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THE EFFECT OF MID-ROTATION FERTILIZATION ON THE WOOD PROPERTIES OF LOBLOLLY PINE (*PINUS TAEDA*)

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SUMMARY

Mid-rotation fertilization is a common practice in the management of loblolly pine (*Pinus taeda* L.) plantations, typically providing large improvements in growth. However, concerns exist about the quality of wood produced following fertilization. The objective of this study was to develop an understanding of wood property changes following fertilization. Wood samples from a study involving four levels of fertilization applied to a thinned mid-rotation loblolly pine plantation located on the lower coastal plain of North Carolina were sampled. The study was laid out in a randomized complete block design involving four blocks and four levels of nitrogen fertilizer: Control-000, 112, 224 and 336 kg/ha, along with 28 kg/ha of phosphorus with all treatments. Thirty-two trees were felled and disks were cut at five heights from each tree. Wood properties including modulus of elasticity, air-dry density and tracheid anatomical properties were measured for each of the three post-fertilization annual growth rings using near infrared (NIR) spectra obtained from the radial face of strips cut from the disks. An analysis of variance was conducted on three-year basal area weighted average stiffness, air-dry density, and tracheid anatomical properties. A decrease in stiffness, air-dry density, tracheid wall thickness, and an increase in tracheid radial diameter were observed for the heaviest fertilizer treatment (336 kg/ha) compared to the control and 112 kg/ha of nitrogen. Microfibril angle (MFA), cell tangential diameter, and tracheid perimeter showed little change. Wood properties of trees receiving fertilizer rates of 112 and 224 kg/ha were not significantly affected.

Key words: Loblolly pine, mid-rotation fertilization, modulus of elasticity, *Pinus taeda*, NIR spectroscopy.

INTRODUCTION

The southern region of the United States of America (USA) supplies approximately 58 percent of the total wood used in the USA and 16 percent of wood supplied to the world

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timber market (Wear & Greis 2002). The primary species in this region is loblolly pine (*Pinus taeda* L.) which occupies approximately 31.3 percent of the total land area of the southern USA (USDA FS-FIA 2002). Its rapid growth, ability to survive on a wide range of sites, and the suitability of its wood for pulp and paper, sawn lumber and composite wood products has made loblolly pine the leading plantation species in the southern USA.

Mid-rotation fertilization following thinning has proven to be a successful practice in southern pine plantation management with growth improvements in the range of 3.5–6.9 m^3/ha/year (Fox *et al.* 2006). An increase in the area of fertilized pine plantations (from 81,000 ha in 1997 to 0.6 million ha in 2002), indicates the rapid adoption of this practice in the management of southern pine plantations (Forest Nutrition Cooperative 2006). The increased volume production of pine plantations following mid-rotation fertilization and thinning was attributed to the removal of nutrient limitations from the site and a subsequent increase in light interception capacity (facilitated by an increase in the leaf area of fertilized trees) (Fox *et al.* 2006).

The potential of mid-rotation fertilization to change the mechanical, physical and anatomical properties of wood produced in the post-fertilization period has raised concerns within the forest products industry. Studies examining the effects of mid-rotation fertilization on wood properties have mainly been confined to specific gravity because of its ease of measurement and strong associations with the strength properties of solid wood and pulp yield (Cown & McConchie 1981; Nyakuengama 1991; Morling 2002). However, industries are also interested in understanding the changes in mechanical, physical and anatomical properties, other than specific gravity, following mid-rotation fertilization. Modulus of elasticity (MOE), a measure of wood deformation under an applied load, is a property of particular interest to the lumber industry. The changes to tracheid anatomical properties such as MFA, tracheid wall thickness, and tracheid radial diameter are also of interest, and can provide a meaningful explanation of the changes observed in specific gravity and MOE following mid-rotation fertilization. Various studies have examined the effect of fertilization on wood properties (specific gravity, MFA and tracheid wall thickness) of different species, especially Douglas fir (*Pseudotsuga menziesii* (Mirbel) Franco) and radiata pine (*Pinus radiata* D. Don), and have reported changes in these properties following fertilization (Erickson & Harrison 1974; Downes *et al.* 2002; Nyakuengama *et al.* 2003, 2004).

Studies evaluating changes in wood properties following different silvicultural treatments have been restricted in practice because of the time and cost associated with measuring wood properties on a large scale. The development of SilviScan (Evans 1994, 1998, 2006) has made the routine measurement of several wood properties possible, facilitating the examination of mid-rotation fertilization on wood properties other than specific gravity. Additionally, recent advances in the application of near infrared (NIR) spectroscopy for wood property assessment have demonstrated that NIR spectroscopy can be used to rapidly estimate many wood properties from radial strips (Schimleck & Evans 2002a, 2002b, 2003, 2004; Jones *et al.* 2005) and therefore provide data that can be used to examine the effects of mid-rotation fertilization on wood properties.

The objective of this study is to examine the effects of mid-rotation fertilization on the mechanical, physical and anatomical properties of loblolly pine wood produced in three rings following fertilization and thinning (1984–1986) at different height levels based on wood properties measured using NIR spectroscopy. The present study also evaluates the ability of NIR spectroscopy to be used as a rapid and reliable tool for estimating wood properties following the application of various silvicultural practices.

MATERIALS AND METHODS

Site description

The study was conducted in a loblolly pine plantation planted in 1970 by the Forest Nutrition Cooperative in New Bern, North Carolina, USA. The study was laid out in a randomized complete block design, replicated four times, with four treatments (16 plots): Control (000N), 112 (112N), 224 (224N), and 336 (336N) kg/ha of nitrogen, with 28 kg/ha phosphorous applied to all treatments. The normal practice in the field is to apply 224 kg/ha of nitrogen along with 28 kg/ha phosphorous. Blocking was done in order to account for variation in dominant height, basal area, stem number per hectare, and soil type. All the plots in the study were thinned to 613 trees per hectare in 1983 and fertilized in March, 1984. The trees for the present study were harvested in 2003.

Sample preparation

Within each block by treatment combination (16 plots), two trees corresponding to two subsamples, representing the average DBH (diameter at breast height) of the trees in the treatment plot, were felled. From the felled trees, disks 25 mm thick were cut at five height levels: 1.4, 3.0, 4.5, 7.6 and 10.6 m. Because of budget constraints, we restricted the present study to disks collected from two trees per plot. Two pith-to-bark radial sections (13 mm tangentially by 13 mm longitudinally, with the radial dimension depending on the size of the tree) were cut from both sides of these disks, oven-dried to a moisture content of 8 percent and glued into core holders. Two radial strips approximately 1.6 mm tangentially by 13 mm longitudinally were cut from the radial sections described above representing each sampled tree (total of 320 strips). One of the radial strips from the two was used for making further observations in this study.

From each felled tree, three 0.6 m bolts were cut such that the midpoints of the bolts were at heights of 2.4, 7.3 and 12.2 m (each bolt representing the mid point of saw logs 4.9 m long). Static bending samples with dimension 25 mm radially by 25 mm tangentially by 406 mm longitudinally were cut radially from the bolts. The static bending samples were cut to include wood produced directly after the fertilization treatments, *i.e.* the 25 mm of wood (in the radial direction) produced immediately following the 1984 fertilization. A total of 162 bending samples were available for testing (a total of 192 samples were collected from 32 trees, 3 heights, 2 sides of the bolt and 30 of the possible 192 samples could not be tested owing to defects) and from these, a sub sample of 75 was selected randomly to provide radial samples for SilviScan analysis. From these samples, blocks 25 mm radially by 25 mm tangentially by 25 mm longitudinally were cut from one end. Radial strips 2 mm tangentially by 7 mm longitudinally by 25 mm radially were cut from the blocks and solvent extracted at CSIRO Forestry and Forest Products, Australia. The data from this sample set were used for developing calibration models to measure wood properties by NIR spectroscopy.

SilviScan measurements

Wood properties of the radial strips of size 2 mm × 7 mm × 25 mm prepared from static bending samples (described in the above section) were measured using SilviScan (Evans 1994, 1998). All measurements were made in a controlled environment (40 percent relative humidity, temperature 20 °C). Air-dry density was measured at an interval of 25 µm using X-ray densitometry. MFA was measured using scanning X-ray diffractometry and averaged over an interval of 1 mm. An estimate of wood stiffness (at the same resolution as MFA) was obtained by combining the X-ray densitometry and X-ray diffraction data (Evans 2006). On a prepared transverse surface, tracheid anatomical properties – radial and tangential diameters and wall thickness – were measured at an interval of 25 µm using image analysis (Evans 1994). The high resolution SilviScan data was averaged over 5 mm sections for correlation with NIR spectra.

NIR measurements

Diffuse reflectance NIR spectra were collected from the radial face of left over blocks (unextracted) used for preparing the SilviScan strip in 5 mm intervals using a FOSS NIRSystems Inc. Model 5000 scanning spectrophotometer. The blocks were physically matched up with the extracted SilviScan strips to ensure that the NIR scans were collected from the neighboring surface from which SilviScan data collected. Samples were held in a custom-made holder with a 5 mm by 5 mm mask to ensure an area of constant size was analyzed (Schimleck *et al.* 2001). The spectra were collected at 2 nm intervals over the wavelength range 1100–2500 nm. The instrument reference was a ceramic standard. A total of 375 spectra were collected from the 75 SilviScan blocks. The spectral data from the unextracted blocks along with the SilviScan data were used for developing calibration models (described in the next section in detail).

NIR spectra were also collected from the radial face of the unextracted strips cut from the disks collected at five heights. NIR spectra were measured in 5 mm increments from the three rings produced following fertilization (1984–1986). We restricted the measurement of NIR spectra to three rings following treatment based on the significant treatment effect reported for three year average specific gravity based on X-ray densitometry (Antony 2006). The spectra were used to estimate wood properties of this post-fertilization region using the NIR-based calibration models.

Calibrations for the estimation of wood properties

The SilviScan wood property data averaged over 5 mm and NIR spectra collected from the SilviScan blocks were used to develop Partial Least Squares (PLS) regression calibrations. Here all the models were based on NIR spectra collected from the radial face of unextracted blocks used to provide the SilviScan strips, with the SilviScan data from extracted radial strips. The wood property calibrations were developed using Unscrambler Software (Version 9.2) and second derivative spectra (left and right gaps of 8 nm were used for the conversion). Summaries of calibrations developed for each wood property are presented in Table 1. Up to 10 factors were used for calibration development with four cross validation segments. The software recommended the final number of factors used for each calibration. The PLS regression calibrations were used to estimate

Table 1. Summary of calibrations developed for each wood property using NIR spectra collected from the radial face of wood samples.

Property	No. of factors	R^2	SECV	SEC	R^2	SEP	RPD
MOE (GPa)	5	0.80	2.1	2.1	0.78	2.2	1.6
Density (kg/m^3)	5	0.79	65.6	65.7	0.78	68.5	1.6
MFA (degrees)	9	0.87	2.2	2.2	0.83	2.4	1.8
Wall thickness (µm)	5	0.82	0.4	0.4	0.80	0.4	1.7
Radial diameter (µm)	7	0.63	1.8	1.8	0.50	2.0	1.2
Tangential diameter (µm)	6	0.57	1.7	1.7	0.50	1.8	1.1
Perimeter (µm)	6	0.60	5.9	5.9	0.51	6.4	1.1

SECV = standard error of cross-validation; SEC = standard error of calibration; SEP = standard error of prediction; RPD = ratio of performance to deviation.

the air-dry density, tracheid properties, MFA and stiffness of the wood produced post thinning and fertilization (the wood produced from 1984–1986) using the NIR spectra collected in 5 mm increments from the unextracted radial strips cut from the disks collected at five heights from each tree. All estimated wood properties were weighted using the basal area of each scan relative to the total NIR scanned basal area.

Statistical analysis and model development

A separate analysis of variance (ANOVA) was performed on each wood property. Tukey's Honestly Significant Different (HSD) test was used to conduct all pairwise mean comparisons. Linear contrasts were used to compare wood properties of unfertilized trees (000N) with the fertilized trees (112N, 224N and 336N) and to compare wood properties of trees which received the lowest rate of fertilization (112N) with the higher rates of fertilization (224N and 336N). Higher order polynomial contrasts were used to understand the wood property changes with the rate of fertilizer applied.

Individual tree effects can be represented as a random-effect and their contribution to the variance of wood properties can be estimated. In addition, to account for the subsampling of two trees within each experimental unit (plot), it is necessary to distinguish tree-to-tree variability and within-tree variability, from variability across the experimental units. These considerations dictate that a mixed-effects model should be employed to account for the distinct sources of variability in the experiment.

In particular, the full linear mixed model used for the analysis can be written as

$$y_{ijkl} = \mu + F_i + H_l + (FH)_{il} + b_j + (Fb)_{ij} + T_{ijk} + (FbH)_{ijl} + e_{ijkl}$$
$$i = 1,\ldots,4,\ j = 1,\ldots,4,\ k = 1,2,\ l = 1,\ldots,5.$$

where y_{ijkl} = the property of interest at the l^{th} height, of the k^{th} tree, of the j^{th} block, receiving the i^{th} fertilization treatment; μ = the population mean; F_i = the i^{th} fertilization effect; H_l = the l^{th} height effect; $(FH)_{il}$ = the interaction of the i^{th} fertilization and l^{th} height effect; b_j = the random effect of the j^{th} block with $b_j \sim NID(0, \sigma_b^2)$; $(Fb)_{ij}$ = the interaction random effect of the i^{th} fertilization and j^{th} block effects with $(Fb)_{ij} \sim NID(0, \sigma_{Fb}^2)$; T_{ijk} = the random effect of the k^{th} tree of the j^{th} block receiving the i^{th}

fertilization with $T_{ijk} \sim NID(0, \sigma_T^2)$; $(FbH)_{ijl}$ = the interaction of the i^{th} fertilization, j^{th} block and l^{th} height effect with $(FbH)_{ijl} \sim NID(0, \sigma_{FbH}^2)$; and e_{ijkl} = residual error, with $e_{ijkl} \sim NID(0, \sigma^2)$.

All analyses were conducted using the MIXED procedure available in SAS version 9.1 (SAS Institute Inc. 2004).

Table 2. P-values for each factor from the analysis of variance.

Property	Source		
	F	H	F*H
MOE (GPa)	0.0468	0.0001	0.8529
Density (kg/m³)	0.0766	0.0001	0.9810
MFA (degrees)	0.2080	0.0001	0.7856
Wall thickness (μm)	0.0871	0.0001	0.9717
Radial diameter (μm)	0.0930	0.0001	0.1259
Tangential diameter (μm)	0.1981	0.0001	0.3455
Perimeter (μm)	0.1912	0.0001	0.2745

RESULTS

The results of the ANOVA (analysis of variance) for all properties are presented in Table 2. The effect of height was found to be significant for all wood properties at the 0.05 level of significance. No significant interaction effects were found at the 0.05 level of significance for all wood properties (Table 2). Since our purpose was to understand the effect of fertilization, we will restrict our results and discussion sections to significant fertilization effects and the interaction between fertilization with height.

The effect of fertilization on wood stiffness was significant (p-value = 0.0468) (Table 2) and follows a linear trend (p-value = 0.0175, Fig. 1). Modulus of Elasticity (MOE) ranged from 12.7 (112N) to 10.7 GPa (336N) and pairwise comparisons indicated significantly higher MOE for the 112N treatment compared to the 336N treatment (P-value = 0.0404). A significant difference was found in the stiffness of trees which received a lower rate (112N) of fertilizer compared to trees which received a higher rate (224N and 336N) with a p-value of 0.0155.

Air-dry density was significantly influenced by mid-rotation fertilization (p-value = 0.0766, $\alpha = 0.10$) (Table 2) with the density of the 336N treatment (545 kg/m^3) significantly lower than the 112N treatment (591 kg/m^3) with a p-value of 0.0775. The effect of fertilization on density was described by a linear trend (p-value = 0.0503, Fig. 1). Tracheid wall thickness was significantly influenced by mid-rotation fertilization (p-value = 0.0871, $\alpha = 0.10$) (Table 2). Pairwise comparisons indicated a significant difference between the 112N and 336N treatments with a p-value of 0.0781 ($\alpha = 0.10$). The changes followed a linear trend (p-value = 0.0579, Fig. 1) with treatment. A significant difference was found in the stiffness of trees which received a lower rate (112N) of fertilizer compared to trees which received higher rates (224N and 336N) with a p-value of 0.0767 ($\alpha = 0.10$).

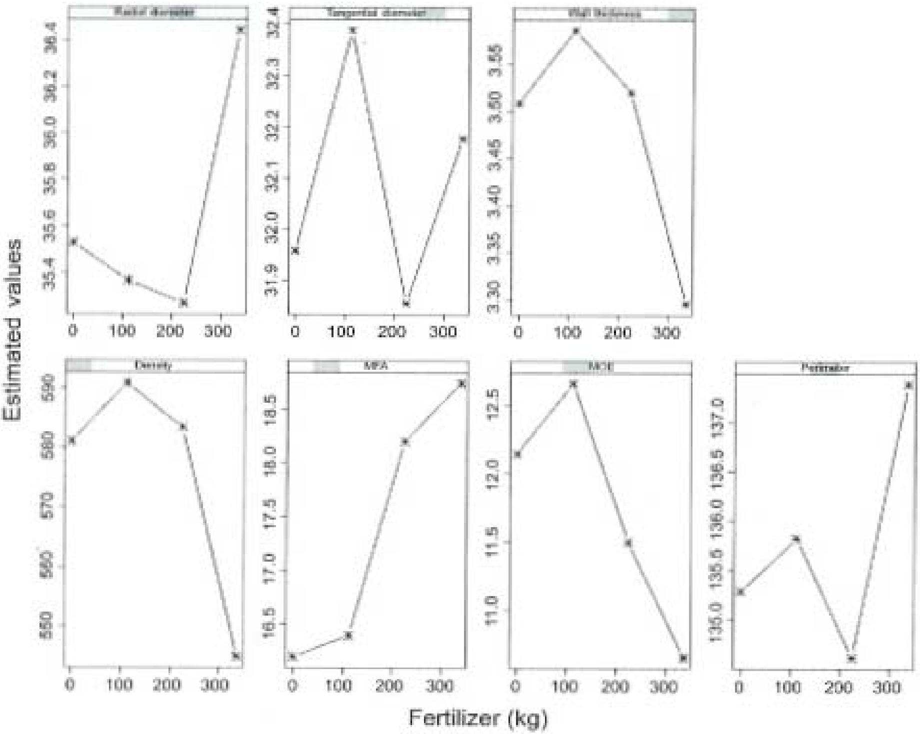

Figure 1. Plots of estimated mean wood property versus fertilization levels.

The effect of fertilization on MFA was not statistically significant. The effect of fertilization on MFA was described by a linear trend (p-value = 0.0511, Fig. 1). Radial diameter was significantly changed by fertilization (p-value of 0.0930, $\alpha = 0.10$) (Table 2). The 336N treatment had a substantially higher mean (36.4 µm) than the others; however, no statistically significant differences among treatments were revealed by pairwise comparisons. The fertilization effect followed a linear trend across treatments (p-value = 0.0932, Fig. 1). Tracheid tangential diameter and perimeter were not influenced by mid-rotation fertilization (Table 2).

DISCUSSION

A significant decrease in stiffness (at $\alpha = 0.05$), air-dry density, and cell wall thickness (at $\alpha = 0.10$) was observed for the heaviest application of fertilizer (336N). The average stiffness of trees for this treatment was 12 percent less compared to that of the control and 16 percent less compared to trees treated with 112N (the stiffness of trees treated with 112N increased by 4 percent compared to the control). A decrease in stiffness was also reported in boards cut from wood produced following thinning and mid-rotation fertilization in radiata pine by Nyakuengama *et al.* (2004). The air-dry density and cell wall thickness was lower (6 percent reduction) for trees which received the heaviest application of nitrogen compared to the control. The decrease in density and cell wall thickness immediately following fertilization was also reported in slash pine (*Pinus elliottii* Engelm.) (Williams & Hamilton 1961), Douglas fir (Erickson & Harrison 1974) and radiata pine (Nyakuengama *et al.* 2002). However, the stiffness, density and wall thickness for treatments 112 N and 224 N did not change considerably compared to the control. This indicates that mid-rotation fertilization (at appropriate levels) following thinning can achieve considerable gains in growth while not impacting on the properties of the wood produced in loblolly pine. A 2 to 3° increase in MFA and a 1 µm increase in tracheid radial diameter were observed for the heaviest fertilizer treatments compared to the control, which was also observed in fertilized Douglas fir (Erickson & Arima 1974). However, tracheid properties (MFA, tracheid radial diameter, tracheid tangential diameter and tracheid perimeter) of the treatments 112 N and 224 N did not change significantly following fertilization.

Stiffness is a property of particular interest to the timber industry and determines the value of sawn timber from the mill in terms of its product quality. A strong correlation among stiffness, density and MFA of wood has been reported in previous studies (Megraw *et al.* 1999; Cown *et al.* 1999; Cave & Walker 1994; Evans & Ilic 2001; Larson *et al.* 2001). The decrease in stiffness for the heaviest application of N can be explained by the decrease in air-dry density and the increase in MFA following fertilizer application. The decrease in air-dry density and thus stiffness to some extent can be elucidated by examining the changes that occurred to the anatomical properties of tracheids, mainly wall thickness and MFA, and to a lesser degree, radial diameter, produced after mid-rotation fertilization. The significant decrease in density and cell wall thickness for the heaviest N application may be attributed to increased foliar growth following fertilization and a subsequent reduction in the availability of photosynthates for secondary cell wall thickening (Larson *et al.* 2001). The 2 to 3° increase in MFA for the higher fertilization rates may be due to the production of shorter tracheids following increased anticlinal division of the secondary cambium following fertilization.

The present study used NIR spectroscopy to estimate the wood properties of the majority of samples. Recent studies (Schimleck *et al.* 2004; Jones *et al.* 2005) have demonstrated that NIR spectroscopy can be used to measure a wide range of loblolly pine wood properties rapidly and non-destructively. Unlike, high resolution measurement techniques such as X-ray densitometry and SilviScan, NIR spectroscopy is a relatively low resolution method and the wood property measurements are usually averaged over a distance of 5 mm or 10 mm. However, NIR spectroscopy provides data for a wide range of properties at a far lower cost than SilviScan. Calibrations based on SilviScan data and NIR spectra collected from the radial strips analyzed by SilviScan provided predictions of wood properties that were used to analyze the effects of mid-rotation fertilization. Our findings were in accordance with other fertilization studies and the X-ray densitometry data collected on these samples (Antony 2006); however, our NIR estimated wood properties may have lacked the sensitivity required to detect variation between individual fertilization treatments, particularly 112 N and 224 N. The small sample size used for this study could be a contributing factor to the reduced sensitivity of the NIR measurement. Increasing the number of trees sampled per plot may be an option to increase the precision of NIR measurements in silvicultural response studies.

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