Mapping and modeling within-tree variation for loblolly pine pulp yield and lignin content

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Abstract

We examined the within-tree variation of pulp yield and lignin content for loblolly pine (*Pinus taeda* L.) trees aged 13 and 22 years. Radial trends in pulp yield (increase) and lignin (decrease) were consistent with what would be expected for loblolly pine as were changes in properties related to maturation. Maps, based on the average of 18 trees at each age, depicting pulp yield variation within-tree were similar to loblolly pine maps reported for microfibril angle and stiffness, while lignin maps resembled the inverse of those reported for density and related properties. Mixed-effects models for both properties were developed with the base model for pulp yield explaining 64% of the observed variation, with the inclusion of tree height improving the model slightly, whereas models for lignin content explained 44% of the variability. The models could be incorporated into growth and yield prediction systems, or procurement model systems that predict within-tree wood properties based on age and tree size.

Keywords Lignin content · Loblolly pine · Near-infrared spectroscopy · Nonlinear wood property models · *Pinus taeda* · Pulp yield · Wood property maps

1 Introduction

Loblolly pine (*Pinus taeda* L.), native to the southeastern USA, is one of the most important plantation species in world. It is heavily utilized by the pulp industry in the US south, historically a major producer of Kraft pulp [27], and in 2016, 78% of total pulpwood production in the region was from conifer roundwood and residues [20]. Loblolly pine is used to produce a range of pulps (Kraft is the most common) and to manufacture a variety of products [26, 47, 58]. The revenue generated by the pulp and paper sector is significant; for example, in Georgia this sector generates 65% of the total forestry and forest products revenue [18]. Despite the importance of loblolly pine for Kraft pulp production, limited information exists for this species (and hardwood and softwood species in general) regarding

within-tree variation of pulp yield and lignin content, two properties directly related to the economics of pulp production [21, 64].

The lack of information on within-tree variability relates directly to the high cost associated with determining many of the properties considered important for pulp manufacture, particularly pulp yield, and to a lesser degree lignin content, which both require detailed wet chemistry methods for determination. Koch [27] reports that for a Kappa number of 109 southern pine pulp yield will be approximately 57%, whereas the typical lignin range for southern pine pulpwood is from 26 to 29%. More recently, Kim et al. [26] reported screened Kraft pulp yields of 44.6% (Kappa number = 27.4) for loblolly pine thinnings and 45.6% (Kappa number = 24.2) for mature wood, whereas lignin

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contents varied from 29.8% (thinnings) to 28.5% (mature wood).

In conifers, it is recognized that corewood (or juvenile wood) has lower cellulose (included here as an indicator of pulp yield) and higher lignin content compared to outerwood (or mature wood) [45, 63]. Specifically, a radial trend of increasing pulp yield for Norway spruce (*Picea abies* (L.) Karst.) was observed by Molteberg and Høibø [40], which is consistent with the radial increases in α -cellulose and glucan (especially for the first 10–15 growth rings after which it increased slowly), and decreasing trends of pentosans and lignin observed by Harwood [22] and Uprichard [62] in radiata pine (*Pinus radiata* D. Don). Various authors have reported a decrease in lignin, including Larson [29] for red pine (*Pinus resinosa* Sol. ex Aiton), Ericksen and Arima [13] for Douglas fir (*Pseudotsuga menziesii* (Mirb.) Franco) and Via et al. [63] for longleaf pine (*Pinus palustris* Mill.). In comparison, Gierlinger and Wimmer [19] reported increasing radial trends for lignin in European larch (*Larix decidua* Mill.) from sites in Austria and France; however, the trees were much older than those examined by the other authors (146 and 250 years old, respectively). More recently, near-infrared hyperspectral imaging (NIR-HSI) has been utilized [37, 59, 60] to examine within-tree variation for various wood sugars and lignin content in radiata pine. Meder et al. [37] reported radial trends of increasing glucose and decreasing lignin for spectra from increment cores, while Thumm et al. [59] mapped wood property variation for a radiata pine disk, finding elevated levels of galactose and lignin, and lower glucose in a region with pronounced compression wood.

Studies of within-tree variation of pulp yield in eucalypts are more common. Batchelor et al. [3, 4] in Australia and Manfredi [35] in Brazil reported yield variation with height, with the earliest maps of yield variation within-trees reported by Crawford et al. [7] for two large Tasmanian grown alpine ash (*Eucalyptus delegatensis* R. T. Baker) trees (large trees were required to provide sufficient sample for pulping). Schimleck and Michell [53] developed maps illustrating pulp yield variability within plantation-grown shining gum (*Eucalyptus nitens* (H. Deane & Maiden) Maiden) based on NIR spectroscopic estimates of pulp yield for individual ring samples. In recent studies, Downes et al. [11, 12] have utilized NIR spectroscopy to examine pulp yield variation at high spatial resolution (1-mm increments) for shining gum and blue gum (*Eucalyptus globulus* (Labill.)).

In addition to chemical changes, pulp product properties are influenced by fiber (tracheid) property changes that occur within-trees. For properties such as tracheid length and width, which can be determined at a relatively low cost compared to pulp yield, some maps are available. Examples include Lundqvist et al. [31] and Dahlen et al.

[9] for loblolly pine, Mäkinen et al. [34] for Norway spruce, while Ikonen et al. [23] developed maps for tracheid length only for Scots pine (*Pinus sylvestris* L.). In general, available literature is limited to plots of radial variation (sometimes at multiple heights) and the number of trees, by necessity, is limited [44]. The development of SilviScan [14] facilitated rapid measurement of tracheid dimensions and, coupled with density estimates, allowed the derivation of several additional properties including coarseness and wall thickness. Maps showing how these properties varied within radiata pine trees were reported by Evans et al. [17], while Defo et al. [10] utilized SilviScan data to develop coarseness maps for Douglas fir.

Recently, we have utilized NIR spectroscopy, calibrated with SilviScan reference data [14, 15, 16], to map wood property variation in loblolly pine [42, 54] at different ages [55, 57]. The wood property models used SilviScan data and NIR spectra collected in 10-mm increments from selected sections of a subsample of radial strips from multiple trees and at specific heights. NIR-based wood property models predicted properties in 10-mm increments for all strips at all heights, and with these data, whole-tree maps were developed (for these studies the maps represented the average of 18 trees at age 13 and another 18 trees at age 22). Presently, maps based on this approach have been reported for air-dry density, microfibril angle (MFA) and stiffness (MOE) and for a range of tracheid-related properties such as coarseness, radial and tangential diameter, specific surface and wall thickness. Using a similar approach, but with a far larger data set (NIR spectra were collected from 11,428 10-mm sections), Dahlen et al. [9] developed maps demonstrating within-tree variation of tracheid length and width for loblolly pine samples from across the southeastern USA.

Most of these studies of within-tree variation involved using a “spatial-specific” model [56] as wood property data and the corresponding spectral data are measured from pith-to-bark and at multiple heights at the same spatial resolution (e.g., 10-mm radial increments). An alternative approach is to use a “spatial-interpolated” model [56] which involves “measuring a property at a broad scale, e.g., whole-tree, calibrating this data against NIR spectra representing the equivalent scale and then using the calibration to predict the property at higher resolution.” Recently, Schimleck et al. [56] used both approaches to examine within-tree variation of density and coarseness and found that patterns of variation for maps obtained using the two approaches were comparable. Density was measured using SilviScan (= spatial-specific model data) and on chips produced from bolts (= spatial-interpolated model data). Similarly, tracheid coarseness was measured using SilviScan (spatial-specific) and on the pulp produced from the chips (spatial-interpolated). Coarseness values

were adjusted to facilitate these comparisons as SilviScan measurements represented a “dry” coarseness measurement on non-pulped samples (i.e., cell wall thickness unaltered), while measurements on whole-tree measurements were on pulp samples suspended in water (cell wall thickness reduced/collapsed during pulping) [6, 25, 52]. Validation of the spatial-interpolated model approach is important as it gives confidence when using NIR models, developed for properties measured on whole-tree samples (e.g., pulp yield), to predict properties at a much higher resolution, e.g., 10-mm sections of a radial strip. It is important to note that the critical step in utilizing this approach is that NIR spectra collected in 10-mm increments at various radial positions and heights are area weighted to provide a representative whole-tree average spectrum for each tree. An assumption is that the spectra adequately represent the original whole-tree composite samples used for wood property analysis but small discrepancies owing to the different approaches used to obtain the two sets of whole-tree spectra are to be expected.

For the loblolly pine trees examined in Mora and Schimleck [42], Schimleck et al. [54, 55, 57] wood properties such as pulp yield, and lignin content were measured on whole-tree samples and provide an opportunity to utilize the spatial-interpolated model approach to investigate the within-tree variation of these properties. Hence, the goals of this study are:

- a. To develop NIR-based whole-tree models for pulp yield, and lignin content utilizing the NIR dataset previously described;
- b. Use the models to predict each wood property in 10-mm increments (spatial-interpolated approach); and
- c. Model and examine the patterns of within-tree (radial and longitudinal) variation in pulp yield and lignin content.

2 Material and methods

2.1 Sample origin

Two sets of eighteen loblolly pine trees aged 13 and 22 years, respectively, were sampled from a half-sib progeny trial planted at International Paper's (IP) Southlands facility located in the lower coastal plain near Bainbridge, GA. The trees were selected to encompass the range of cellulose, lignin and specific gravity variation within the larger populations as measured by IP. Average diameter at breast height for selected trees was 82.6 mm (age 13) and 96.1 mm (age 22), respectively. The trees were felled and samples removed for wood property and NIR analyses.

Disks (25-mm thick) were taken at 1.5-m intervals along the stem of each tree, giving 9 to 13 disks per tree (number depended on tree height). A total of 411 disks were used for the study: 191 disks collected from trees aged 13 years and 220 from the 22-year-old trees. In addition, the stems of each sampled tree were divided into four sections of equal length and a 0.75-m bolt was cut from the center of each section.

2.2 Pulp property measurements

The four 0.75-m bolts collected for each tree were chipped using a 1.2-m Carthage chipper at the Institute of Paper Science and Technology (IPST), Atlanta, GA, and then thoroughly mixed to give a single composite sample per tree. Several properties were measured on each composite sample; however, for this study we will only focus on pulp yield and lignin content. White et al. [64] provide a detailed description of the Kraft pulping process. Briefly, for each whole-tree composite sample duplicate cooks were performed on 1 kg of oven-dried whole-tree chips in a 10L M/K digester. Two pulp grades (linerboard at 100 Kappa and bleachable at 30 Kappa) were obtained for each sample, and Klason lignin content was determined according to the TAPPI T222-om-88 standard [64].

2.3 Near-infrared spectroscopy

2.3.1 Sample preparation: radial strips

Pith-to-bark radial sections were cut from each disk using a bandsaw. Section dimensions were 12.5 mm longitudinally by 12.5 mm tangentially with the radial length corresponding to the radius of the disk. Radial sections were dried in a 50 °C oven for 24 h and then air-dried, glued into core holders and cut using a twin-blade saw to give strips 2-mm-thick (tangentially) using the methodology described in Jordan et al. [24].

2.3.2 Collection of NIR spectra

All strips were conditioned and tested in a controlled environment room (40% relative humidity, 20 °C). NIR spectra were collected in 10-mm increments from the radial-longitudinal face of each radial strip using a FOSS NIRSystems Inc Model 5000 scanning spectrometer fitted with a 5 by 10 mm Teflon mask (to ensure spectra were collected from an area of consistent size). NIR spectra (an average of 50 scans for each 10 mm section) were collected over the wavelength range 1100–2498 nm in 2-nm increments. The total number of spectra collected was 2,569 (1,114 from 13-year-old trees and 1,455 from trees aged 22 years). Schimleck et al. [54] provide a more detailed description of

the methodology used to collect NIR spectra. The annual rings were counted for each 10-mm interval to determine the mean cambial age for each 10-mm section, which was used for the modeling efforts.

2.4 Data analysis

The data analysis and figures were produced in the R statistical programming environment [49] with the RStudio interface [50]. Data wrangling was done using the tidyverse series of packages [66]. NIR partial least squares (PLS) regression models were developed using the pls package [39]. Tree maps were produced using the akima [1], fields [43], and lattice [51] packages, while the other data visualizations were produced using ggplot2 [65]. Nonlinear mixed-effects models were developed using the nlme library [46]; those were compared using fit indices (R^2) of the fixed, and fixed plus random (stand, tree) components of the models, and root mean square error (RMSE) values. All models were developed iteratively, keeping only those parameters with p -values less than or equal to 0.05 in the models.

2.4.1 Determination of weighted whole-tree spectra

Our NIR-based models for pulp yield and lignin content were developed at the whole-tree level (i.e. "spatial-interpolated"). The approach is described in Schimleck et al. [56] whereby the objective is to take the individual 10-mm radial spectra (2,569 in total) and weight them by their basal area in order to obtain one spectrum that represents the individual tree (36 in total). The representative spectra for each tree are then used to develop calibration models for the properties measured at the whole tree level.

2.4.2 Lignin and pulp yield calibrations

Lignin and pulp yield PLS regression calibrations with leave-one-out cross-validation were developed. Calibration performance was assessed using the following parameters:

- Coefficient of determination (R^2), the proportion of variation in the calibration set that was explained by the calibration;
- Coefficient of determination of cross-validation (R_{cv}^2), the proportion of variation in the cross-validation set that was explained by the calibration;
- Standard error of calibration (SEC) determined from the residuals of the final calibration;
- Standard error of cross-validation (SECV) determined from the residuals of each cross-validation phase;

- Ratio of performance to deviation (RPD_c) [67], calculated as the ratio of the standard deviation of the reference data to the SECV.

Determination of RPD allows comparison of calibrations for different properties that have differing data ranges and units, the higher the RPD, the more accurate the data are described by the calibration. The PLS models were then used to predict pulp yield and lignin for all 2,569 spectra in 10-mm increments.

2.4.3 Maps of wood property variation within trees

Mora and Schimleck [42] provide a detailed description of the methodologies they used to develop maps of within-tree variation for air-dry density, MFA and MOE. Briefly, three different methods were explored: Akima's interpolation, universal kriging and semiparametric smoothing. Mora and Schimleck [42] reported that for loblolly pine, maps generated by Akima's interpolation method provided good representation of the expected trends in air-dry density and MFA. In addition, they concluded that the principal advantage of Akima's algorithm over universal kriging and semiparametric smoothing techniques was that only straightforward procedures were required, and there were no problems concerning computational stability or convergence. Owing to these features, only Akima's interpolation method was used in this work.

2.4.4 Estimation of whole-tree averages

Following the Akima's interpolation method, mean pulp properties for each age were calculated by weighting the volume of each radial point compared to the overall volume and then summing each respective height. To do this, the basal area for each 10-mm radial section within a disk was calculated using:

$$\text{area}_{\text{disk}} = \pi r_o^2 - \pi r_i^2 \quad (1)$$

where $\text{area}_{\text{disk}}$ is the area of radial section within a disk, r_o is the outside radius, and r_i is the inside radius. The weighted average property (pulp yield or lignin content) within a disk was calculated using:

$$\text{property}_{\text{disk}} = \sum_{i=1}^n \frac{\text{area}_i \times \text{property}_i}{\text{area}_{\text{disk}}} \quad (2)$$

where $\text{property}_{\text{disk}}$ is the weighted average property for a specific disk, area_i is the area that a specific 10-mm radial section represents within a disk, property_i is the property at a specific 10-mm radial section, $\text{area}_{\text{disk}}$ is the total area of a disk, and n is the number of 10-mm radial sections

within each disk. The weighted average property was then calculated using:

$$\text{property}_{\text{tree}} = \sum_{i=1}^k \frac{\text{area}_{\text{disk}_i} \times \text{property}_{\text{disk}_i}}{\text{area}_{\text{tree}}} \quad (3)$$

where $\text{property}_{\text{tree}}$ is the weighted average property for a specific tree, $\text{area}_{\text{disk}_i}$ is the area that a specific disk represents, $\text{property}_{\text{disk}_i}$ is the calculated property for each disk, $\text{area}_{\text{tree}}$ is the total area for each tree, and k is the number of disks within each tree. Note that the weighted average properties at lower height levels within each tree (such as the stump) represent a greater amount of the total area of the disks than the property at the tip of the tree. The calculated values were compared to measured values reported in White et al. [64].

2.4.5 Within-tree pulp yield and lignin content model development

Models for predicting pulp yield and lignin content as a function of cambial age (ring number) and height within tree were fitted. The within-tree variability of wood and fiber properties of plantation softwood species tend to vary based on these two variables (cambial age and height) more so than other variables such as the size of trees [2, 5, 28]. The model forms were selected by visual examination of the data. General radial trends found were an increase in pulp yield from pith to bark, and a consistent increase in pulp yield with height for a given ring for both corewood and outerwood. Lignin showed opposite radial trends, with a decrease in lignin content from pith to bark, and, for a given ring in the corewood, lignin content was lower with increasing tree height. Differences in lignin content based on height within tree for outerwood were not detected. For both models the effect of disk height was not linear. We took the natural logarithm of disk height (in meters) and added 1 m to each disk height value to prevent negative infinity values, and to reduce the slope of the natural log effect that occurs with values below 1 [9, 41].

For pulp yield, a three-parameter logistic function [48] was selected. This model form has been successfully used to model latewood specific gravity in loblolly pine [8] and tracheid width measured on macerated samples of loblolly pine [9]:

$$\text{pulp} = \frac{\beta_0}{1 + e^{\left(\frac{\beta_1 - \text{CA}}{\beta_2}\right)}} \quad (4)$$

where pulp is the mean ring pulp yield content (%), CA is the cambial age (ring number), β_0 is the asymptote as

cambial age approaches $+\infty$, β_1 the inflection point, and β_2 a scale parameter. In order to vary pulp yield by disk height in both corewood and outerwood, we incorporated disk height into the model:

$$\text{pulp} = \frac{\beta_0}{1 + e^{\left(\frac{\beta_1 - \text{CA}}{\beta_2}\right)}} + \beta_3 \log(\text{DH} + 1) \quad (5)$$

where β_3 is the fixed effect parameter, and DH is the disk height. Considering random effects, β_0 was allowed to vary by site and tree:

$$\text{pulp} = \frac{\beta_0 + a_i + a_{ij}}{1 + e^{\left(\frac{\beta_1 - \text{CA}}{\beta_2}\right)}} + \beta_3 \log(\text{DH} + 1) \quad (6)$$

where a_i and a_{ij} represent the nested random effects of the asymptote parameter β_0 at the site i and tree j levels, respectively.

For lignin content, a variation on the three-parameter logistic function [48] was used which has previously been used to model earlywood specific gravity in loblolly pine [8]:

$$\text{lignin} = \beta_0 \times e^{\left(\frac{\beta_1}{\text{CA} + \beta_2}\right)} \quad (7)$$

where lignin is the mean ring lignin content (%), CA is the cambial age (ring number), β_0 is the asymptote reached when cambial age approaches $+\infty$, β_1 and β_2 both control the lignin content at the pith and the rate of change from pith to bark. In order to vary lignin content at the pith by disk height, we incorporated disk height into the dominator:

$$\text{lignin} = \beta_0 \times e^{\left(\frac{\beta_1}{(\text{CA} + \beta_2 + \beta_3 \log(\text{DH} + 1))}\right)} \quad (8)$$

where β_3 is the fixed effect parameter and DH is the disk height. Considering random effects, β_0 was allowed to vary by site and tree:

$$\text{lignin} = (\beta_0 + a_i + a_{ij}) \times e^{\left(\frac{\beta_1}{(\text{CA} + \beta_2 + \beta_3 \log(\text{DH} + 1))}\right)} \quad (9)$$

where a_i and a_{ij} represent the nested random effects of the asymptote parameter (β_0) at the site i and tree j levels, respectively.

3 Results and discussion

3.1 Spatial-interpolated whole-tree lignin and pulp yield calibrations

Spatial-interpolated NIR-based models for pulp yield and lignin were developed using data obtained for these

properties on a whole-tree basis and basal area weighted 10-mm spectral data from each tree averaged to give a single representative spectrum per tree. Models are reported in Table 1 and plotted in Fig. 1, with the pulp yield (6 factors, $R^2=0.72$, $R_{cv}^2=0.53$, $SECV=1.10\%$) and lignin models (7 factors, $R^2=0.82$, $R_{cv}^2=0.51$, $SECV=0.76\%$) being comparable. The number of factors recommended for both models corresponded to a clear minimum in explained variance and R_{cv}^2 values continued to improve as the number of factors increased indicating that the models were not over-fitted. The pulp yield model compares well with that reported by White et al. [64] that was based on the same dataset but used NIR spectra collected from a representative chip sample processed from the whole-tree samples (intact chips were analyzed by NIR when green, after drying and after milling).

3.2 Lignin and pulp yield maps

Figures 2 and 3 show pulp yield and lignin maps that represent the average of 18 loblolly pine trees at ages 13 and 22, respectively, with age-related trends of increasing pulp yield, and decreasing lignin apparent. At both ages, the highest pulp yields (and lowest lignin contents) are observed at the periphery of the trees with the region of highest yield at a height of 4.5 m. The range in within-tree

property values is similar (approximately 7%) for the 22-year-old maps, but the differences in the lignin content map appear more subtle as much of the variability is concentrated near the pith. Examination of the probability density plots (Fig. 4) of lignin content for the 22-year-old tree map shows that the majority of values fall in the range of 30–30.5% with very few lignin values less than 30%.

The map for pulp yield (Fig. 2) is very similar to the loblolly pine maps reported for MFA, stiffness and tracheid length [9, 40, 53], with trends present in both the radial and longitudinal directions. While the maps were reported for shining gum, the variation observed by Schimleck and Michell [53] was also similar to the pulp yield maps reported for loblolly pine.

The maps for lignin (Fig. 3) initially appear dissimilar to other maps showing within-tree variation in conifers. Closer inspection indicates that the variation, particularly for the 13-year-old lignin map, is similar to what has been reported for density in loblolly pine [42, 55] and radiata pine [17, 61] with lignin content decreasing radially as opposed to increasing radially as with density. In their summary of density variation in radiata pine and loblolly pine, Burdon et al. [5] note that this pattern of variation is typical of properties that demonstrate a radial increase (or decrease as observed for lignin) at all heights.

Table 1 Summary of the multiple height loblolly pine wood property calibrations developed for each wood property using all 36 NIR spectra

Wood Property	Calibration set (36 spectra)					
	# factors	R^2	R_{cv}^2	SEC	SECV	RPD _c
Pulp yield (%)	6	0.72	0.53	0.80	1.10	1.9
Lignin content (%)	7	0.82	0.51	0.46	0.76	2.4

Fig. 1 Calibration plots for pulp yield and lignin content

Fig. 2 Maps showing within-tree variation of pulp yield (%) for loblolly pine trees aged 13 and 22 years. Maps at each age represent the average of 18 trees and were developed using Akima's interpolation method

In their study of Norway spruce, Molteberg and Høibø [40] observed that trees with larger diameter had a reduction in pulp yield. They related this observation to the wider rings of larger diameter trees having more earlywood, which owing to thinner cell walls and a reduction in the low lignin S2 layer of the cell wall, results in a relative increase in the amount of lignin. Similarly, an increase in density with age can be related to a decrease in lignin content (and an increase in cellulose content). Density in loblolly pine increases as the proportion of latewood [30, 24] increases and as corewood transitions to outerwood [5, 45]. Latewood cells have a thick S2 layer that is relatively

low in lignin and high in cellulose; hence, an increase in latewood percent (and density) should correspond to an overall reduction in lignin content. Burdon et al. [5] note the presence of such trends in radiata pine.

3.3 Whole-tree averages

Pulp yield and lignin averaged on a whole-tree basis were determined for individual trees at age 13 and 22-years. Pulp yield increased with an increase in age, whereas lignin decreased. The change in average tree values for both properties with age was relatively small with pulp

Fig. 3 Maps showing within-tree variation of lignin content (%) for loblolly pine trees aged 13 and 22 years. Maps at each age represent the average of 18 trees and were developed using Akima's interpolation method

yield increasing from 57% (13 years old) to 58.7% (22 years old), and lignin content decreasing from 30.8% at 13 years to 30.6% at 22. These values are comparable to those reported by McDonough et al. [36] who reported averages for pulp yield of 56.8% at age 13 and 59.4% (average Kappa number was 112) at 22 years and 30.4% and 30.6% for lignin content at 13 and 22 years, respectively.

Probability density plots (Fig. 4) show the distribution of pulp yield and lignin content for individual trees at ages 13 and 22 and age-related shifts in property means. As noted in [57], the 22-year-old trees have considerably more volume (approximately 50%) than the 13-year-old trees.

3.4 Models

Plots showing variation in loblolly pine pulp yield and lignin content by cambial age and with height (0.15, 5.2 and 10.4 m) within tree are shown in Figs. 5 and 6, respectively.

The pulp yield model (see Table 2 for parameter estimates) closely followed the tree map, whereby pulp yield increased radially, and pulp yield increased with height for a given cambial age. The increase was consistent throughout both the corewood and the outerwood, and the pulp yield change could be detected with height. Lignin

Fig. 4 Probability (density) plots showing the variation of the predicted chemical properties for pulp yield and lignin content. The area under the density curves has a probability of one

Fig. 5 Variation in loblolly pine pulp yield by cambial age and height within tree. Lines represent the predictions from Eq. 3 at the following disk heights: 0.15 m (solid), 5.2 m (dotted), 10.4 m (dashed)

Fig. 6 Variation in loblolly pine lignin content by cambial age and height within tree. Lines represent the predictions from Eq. 6 at the following disk heights: 0.15 m (solid), 5.2 m (dotted), 10.4 m (dashed)

Table 2 Parameter estimates for the pulp yield and lignin content models

Property	Parameter	Model	
		Base (Eq. 5)	Disk height (Eq. 6)
Pulp yield (%)	β_0	59.164	58.725
	β_1	-11.614	-12.704
	β_2	4.776	5.444
	β_3	-	0.451
Lignin content (%)		Base (Eq. 8)	Disk height (Eq. 9)
	β_0	26.273	26.992
	β_1	3.408	2.303
	β_2	14.596	9.865
	β_3	-	0.507

content also varied with height, but only in the corewood. In the outerwood, the lignin content for all heights converged to a common value (~28%). Differences in height were more subtle than pulp yield. The pulp yield model had better fit statistics than the lignin model, with the fixed effects for the final pulp yield model having an R^2 of 0.44 versus 0.20 for the lignin model (Table 3). In both cases, including disk height improved the model significantly (p -value < 0.001), although for lignin the difference was subtle. Even though the R^2 was much higher for pulp yield, the RMSE was similar (1.49% vs. 1.68%). The random variation by stand (age 13 and 22) was the same for both pulp yield and lignin (4%); however, since there were only two stands, this may not reflect the variation that exists on the landscape. The random variation attributed to individual trees was similar to pulp yield (19%) and lignin (20%).

We utilized the spatial-interpolated approach described in Schimleck et al. [56] to generate NIR-predicted wood property data that was subsequently used to examine within-tree variation of pulp yield and lignin content in loblolly pine trees aged 13 and 22 years. This approach

Table 3 Fit indices and model errors for the pulp yield and lignin content models

Property	Model ^a	Fit indices (R^2)			Model errors
		Fixed	Stand	Tree	
Pulp yield (%)	Base	0.39	0.45	0.64	1.55
	DH	0.44	0.48	0.67	1.49
Lignin (%)	Base	0.20	0.23	0.44	1.69
	DH	0.20	0.24	0.44	1.68

^aBase model does not include disk height, DH = disk height model

^bRoot mean square error (RMSE) and mean percentage error (|E%|) are calculated from the fixed part of each model

relies on utilizing wood property data collected on whole-tree composite samples and NIR spectra collected at much high resolution (10-mm increments from radial strips sampled at multiple heights) but basal area weighted to provide a single spectrum per tree. The pulp yield and lignin maps are, to the best of our knowledge, the only whole-tree maps of these properties published for loblolly pine

and the only visual representation of the change in these properties associated with maturation. The radial trends we observed for both properties are consistent with our expectations for this species based on literature reported for other pines [5, 29] and conifers in general [13, 40]. In addition, our NIR-based estimates for pulp yield and lignin agree with those published by Koch [27] and McDonough et al. [36].

Ideally more samples would have been available for model development but owing to the high cost of pulp yield determination, and to a lesser degree lignin analysis, this was not possible. Having more samples may have also decreased the difference between calibration and cross-validation statistics but as each tree represents a unique addition to the model there is no guarantee that this would occur and the errors that exist may largely be a result of analytical errors in the determination of lignin and pulp yield. It is also important to recognize that the errors represented in the calibration phase that may arise owing to the unique aspects of a given individual are not critical in terms of the prediction of 10 mm sections, as all samples were included in the final model. Hence, variability in the prediction set was incorporated in the calibration set unlike the situation encountered during the cross-validation phase and is reported in Table 1.

Other approaches have been utilized to examine the within-tree variation of pulp yield. Specifically, Crawford et al. [7] pulped individual samples collected at various radial positions and heights in two Tasmanian grown alpine ash. This approach is the most desirable as it provides wet chemistry data for each measurement point, but owing to the time and cost involved in determining pulp yield [38], only a limited number of samples, and ultimately trees, can be sampled. Schimleck and Michell [53] utilized NIR spectroscopy and minipulping to examine pulp yield variation within 15 plantation-grown shining gum aged 18. For these trees, individual rings (or two rings when very close together) were sampled using a drill, and the milled wood used to collect NIR spectra. Minipulping was conducted on outerwood, multi-ring samples collected at three heights from 3 trees. These data were then incorporated into an existing shining gum calibration for the region in which the trees grew. The model was used to estimate the pulp yield of individual rings, and the data were subsequently used to generate maps of pulp yield within individual trees and for an average of all trees. The approach utilized in this study allowed many more trees to be examined, and while it was observed that the variability of maps for individual trees made it impossible to determine any consistent pattern of variability, the utilization of maps based on 3 (those that provided minipulping samples) and 15 trees made conclusions regarding patterns of variability possible.

Our approach differs markedly to that of Schimleck and Michell [53], in terms of the methods used to collect spectra (milled ring samples vs spectra collected in 10-mm increments from radial strips), and presents a more consistent approach in terms of the collection and utilization of spectral data. As noted by [55] the time required to prepare samples is greatly reduced by avoiding the need to grind samples facilitating the analysis of more trees. With more trees (and samples) analyzed, maps based in the data are far more useful in terms of understanding wood property variation within loblolly pine, compared to data collected on a relatively small number of trees or even individual trees as with most studies of wood properties such as pulp yield and lignin.

Development of NIR systems now allows the collection of NIR data at higher spatial resolution. Recent studies have used either fiber optic probes [11, 37] or NIR-HSI [32, 33, 59, 60] with both approaches utilizing linear translation systems to collect data across samples. Both permit the investigation of within-tree variation of wood properties at the ring, or within-ring level and the production of more detailed maps [60]. The system utilized by Meder et al. [37] and Downes et al. [11] allowed wood property estimates at 1-mm resolution; however, very precise identification of NIR measurement points was required (it was achieved in their research by integrating a NIR probe with the SilviScan system). The resolution of NIR-HSI measurements is instrument specific. For example, [59] reported a resolution of approximately 2 mm, whereas Ma et al. [32] collected data at much higher resolution (156 μm). Whether there is value in utilizing within-ring data for mapping the variation of wood properties within-trees (owing to the difficulties of managing very large data sets and ease of producing models at the ring level) remains to be determined. A higher-resolution system does allow for detecting narrow rings, which could be determined with the settings used by Ma et al. [32] (156 μm) but not in the case of [59] (2 mm). NIR-HSI collects images and data collection is rapid; subsequently, many samples can be scanned in a day compared to the use of a fiber optic probe where spectra are collected in incremental steps. While data collection is greatly simplified, the storage and processing of data becomes more computationally intensive.

The models presented herein are the first nonlinear models developed for pulp yield and lignin content for loblolly pine to be reported in the literature. The models could be integrated into growth and yield prediction systems to estimate these properties for different scenarios (e.g., clear cut, thinning, base versus tops of trees, etc.). More research is needed to determine whether these models are applicable beyond the range of the data. The trees for this study were obtained from southwest Georgia, and likely, they do not represent the wood properties variation

throughout the US south [9, 24]. Due to advancements in NIR-HSI, we anticipate that within-tree models for lignin content will be developed more frequently than in the past; however, the same cannot probably be said for pulp yield as few laboratories are focused on generating this information.

The pulp yield model has a similar shape as the model for Norway spruce by Molteberg and Høibø [40] but the values are very different. These authors reported pulp yield values of 37% at the pith compared to the 55% found here; differences likely related to the target Kappa of 27–30 used by Molteberg (2004) compared to the Kappa 109 targeted here. As Kappa number decreases, chemical usage increases and therefore residual lignin content decreases which, in turn, results in lower pulp yields.

Via et al. [63] reported Klason lignin values for longleaf pine at varying heights within ten trees; these data indicate that in the corewood lignin is approximately 31%, and in outerwood lignin decreases to approximately 27%. The model presented here is mostly in agreement with this result for outerwood, although we found higher lignin contents near the pith (33%). This could be due to species differences between loblolly pine and longleaf pine, differences in sites, or indicative of growth rate differences between the trees in the respective studies, among other site factors. The differences could also be due to the prediction of lignin using the spatial-interpolation approach deployed here as [63] measured lignin of specific rings.

An interesting observation is the similarity of maps reported for pulp yield and lignin to other loblolly pine wood property maps [9, 42, 54, 55, 57]. It is possible that separate maturation processes influence radial (corewood to outerwood) and axial (juvenile to mature) development in such a way that properties may be weakly related but still have similar internal trends. Discussion of processes is beyond the scope of this manuscript, but more information is needed from a broad sample size where the full suite of properties is measured at the same spatial scale to more clearly indicate similarities or differences between property maps.

4 Conclusions

The within-tree variation of pulp yield and lignin content in loblolly pine trees aged 13 and 22 years was examined utilizing wood property data obtained using the spatial-interpolated approach described in Schimleck et al. [56]. This methodology used whole-tree wood property and NIR spectral data (originally collected in 10-mm increments at multiple radial positions and heights but basal area weighted to provide a single spectrum per tree) to successfully generate calibrations for pulp yield and lignin

content. The models estimated both properties in 10-mm increments with these data used to develop maps depicting pulp yield and lignin content variation for the trees at both ages. The radial trends observed for pulp yield (increase) and lignin (decrease) were consistent with expectations for loblolly pine owing to maturation. Radial variation in properties was also modeled using mixed-effects models.

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Declarations

Conflict of interest The authors declare no conflict of interest.

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