during the cold month of February this variation was reduced likely as a consequence of lower temperatures uniformly influencing the microbial population activity. The consistently high rates of N mineralization and LF total N in the same Dawsonville H plot, however, indicate some capacity to repeatedly measure LF changes with N mineralization over time.

Changes in light fraction organic matter have been previously related to changes in N cycling. In agricultural systems Dalal and Mayer (1987) using $\rho = 2$ g cm^{-3} to separate the light fraction material, report that the amount of the light fraction increased nutrient cycling for soil under continuous cultivation and cereal cropping. These authors found that the loss of total N from different textured fractions was positively correlated to the amount of the light fraction organic C and N. In agricultural lands, the loss of the soil light fraction organic material is a well-known negative effect due to management practices (Janzen, 1987). In the current study, the fact that the fertilization treatment significantly increased total N concentration in the soil organic matter light fraction indicates a beneficial effect of this treatment that was not clearly detected by simply measuring percent soil total N (Sartori, 2004).

The C:N ratios by location and treatment (Figure 4.1) also clearly reflect the effect of fertilization. In the Dawsonville site, in particular, the C:N ratio indicates that LF for both the H and HF treatments has a relatively higher N concentration that corresponds also to relatively higher mineralization rates. Across the three locations of this study the quality of the soil LF seems to have a greater effect in governing net nitrogen mineralization compared to textural differences that may also have a relevant influence (Hassink, 1995; Van Veen and Kuikman, 1990). In fact, despite the higher clay content that could potentially reduce the LF turnover rates, the Dawsonville site (Table 4.1) has generally higher soil N availability.

The overall relationships found between soil available N and total N in the light fraction material support the findings that the light fraction material is an important short-term sink for inorganic N (Compton and Boone, 2002). Because the herbicide treatment was applied since stand establishment and plots were carefully controlled throughout the entire life of the stand the organic cycling pools due to litter and fine root decomposition depend only on loblolly pine species without any interference on the substrate quality derived from the understory vegetation. Due to the similarity in pine inputs for H and HF plots, the fact that the HF plots have a lower C to N ratio indicates that the prolonged fertilization treatment has induced significant transformation in the quality of the soil organic matter light fraction. This result is in agreement with Janzen et al. (1992), who found that fertilizer applications tended to increase the N concentration of LF when studying the effect of cropping practices.

At Harvard Forest MA, U.S.A., Compton and Boone (2002) found that added ^{15}N-ammonium and ^{15}N-nitrate were rapidly accumulated into the LF rather then the heavy fraction (HF). These findings likely explain the significant increase in total N in the LF under the yearly fertilization treatment since stand establishment in this study. Long term effects of past agricultural fertilization on intensively managed loblolly pine stands have also been shown to increase the N content of the soil organic matter (Richter et al., 2000).

The results regarding cumulative soil mineralizable nitrogen, N_t, also demonstrate an important effect of fertilization on N availability. The cumulative N_t value was found to always be higher in the fertilized plots (HF) compared to the non-fertilized (H) plots (Figure 4.4) suggesting that the continuous fertilizer additions over the years have induced changes in the substrates decomposable by the bacterial population in laboratory incubations. This result demonstrates that the inorganic fertilizers have been incorporated into the soil organic N cycle. These results agree with the findings of a factorial fertilization experiment under loblolly pine conducted by Maimone et al. (1991). These authors also found that surficial N_0 increased with N fertilization level. The N_0 values for the fertilizer treated plots very likely reflect the organic N availability in the light and other more recalcitrant organic fractions (e.g. the heavy fraction) and maybe dependent on the length of incubation (Paustian and Bonde 1987). The values estimated with the mixed bead resin core technique, instead, are assumed to be more a consequence of the readily available ammonium nitrate fertilizer application in early spring of each year as well as field temperature and moisture conditions. Regardless of these differences, estimates of N availability using the laboratory incubations (N_t) and *in situ* measurement (resin core technique) were well correlated despite not accounting for differences across location and treatment as possible confounding factors. These results support the conclusions of Maimone et al. (1991)

that long term incubations are a valuable means to assess the biologically active soil N in forest soils.

Conclusions

In loblolly pine stands under conditions of complete competition control the light fraction total N was found significantly correlated to field N mineralization estimates using the mixed bead resin core technique. Previous studies have demonstrated the significant relationship between laboratory incubations and the quality of the soil organic matter light fraction. For most collection dates these relationships were not as strong as those found by other authors under laboratory controlled conditions. The annual fertilizer application was found to drastically increase the readily available soil N and to be an important confounding factor in trying to develop these relationships. For only the November sampling date there was a strong correlation between *in situ* soil available N and the LF total N across site and treatment. Continuous fertilization since stand establishment significantly increased total N in the light fraction organic matter indicating that the LF is a short term sink for soil available N. The soil potentially mineralizable N was found to be well correlated to field estimates of N availability supporting the conclusions that this technique is a valuable means of representing *in situ* rates of readily available N.

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Table 4.1. Average soil textural composition by location and treatment (Herbicide or Herbicide plus Fertilizer) in the upper 0-10 cm mineral soil surface based on two replicate plots (n=2) in Athens and Eatonton, and one plot (n=1) in Dawsonville, Georgia. Samples were collected in March 2003.

Location	Treatment	% Sand[†]	% Silt	% Clay
Athens	H	61.4	24.7	13.9
	HF	55.5	25.6	18.9
Eatonton	H	52.8	26.5	20.7
	HF	58.8	23.3	17.9
Dawsonville	H	38.0	32.8	29.2
	HF	NA	NA	NA

[†] Soil particle size analysis was determined using the hydrometer method.

Table 4.2. *In situ* estimates of soil available NO3-N and NH4-N (mean ± 1 SE) using the mixed bead resin core technique in the 0-10 cm depth for paired H-HF plots in the three study locations, Athens, Eatonton, and Dawsonville. The mixed bead resin bags were collected from three one-month *in situ* incubations on 11/26/02 (November), 02/26/03 (February), 04/02/03 (March collection), and on 08/15/03 (August) for only one bi-weekly incubation.

		Athens				Eatonton				Dawsonville			
	Treatment	H	H	HF	HF	H	H	HF	HF	H	H	HF	HF
	Block	1	2	1	2	1	2	1	2	1	4	1	4
		ug g^{-1} resin				ug g^{-1} resin				ug g^{-1} resin			
November	NO_3-N[1]	17.6	9.4	1444.4	754.9	NA	3.3	252.7	243.5	38.1	12.3	25.2	64.7
	SE(NO_3)	0.5	0.4	54.4	50.7	NA	0.3	16.0	15.2	1.1	0.6	1.4	1.5
	NH_4-N	0.9	2.7	24.7	3.8	NA	0.9	5.9	1.9	0.5	0.8	2.5	1.2
	SE(NH_4)	0.0	0.0	2.2	0.3	NA	0.0	0.2	0.1	0.0	0.0	0.2	0.0
	[2] NO_3+NH_4	18.5	12.1	1469.1	758.7	NA	4.2	258.6	245.5	38.6	13.1	27.6	65.9
February	NO_3-N	8.5	4.0	110.9	90.4	16.7	10.4	116.1	139.3	55.3	8.9	37.8	33.8
	SE(NO_3)	0.3	0.1	1.0	1.9	0.5	0.6	1.9	6.7	1.1	0.7	0.8	1.6
	NH_4-N	6.7	4.0	6.3	11.1	8.5	2.0	4.7	6.8	3.5	1.4	2.1	1.4
	SE(NH_4)	0.2	0.1	0.1	0.3	0.6	0.1	0.1	0.5	0.3	0.1	0.0	0.1
	NO_3+NH_4	15.2	7.9	117.2	101.6	25.2	12.4	120.8	146.1	58.7	10.4	39.9	35.2

Table 4.2 (Continued)

March	NO_3-N	10.5	4.4	133.6	118.5	8.2	5.0	69.1	93.6	92.5	6.8	12.0	8.4
	SE(NO_3)	0.3	0.2	3.1	1.7	0.2	0.5	1.7	5.0	4.8	0.6	1.0	0.4
	NH_4-N	2.5	3.2	21.4	14.6	13.0	14.3	130.6	63.9	5.7	3.0	2.3	1.9
	SE(NH_4)	0.1	0.1	0.9	1.1	1.4	0.8	5.3	3.8	0.1	0.1	0.1	0.0
	NO_3+NH_4	13.0	7.7	155.1	133.1	21.2	19.3	199.6	157.4	98.2	9.8	14.3	10.3
August	NO_3-N	2.3	1.4	226.8	233.6	2.3	2.6	209.2	296.9	110.9	3.7	126.8	1.6
	SE(NO_3)	1.1	1.2	68.6	39.1	1.4	2.4	59.5	105.3	29.4	2.3	93.3	1.1
	NH_4-N	0.5	2.5	5.9	10.8	3.3	3.9	14.7	3.2	2.7	8.4	22.0	1.6
	SE(NH_4)	0.3	0.9	2.3	5.5	1.1	1.5	6.6	0.6	0.9	4.6	16.2	0.4
	NO_3+NH_4	2.8	3.9	232.6	244.4	5.6	6.6	223.8	300.0	113.6	12.1	148.8	3.3

[1] The mean value was estimated from four (n=4) bags per plot for the November collection and six (n=6) bags for the remaining collection dates.

[2] NO_3+NH_4 is the sum of averaged NO_3-N plus NH_4-N.

Table 4.3. Average weight, mass of carbon, mass of nitrogen, and the C to N ratio by location and treatment for the soil organic matter light fraction ($\rho < 2$ $g \cdot cm^{-3}$) in Athens and Eatonton (H: n=2; HF: n=2) and in Dawsonville (H: n=3; HF: n=2), Georgia. Samples for the light fraction separation were collected after one-month *in situ* incubations on 26-Nov-02, 26-Feb-03, and 2-Apr-03, and after a two-week incubation on 15-Aug-03.

Date	Location	Treat[1]	Wt	C[2]	N[3]	CN[4]
			g	——mg kg^{-1} soil——		
November	Athens	H	0.124	9.3	0.18	52
		HF	0.131	10.0	0.26	38
	Eatonton	H	0.185	13.0	0.26	49
		HF	0.152	10.8	0.26	41
	Dawsonville	H	0.147	8.7	0.22	40
		HF	0.142	9.5	0.25	38
February	Athens	H	0.133	9.4	0.19	51
		HF	0.162	11.2	0.32	35
	Eatonton	H	0.149	10.3	0.20	53
		HF	0.137	10.1	0.22	46
	Dawsonville	H	0.188	9.8	0.26	38
		HF	0.135	8.7	0.25	35
March	Athens	H	0.123	8.9	0.19	47
		HF	0.118	8.9	0.23	40
	Eatonton	H	0.116	8.0	0.12	67
		HF	0.120	8.7	0.18	47
	Dawsonville	H	0.122	9.0	0.20	44
		HF	0.115	8.2	0.22	36
August	Athens	H	0.140	10.0	0.17	58
		HF	0.138	10.3	0.25	41
	Eatonton	H	0.125	9.1	0.17	55
		HF	0.130	10.0	0.22	45
	Dawsonville	H	0.128	8.6	0.22	39
		HF	0.136	9.6	0.29	33

[1] Treatment: H, continuous herbicide control throughout the entire stand life. HF, combination of herbicide and fertilization treatment.
[2] Mass of C (mg) in the light fraction per kg of soil.
[3] Mass of N (mg) in the light fraction per kg of soil.
[4] Carbon to Nitrogen ratio

Table 4.4. Type III test for fixed effects using SAS PROC MIXED (Wolfinger and Chang, 1995) on the soil organic light fraction C and N concentration. The effect of treatment, block, time, and the interaction between time and treatment were tested in a split-plot design for repeated measures with four sampling dates (i.e. November, February, March, and August). Carbon and Nitrogen were significantly correlated. C and N were analyzed as independent variables assuming a significance level of α=0.05.

	Athens		Eatonton		Dawsonville	
	Carbon	Nitrogen	Carbon	Nitrogen	Carbon	Nitrogen
	p-value		p-value		p-value	
Treat	0.5883	0.0011	0.2421	0.0515	0.0845	0.0235
Time	0.5798	0.3633	0.5837	0.5856	0.2313	0.7896
Treat×Time	0.9624	0.8965	0.9858	0.9128	0.444	0.943
Block	0.0692	0.884	0.2268	0.8554	0.0139	0.0208

Table 4.5. Testing the significance on the fixed effects parameters N0 and k of location and treatment using a multilevel nonlinear mixed effects model with a banded correlation structure of order two for the random effects specific for location and plot (Pinheiro and Bates, 2000). The one-pool model form was used to fit cumulative N0 data grouped by plot and location. When using this modeling approach it is possible to express the fixed parameters N0 and k as a linear function of useful covariates (i.e. location and treatment) that explain part of the variability present in the data.

One pool model parameters		Value	Std.Error	DF	p-value
N_0	Intercept	50.76625	12.69801	43	0.0002
	Location	3.4688	5.81864	43	0.5542
	Treatment	4.38418	5.87773	43	0.4598
k	Intercept	0.03702	0.01309	43	0.0071
	Location	-0.00742	0.00567	43	0.1976
	Treatment	0.02424	0.00759	43	0.0026

Table 4.6. Correlation matrix for resin and extractable available N, soil C, N, C to N ratio, light fraction SOM mass, light fraction C concentration, light fraction N concentration, and light fraction C to N ratio measured on soil samples from plots that received Herbicide treatment. Soils were collected in Athens (2 H plots), Eatonton (2 H plots), and Dawsonville (3 H plots), Georgia. All plots were repeatedly measured 17 times for soil available N using the mixed bead resin core technique and at each collection date a composite soil sample derived from the internal cores of the PVC tube was collected. SOM light fraction was separated on the soil samples using a sodium polytungstate solution ($\rho = 2$ g cm^{-3}). The measured light organic matter fraction variables correspond to only four collection dates: three after one-month *in situ* incubations on 26-Nov-02, 26-Feb-03, and 2-Apr-03, and one after a two-week incubation on 15-Aug-03. Measured soil variables were correlated across a larger number of samples collected on the following additional dates: 23-Sep-02, 23-Oct-02, 20-Dec-02, 20-Jan-03, 2-May-03, 2-Jun-03, 16-Jun-03, and 18-Jun-03.

	AVN[1]	STOTC[2]	STOTN[3]	SNO_3[4]	SNH_4[5]	SEXTRN[6]	LFWT[7]	LFTOTC[8]	LFTOTN[9]	SCN[10]	LFCN[11]
AVN	1	0.38851†	0.46669	0.30671	-0.07712	-0.03292	0.0821	0.12528	0.63026	-0.25243	-0.36966
		0.0002‡	<.0001	0.0045	0.4803	0.7649	0.6964	0.5507	0.0007	0.019	0.069
	114	86§	86	84	86	85	25	25	25	86	25

Table 4.6 (Continued)

STOTC	1	0.76804	0.346	-0.13236	0.02806	0.32218	-0.10962	0.24822	-0.00535	-0.14093
		<.0001	0.0013	0.2244	0.7988	0.1786	0.6551	0.3055	0.961	0.565
	86	86	84	86	85	19	19	19	86	19
STOTN		1	0.59257	-0.17192	0.02276	0.33409	-0.40955	0.29097	-0.63528	-0.40261
			<.0001	0.1135	0.8362	0.1621	0.0816	0.2268	<.0001	0.0875
		86	84	86	85	19	19	19	86	19
SNO_3			1	-0.1155	0.38159	-0.0008	-0.21052	0.40274	-0.47953	-0.44172
				0.2955	0.0004	0.9974	0.387	0.0873	<.0001	0.0583
			84	84	83	19	19	19	84	19
SNH_4				1	0.81634	0.14956	0.10793	-0.09105	0.10207	0.11969
					<.0001	0.5411	0.6601	0.7109	0.3497	0.6255
				86	85	19	19	19	86	19
SEXTRN					1	0.159	0.0378	0.05063	0.00554	-0.03441
						0.5156	0.8779	0.8369	0.9599	0.8888
					85	19	19	19	85	19
LFWT						1	-0.46776	0.05553	-0.16426	-0.36368
							0.0184	0.7921	0.5016	0.0739
						25	25	25	19	25
LFTOTC							1	0.41656	0.53038	0.1494
								0.0383	0.0195	0.476
							25	25	19	25

Table 4.6 (Continued)

LFTOTN	1	-0.18044	-0.7635
		0.4598	<.0001
	25	19	25
SCN		1	0.44988
			0.0533
		86	19
LFCN			1
			25

1 AVN = Soil available N estimated using the mixed bead resin core technique at 0-10 cm depth .
2 STOTC = Soil total C determined by dry combustion after pulverization..
3 STOTN = Soil total N determined by dry combustion after pulverization..
4 SNO3= Soil extractable NO3 determined colorimetrically after extraction with a 2 M KCl solution.
5 SNH4= Soil extractable NH4 determined colorimetrically after extraction with a 2 M KCl solution.
6 SEXTRN = Soil extractable NO3-N plus NH4-N.
7,LFWT = Light fraction weight after fractionation of 5 g-air dried soil sample.
8,LFTOTC = Light fraction total C determined by dry combustion after pulverization.
9 LFTOTN = Light fraction total N determined by dry combustion after pulverization.
10 SCN = Soil C to N ratio.
11 LFCN = Light fraction C to N ratios.
† Correlation coefficient r between the considered variables.
‡ Corresponding p-value for r.
§ Sample size.

Figure legends

Figure 4.1. Average concentration of total C and N, and the corresponding C to N ratio by location and treatment for the soil organic matter light fraction ($\rho < 2$ g cm^{-3}) separated using sodium polytungstate. All locations in Georgia contained two paired Herbicide (H) and Herbicide plus Fertilizer (HF) plots. Composite soil core samples by plot were collected after one-month *in situ* incubations on 26-Nov-02, 26-Feb-03, and 2-Apr-03, and after a two-week incubation on 15-Aug-03.

Figure 4.2. Cumulative soil mineralizable N (NO_3-N plus NH_4-N) versus incubation time for paired H and HF plots in Athens, Eatonton, and Dawsonville, Georgia. Soils from 0 to 15 cm were collected in March 2003 and incubated in polyvinyl chloride (PVC) tubes at constant temperature of 27 °C and 99% relative humidity. Extraction were collected after 7, 21, 35, 63, and 98 days.

Figure 4.3. Diagnostic residual and autocorrelation function plots of the nonlinear mixed effects model to test the effect of treatment on the one pool cumulative model parameters, potentially mineralizable N (N_0, ug g^{-1}) and decomposition rate constant k (day^{-1}).

Figure 4.4. Plot of resin N estimated in the field using the mixed bead resin core technique and soil organic matter light fraction total N (%) for the Herbicide (H) and Herbicide plus Fertilizer (HF) plots. Composite soil core samples by plot were collected after one-month *in situ* incubations on 26-Nov-02, 26-Feb-03, and 2-Apr-03, and after a two-week incubation on 15-Aug-03. Plots were located in Athens, Eatonton, and Dawsonville, Georgia.

Figure 4.5. Comparison between the laboratory incubation method for cumulative mineralizable nitrogen (N_t) on soil samples collected once in May 2003 and *in situ* extractable N based on the mixed bead resin core technique for paired Herbicide and Herbicide plus Fertilizer plots in Athens, Eatonton, and Dawsonville, Georgia. Resin bags were collected after three one-month *in situ* incubations on 11/26/02, 02/26/03, 04/02/03, and on 08/15/03 for only one bi-weekly incubation.

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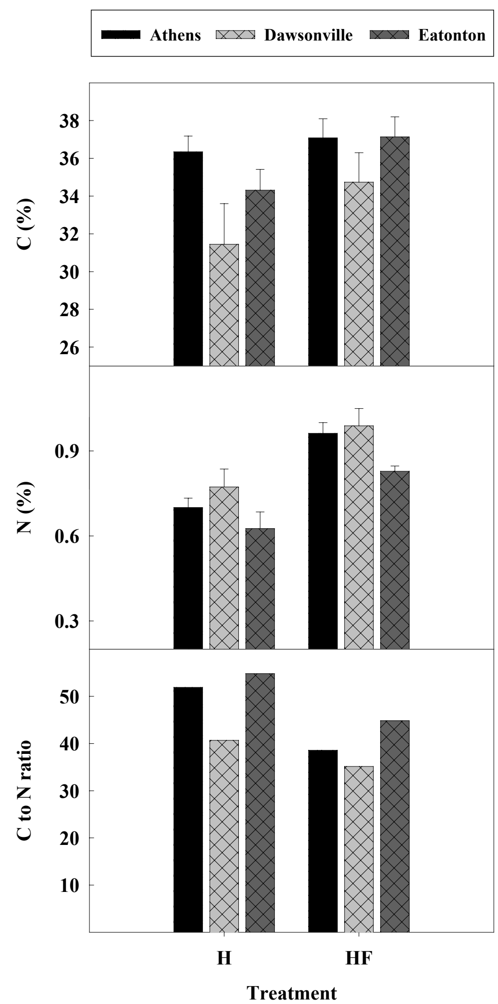

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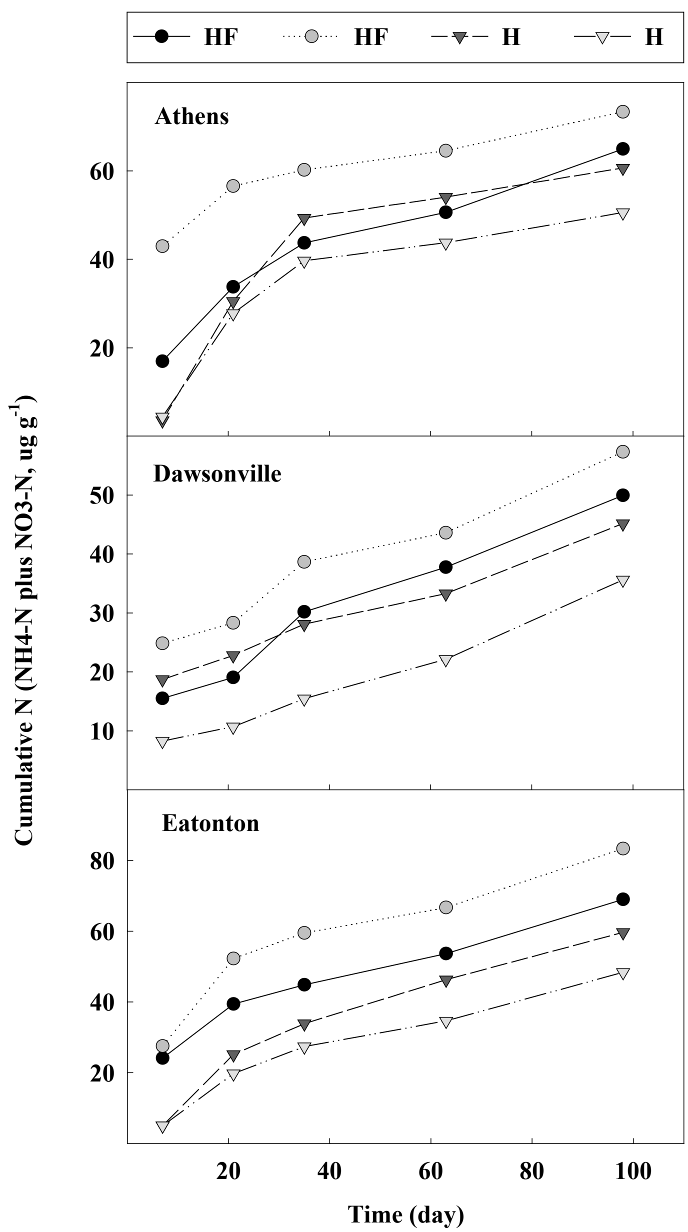

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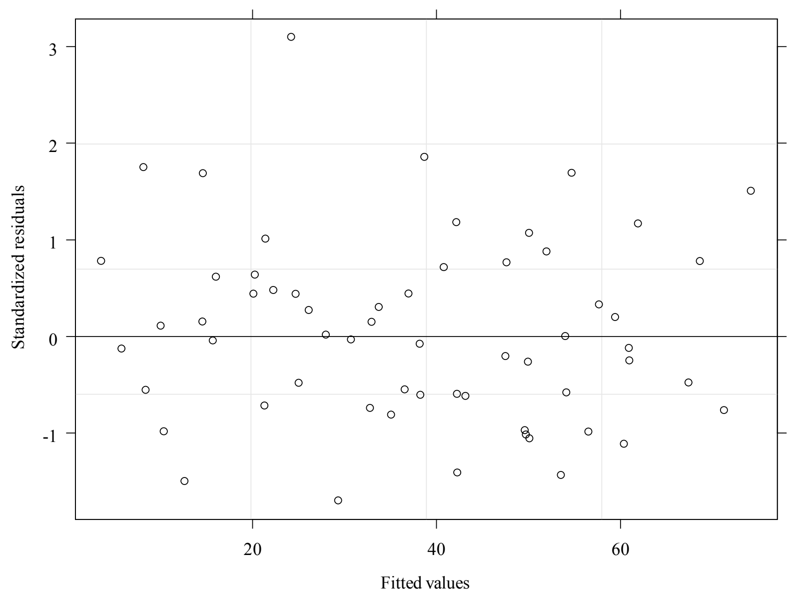

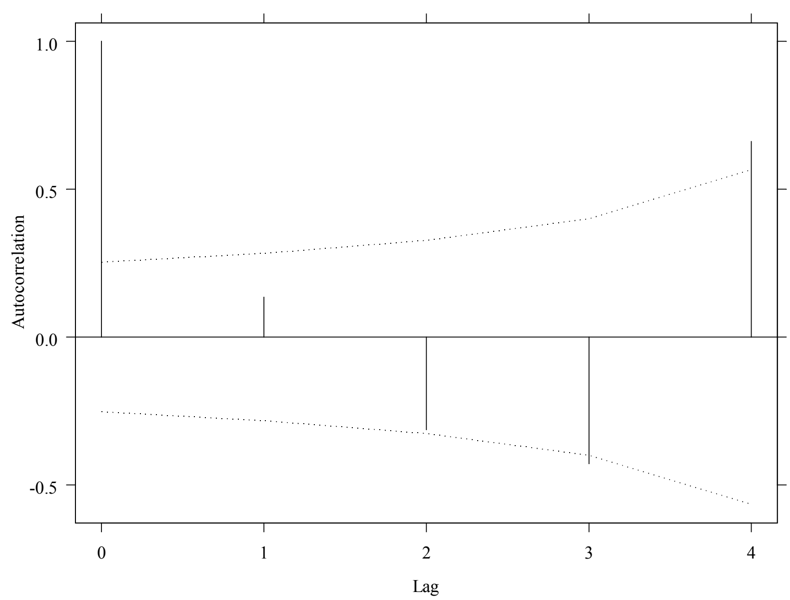

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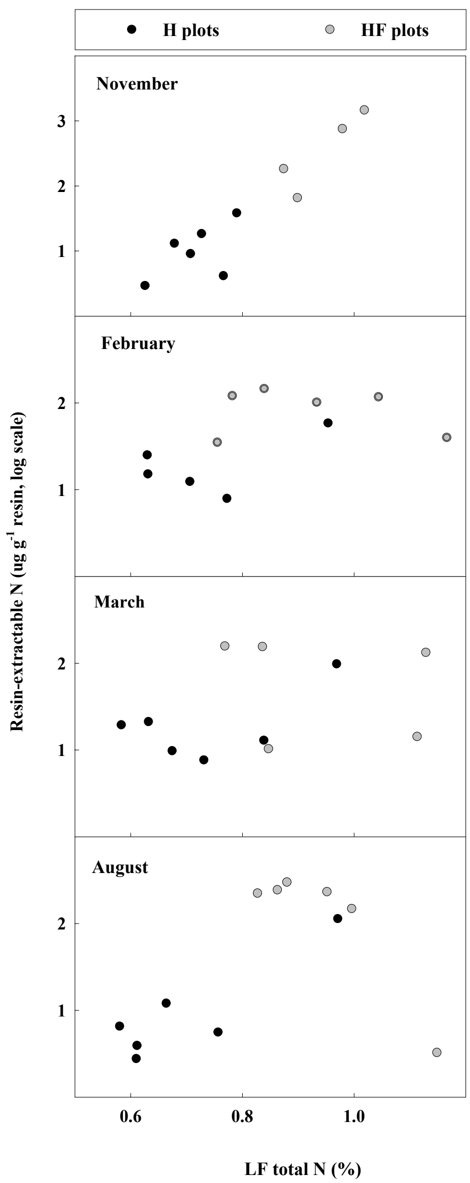

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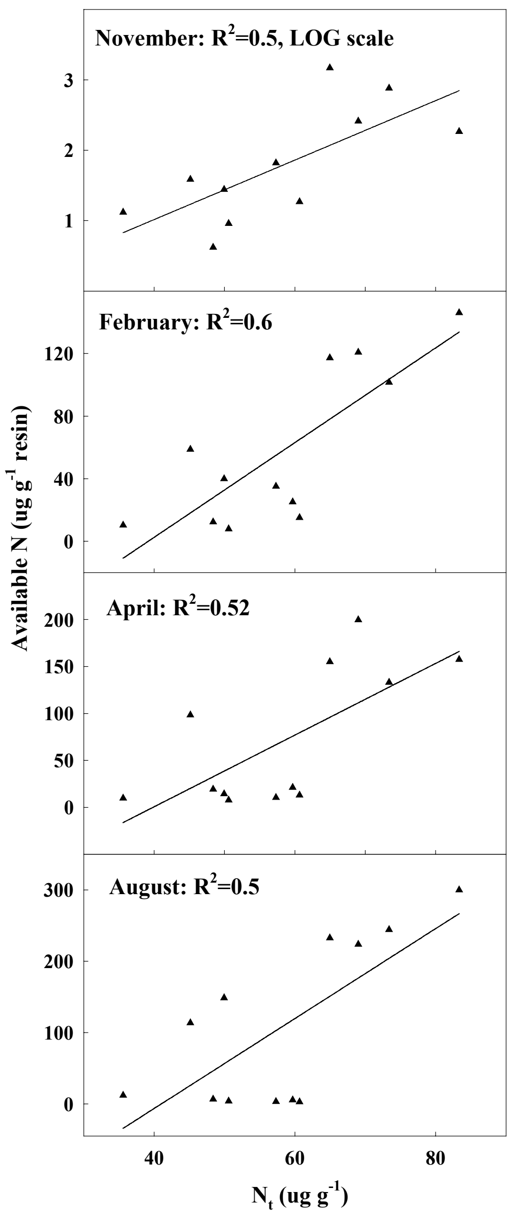

CHAPTER 5

SUMMARY

The major findings of this work have been described in Chapter 2 and are the significant relationship between field estimates of N availability and mean annual increment under conditions of complete competing vegetation control. For 13 of 17 sampling dates during my investigation significant relationships were found between mean annual increment and soil available N underscoring the validity of these results.

The soil nutrient status analysis reported in Chapter 2 supports the claim that soil available N is likely the most important limiting nutrient in these loblolly pine stands. The mixed bead resin core technique could detect major differences between fertilized and non-fertilized plots. Continuous fertilization that extended during the entire life cycle of each stand caused resin extractable N to be on average 10- to 100-fold greater in the fertilized plots than in the corresponding plots without any fertilizer treatment.

The significant direct correlation found between mean annual increment and resin extractable N (R^2=0.7) likely depends partly on the relatively young age (i.e., <17 years old) of the considered stands since nutrient demand is the great and N is allocated rapidly accumulating foliar and stem biomass. Plots that had high values of soil available N also had high growth rates and viceversa.

In these simplified pine ecosystems soil available N was used to model tree height growth with a data based modeling approach new to this field of studies. A multilevel nonlinear mixed effects model (NLMM) was used to test whether including N as a covariate would offer a significant improvement to growth and yield modeling efforts. Based on this modeling approach soil N was a useful covariate to be included in this type of growth and yield models and offered a finer characterization than simply considering the categorical distinction between fertilized and non-fertilized plots.

One possible limitation of this study is that mean annual increment is an approximate average growth rate during the entire stand life, while soil available N refers to a specific year in the life span of these plantations without including any similar long term history information. Another important limitation is that the NLMMs can be applied only under simplified conditions in the presence of one or a few variables related to growth and in the absence of relevant confounding factors as the understory competing vegetation.

In Chapter 3 conclusions regarding nutrient availability and tree growth in the CAPPS experiment were drawn based on nutrient pools and atmospheric N flux measures from this study and already existing information regarding growth. As found in previous studies, N and P were the major drivers of growth for the stands in Athens and Eatonton on relatively nutrient poor Hapludults. No other measured soil nutrients were found to have a similar significant difference and regular pattern across treatment as observed for available N and P on these nutrient limited Hapludults. When such nutrient limitations did not occur, as on the richer floodplain site on Udifluvents in Dawsonville, it was not possible to find such clear correspondence between the levels of soil nutrient pools and N availability and the response to fertilization and herbicide treatment in the above ground biomass.

Other major findings of this work are related to the influence of herbicide and fertilizer application on soil carbon. In all three locations the herbicide treatment always reduced soil C (although not always statistically so) and as a consequence ECEC also reduced. For most sites the fertilization treatment did not increase soil C content suggesting that such treatment could not buffer and contrast the negative effects of the herbicide treatment on soil C.

In chapter 4 it was demonstrated that in loblolly pine stands under conditions of complete competition control the light fraction total N was significantly correlated to field N availability estimates using the mixed bead resin core technique. Previous studies have demonstrated significant relationships between laboratory incubations and the quality of the soil organic matter light fraction. For most collection dates in this study these relationships were not as strong as those found by other authors under laboratory controlled conditions. In the current case, the annual fertilizer application was found to drastically increase the readily available soil N and to be an important confounding factor in trying to develop these LF-soil available N relationships. For only the November sampling date was there a strong correlation between in situ soil available N and the LF total N across site and treatment.

It was well demonstrated, however, that continuos fertilization since stand establishment significantly increased total N in the light organic matter fraction indicating that the LF is a short term sink for soil available N in agreement with other previous studies. The soil potentially mineralizable N was found well correlated to field estimates of N availability supporting conclusions that this technique is a valuable means in representing in situ rates of readily available N.