Effects of loblolly pine wood and pulp properties on sheet characteristics

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ABSTRACT: The effects of wood properties on the strength of bleachable and linerboard grade kraft pulps from 13-year-old loblolly pine (*Pinus taeda*) trees were investigated. Eighteen trees were selected based on breast height wood cores to represent specified ranges of specific gravity and lignin content. Air-dry density and stiffness (modulus of elasticity [MOE]), tracheid coarseness, radial diameter, tangential diameter, specific surface area, wall thickness, and microfibril angle (MFA) were estimated using SilviScan wood analysis technology and near infrared reflectance (NIR) spectroscopy. NIR spectra collected in 10 mm sections from the surface radial strips correlated very well with air-dry density, MFA, MOE, and tracheid wall thickness and were used to develop whole tree predictions. In addition, chemical composition, fiber properties, and handsheet strength were measured for both pulp grades. Statistical analysis indicated that wood density, wood fiber coarseness, and pulp fiber length had the greatest effects on sheet properties.

Application: The information from this study can help forest geneticists determine which traits to focus on in breeding programs that aim to increase kraft pulp yields while obtaining superior fiber properties.

Optimal selection of tree genotypes is a vital requirement for forest products companies to stay competitive in the global market and to improve the value of their existing products. A prerequisite for this is a good understanding of the effects of wood properties on pulp and paper characteristics. The objective of this study was to determine the effects of wood chemical composition and wood specific gravity (SG) on pulp yield and sheet properties. These effects have not been studied previously in a way that allowed each to be assessed independently of the others [1].

Recognizing the need for greater forest productivity, many companies have embarked on detailed studies of intensively managed pine plantations. The increase in growth rates of these stands has been impressive; however, much of this volume increase occurs in the juvenile wood. Because trees from fast growing plantations are harvested earlier, they have greater proportions of juvenile wood, which tends to lower the average density and increase the average lignin content in a mill's fiber supply. This underlines the need for an improved understanding of the yield and properties of pulp from such rapidly grown trees.

Knowing the relationships among wood properties, pulp yield, and pulp properties also would afford the opportunity to evaluate biological technologies such as cloning, enhanced selection methods, molecular breeding, and genetic engineering. Such technologies offer the possibility of producing trees with higher growth rates and fibers tailored to specific processing methods and products.

A previous study indicated that significant improvements in mill profitability could be achieved by decreasing wood lignin content or increasing wood SG [2]. However, the profit increases resulting from higher SG wood relied on relationships between SG and pulp yield obtained in studies that used juvenile early wood and mature late wood to expand the SG range on which predictive equations were based, and the lignin contents were unknown. This lack of knowledge about the effect of lignin content on pulp yield at different wood densities limits the applicability of these previous results. Thus, we sought to determine how whole tree wood density and wood chemical composition independently and together affect pulp yield in trees of a given age. With this information, forest geneticists can make better decisions about which traits to focus on in breeding programs that aim to increase kraft pulp yields, a crucial determinant of mill profitability.

Earlier, we reported the results of a study of the effects of wood SG and chemical composition on the yield of pulp from loblolly pine [1]. The present work extends that study to their effects on pulp and paper properties.

For thin-walled softwood species such as black spruce, microfibril angle (MFA) is the principal determinant of maximum fiber and sheet tensile strength [3,4]. Actual fiber tensile strength is reduced by degradation of cellulose during pulping [5]. Little information is available on the effect of MFA on fiber and paper properties in loblolly pine, which is a thick walled species. Courchene found that MFA is a major determinant of sheet zero-span tensile and other properties, in a study of loblolly pine trees of similar SG, but did not consider wood composition differences [6]. Wimmer found a significant effect of MFA on tensile strength and bending stiffness for *Eucalyptus globulus* after effects of density and fiber length were taken into account [7].

Species, growing conditions, genetics, and harvest age all affect pulp fiber and, consequently, sheet properties. Several studies have been published on the wood characteristics of loblolly pine as they relate to fiber and sheet properties, with particular emphasis on the differences between mature and juvenile wood [8-10]. However, our knowledge of differences in juvenile wood is much more limited.

The analysis of wood properties, previously a time-consuming task, has been considerably improved with automatic or semiautomatic systems such as the SilviScan system developed by CSIRO. This type of analysis system provides rapid measurements of density and MFA, as well as predicted values of tracheid coarseness, specific surface area, and wall thickness in wood samples [11]. Kibblewhite correlated SilviScan-determined wood properties to fiber and sheet properties and showed that the SilviScan-determined properties were good predictors of pulp and sheet properties for radiata pine [12,13]. Schimleck showed that NIR measurements can be correlated to SilviScan tracheid properties for loblolly pine samples [14,15]. In the study reported here, calibrations were developed with SilviScan data and used to predict whole tree average wood properties.

METHODS

Tree selection; pulping

Initially, 13-year-old loblolly pine (*Pinus taeda*) trees, grown as part of a genetic breeding program in southwest Georgia, were selected to represent a range of specific gravities and lignin contents [2]. For the earlier study, samples of wood from each tree were analyzed for chemical composition and then pulped with the kraft process to target kappa numbers of 30 and 100; pulp yields were determined and correlated with wood composition and near infrared reflectance (NIR) measurements. For the present study, each pulp was refined to several different freeness levels, handsheets were made, and the physical properties of the handsheets were determined.

Pulp chemical composition

Concentrations of sugars (arabinan, galactan, glucan, mannan, and xylan), cellulose, hemicellulose, Klason lignin, and acid soluble lignin were measured in each of the 72 pulp samples (18 trees × two samples/condition × two kappa targets). The carbohydrate compositions were determined by high performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) analysis, and lignin concentrations were determined by the Klason method [16]. Cellulose concentration was determined by calculation (cellulose = glucan-1/3*mannan) [17]. Hemicellulose concentration was calculated as the sum of arabinan, galactan, xylan, and glucomannan, plus glucan minus cellulose. We applied some of the methods proposed in the literature to the data to determine pulp yield from carbohydrate composition and found some agreement with the measured yield, but the results showed significant variation.

Pulp and paper properties

For each grade, one pulp from each duplicate set was selected and refined in a Valley beater (36 pulps total). Samples were refined to four CSF levels and made into handsheets at a basis weight of 162 g/m² for the linerboard grade and 60 g/m² for the bleachable grade according to TAPPI T205 "Forming handsheets for physical tests of pulp." Linerboard sheets were tested for tensile (TAPPI T494 "Tensile properties of paper and paperboard"), burst (T807 "Bursting strength of linerboard"), and compression strength (T826 "Short span compressive strength of containerboard"). Bleachable grade sheets were tested for tensile (T494), tear (T414 "Internal tearing resistance of paper [Elmendorf-type method]"), and zero span tensile strength (T231 "Zero-span breaking strength of pulp [dry zero-span tensile]"). Each pulp was analyzed for fiber coarseness and length distribution using a fiber quality analyzer (FQA).

Data analysis

For each pulp grade, statistical software was used to perform multiple linear regression analyses to evaluate a large number of potentially important independent variables. Independent variables were coded to have values from approximately -1 to approximately +1. Preliminary analysis determined which variables had significant effects on sheet strength at the 95% confidence level. Wood chip specific gravity, pulp chemical composition, fiber length, fiber coarseness, and SilviScan wood properties were evaluated.

NIR analysis

For each of the 18 trees sampled, discs were taken at several heights up the tree. Pith-to-bark radial strips were cut from each disc and NIR spectra were collected from the radial surface of each strip in 10 mm increments. A total of 191 strips were analyzed and 1114 NIR spectra collected using an NIR spectrometer. A subsample of 36 strips, representing three height levels (base, 15%, and 30% of total tree height), were selected and analyzed at CSIRO Forestry and Forest Products in Australia. Air-dry density, MFA, and tracheid radial and tangential diameters were measured. Stiffness (MOE) and tracheid coarseness, specific surface area, and wall thickness were determined using methods described by Evans [18,19].

NIR calibrations for density, MFA, and stiffness and tracheid properties (coarseness, wall thickness, etc.) were obtained using Unscrambler v. 9.2 software (Camo AS, Norway), the SilviScan data, and the corresponding NIR spectra. The calibrations were then used to predict the wood properties of each of the 191 strips in 10 mm increments [20]. Based on the NIR-predicted wood properties, basal area-weighted averages were determined to obtain disk averages. Each disk average was used to determine a volume-weighted average of each wood property for each tree. The weighted, whole tree wood property data were used in the regression analysis with sheet properties.

RESULTS AND DISCUSSION

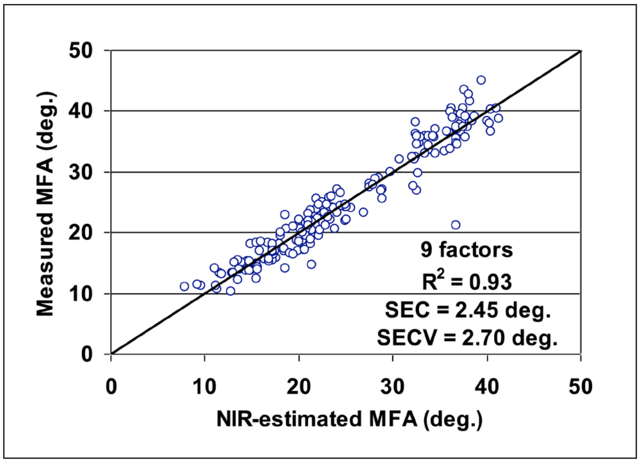
NIR analysis and estimation of whole tree wood properties

NIR spectra collected in 10 mm increments from radial strips and SilviScan air-dry density, MFA, stiffness, and wall thickness data matched well (**Table I**, **Figs. 1** and **2**). Unscrambler software was used to determine factors for each calibration, including the standard error of calibration (SEC) and standard error of cross validation (SECV) (Table I). These calibrations were used to predict the wood properties of the 191 strips in 10 mm increments. Based on the predicted wood property data, volume-weighted, whole tree values were calculated for MFA, wall thickness, density, MOE, coarseness, radial diameter, specific surface area, and tangential diameter (**Table II**).

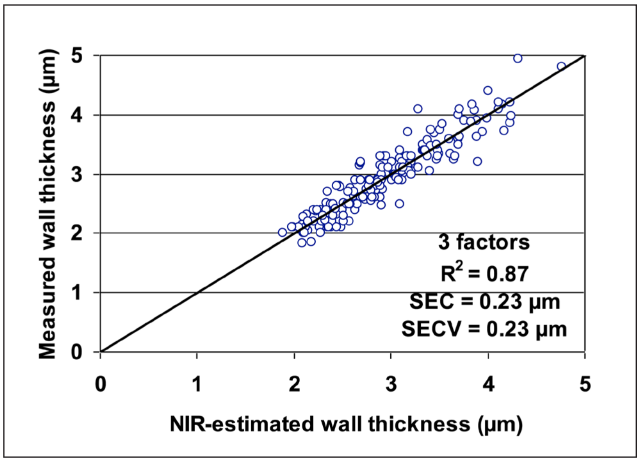
Effect of wood properties on sheet properties

Each pulp was refined to four freeness levels, ranging from about 250-650 CSF. The values for each strength property at 500 CSF were determined and used for the regression analysis. Multiple regression analysis was used to identify statistically significant parameters affecting sheet strength. Independent variables included the following:

- carbohydrate and lignin compositions of the wood and the pulp samples
- volume-weighted whole tree wood properties (density, coarseness, wall thickness, specific surface area, MOE, and MFA) (Table II)
- fiber properties (coarseness and length-weighted fiber length [LWFL]) from FQA
- a chip based estimate of wood specific gravity



1. Relationship between measured microfibril angle (MFA) and near infrared reflectance-estimated MFA for the **Pinus taeda** ***calibration samples.***



2. Relationship between measured wall thickness and near infrared reflectance-estimated wall thickness for the **Pinus taeda** ***calibration samples.***

Wood Property	No. of Factors	R^2	SEC	SECV
Density (kg/m³)	3	0.85	44.03	45.32
Microfibril angle (degree)	9	0.93	2.45	2.70
Stiffness (GPa)	9	0.94	0.98	1.15
Coarseness (µg/m)	3	0.73	43.0	43.74
Specific surface area (m²/kg) surface (m²/kg)	3	0.82	20.61	21.05
Radial diameter (µm)	10	0.56	1.62	1.83
Tangential diameter (µm)	10	0.75	1.11	1.31
Wall thickness (µm)	3	0.87	0.23	0.23

I. Calibration statistics between near infrared reflectance spectra and SilviScan data.

Tree	Density (kg/m³)	Microfibril Angle (degree)	Stiffness (GPa)	Coarseness (µg/m)	Specific Surface Area (m²/kg)	Radial Diameter (µm)	Tangential Diameter (µm)	Wall Thickness (µm)
5	619	21.7	10.8	507	248	32.0	28.9	3.4
14	611	19.2	12.2	497	250	30.7	28.0	3.3
16	551	22.2	10.1	469	276	30.5	27.5	3.0
19	571	19.1	11.8	478	271	30.4	28.1	3.1
20	516	19.9	10.3	450	292	30.7	28.9	2.8
23	517	19.1	10.8	450	289	31.0	28.4	2.8
24	535	22.4	9.4	448	275	29.9	27.4	2.9
26	529	18.6	10.8	445	283	31.2	29.3	2.9
44	557	21.2	10.4	470	266	30.2	27.8	3.0
58	491	23.4	7.8	416	302	31.9	29.2	2.6
63	496	20.5	9.3	437	291	30.8	27.9	2.7
64	529	21.8	9.7	431	287	29.9	27.9	2.8
67	538	18.8	11.9	454	283	30.4	28.6	2.9
71	496	21.2	9.8	428	302	30.4	27.9	2.7
84	549	23.8	9.4	447	286	30.4	27.8	2.9
92	514	20.7	9.7	446	292	31.6	28.9	2.8
99	498	19.5	9.8	427	296	30.8	28.2	2.7
102	565	23.2	9.3	465	271	31.4	28.5	3.1
Average	538	20.9	10.2	454	281	30.8	28.3	2.9
Standard deviation	36	1.63	1.05	23	15	0.60	0.56	0.20
Coefficient of Variation	0.07	0.08	0.10	0.05	0.05	0.02	0.02	0.07

II. Whole tree average property data.

Quadratic and cross-product terms also were included. Multiple combinations of variables were tried and only variables that were significant at the 95% confidence level were retained. Linerboard and bleachable grade data were analyzed separately. Best fit models were determined by the R^2 value.

Independent variables for regression analysis of the linerboard grade (100 kappa) pulps at 500 CSF were defined as follows:

- WD = [(Whole tree wood density in kg/m³) - 539.9]/64.3
- LWFL = [(Pulp length-weighted fiber length in mm) – 2.43]/2.375
- C = [(Whole tree coarseness in µg/m) – 453.6]/45.2

The linerboard analysis provided the following equations:

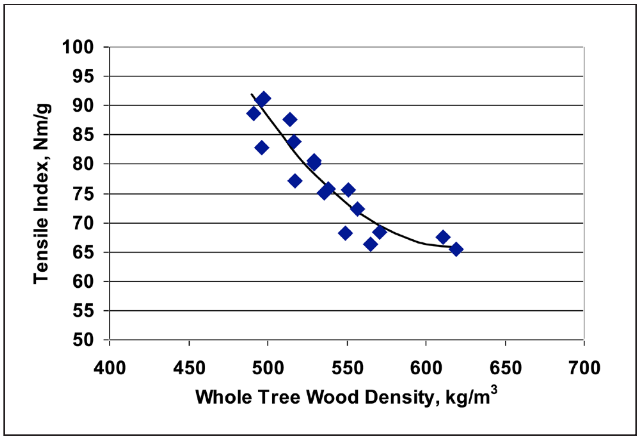
- Sheet density (g/cm³) = 0.631 – 0.112(WD) – 0.0438(LWFL) + 0.0473*(WD)² [R^2 = 0.88] (1)
- Tensile index (Nm/g) = 75.6 – 16.0(WD) + 6.58(WD)² [R^2 = 0.87] (2)
- STFI index (Nm/g) = 30.93 – 3.51(C) [R^2 = 0.84] (3)
- Burst index (kPa.m²/g) = 5.99 – 0.943(WD) [R^2 = 0.74] (4)

Equations for the bleachable grade (30 kappa) pulps at 500 CSF included the following:

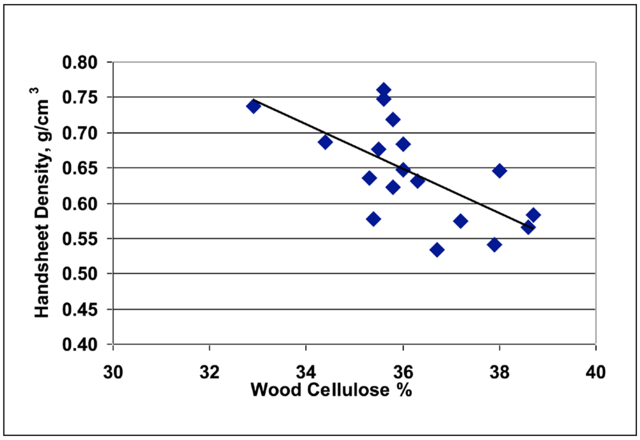
- Sheet density (g/cm³) = 0.628 – 0.0503(WD) [R^2 0.65] (5)
- Tensile index (Nm/g) = 92.53 – 8.48(WD) + 8.18(LWFL) [R^2 = 0.66] (6)
- Tear index (mN.m²/g) = 13.04 + 3.92(WD) – 1.23*(WD) ² [R^2 = 0.85] (7)

The equations indicate strong correlations of specific sheet properties with whole tree wood density (30 and 100 kappa), coarseness (100 kappa), and pulp fiber length (30 and 100 kappa). In agreement with the results reported for radiata pine, wood density was the best predictor of sheet properties for loblolly pine. Lower specific gravity wood contains greater proportions of thinner walled fibers that more readily collapse, resulting in increased bonding (tensile, STFI, burst) and higher sheet density, as reflected in the above equations and in **Figs. 3** and **4**, which show curvilinear relationships with measured whole tree wood density.

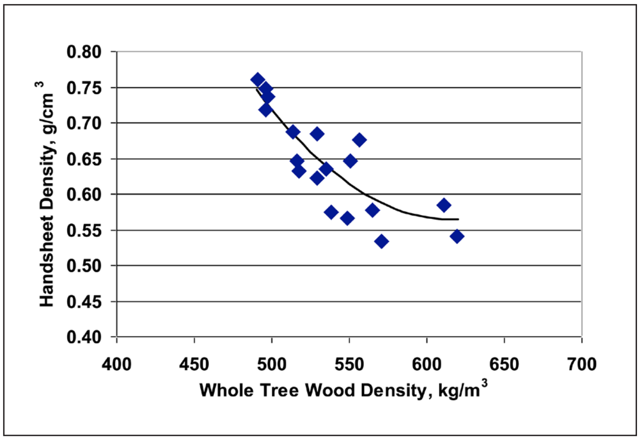
Kibblewhite reported a highly significant but weak corre-



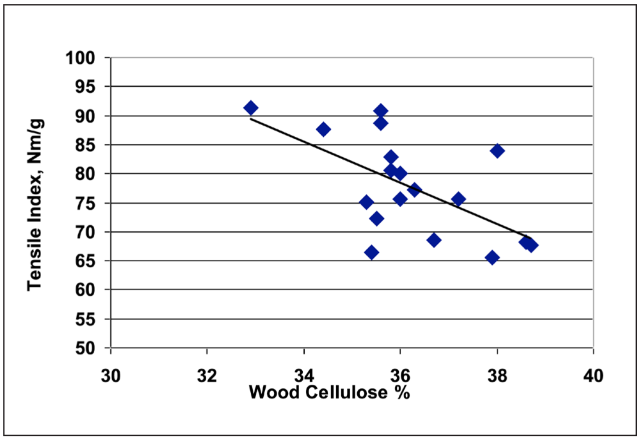
3. Tensile index at 500 mL CSF versus whole tree wood density, 100 kappa.



5. Handsheet density at 500 ml CSF versus wood cellulose content, 100 kappa.



4. Handsheet density at 500 ml CSF versus whole tree wood density, 100 kappa.



6. Tensile index at 500 ml CSF versus wood cellulose content, 100 kappa.

lation between wood glucose content and sheet density, tear, stretch, and tensile energy absorption (TEA) [12]. Here, wood chemical composition was correlated more weakly with sheet properties (100 kappa pulps [at 500 CSF]) and hence does not appear in Eqs. (1) and (2) (**Figs. 5** and **6**). For loblolly pine, competing effects at higher wood cellulose content might account for the differences between species. These effects might include higher fiber strength, leading to higher sheet tensile, but possibly higher fiber wall thickness, leading to less bonding.

Whole tree volume–weighted average MFA did not correlate significantly with sheet tensile properties, as was expected. Kibblewhite's linear regressions showed that MFA (13- and 15-year-old radiata pine trees) had a significant influence on tear index, tensile index, and tensile stiffness index, and less effect on stretch and TEA index, at each of two different refining levels [12]. One possible explanation for the lack of significant correlation of MFA with sheet tensile strength in the current work is that the range of volume-weighted whole tree MFA was relatively narrow (18.6° to 23.8°) compared with that found in previous studies [6,12].

None of the wood properties correlated with zero span tensile strength. We also did not observe a strong correlation between wood and pulp fiber coarseness, possibly because of the variation in the pulp coarseness determinations.

SUMMARY AND CONCLUSIONS

The handsheet properties of bleachable grade (kappa 30) and linerboard grade (kappa 100) laboratory pulps made from 13-year-old loblolly pine (*P. taeda*) trees representing different combinations of wood specific gravity and cellulose:hemicellulose:lignin ratios (lignin contents of 28.6-32.2 and specific gravities of 0.39-0.55) were correlated with whole tree volume-weighted estimates of wood properties obtained from NIR and pulp properties. The effects of wood properties (chip specific gravity, chemical composition, microfibril angle) on kraft pulp physical properties from loblolly pine pulpwood grown in Georgia were quantified.

The results of this study can be summarized as follows:

- For linerboard grade pulps, tensile strength index and burst index correlated most strongly with the inverse of wood density. For STFI compressive strength, the strongest liner grade correlation was with the inverse of whole tree coarseness.

- For the bleachable grade pulps, tensile strength index correlated with fiber length and inversely with wood density.
- The lack of correlation of other wood properties (e.g., whole tree MFA) with sheet properties is believed attributable to the database range for specific properties. Subsequent work by the authors with an expanded database is expected to provide additional insight into the effect of wood properties on sheet properties [21].
- NIR spectra collected in 10 mm sections from the surface radial strips correlated very well with air-dry density, MFA, MOE, and tracheid wall thickness. Although some properties such as basic density and pulp yield can be estimated on a whole tree basis relatively easily from a composite chip sample, other properties such as MFA and wall thickness cannot be estimated from chips and require intensive sampling to obtain a reliable whole tree estimate. Based on NIR predicted data average volume-weighted, whole tree wood properties were determined.

ACKNOWLEDGEMENTS

The authors thank the State of Georgia Traditional Industries Program for funding this work and International Paper for providing the trees and the initial analyses. The work of Blair Carter, Kathleen Patterson, and Michael Buchanan of at the Institute of Paper Science and Technology also is appreciated. **TJ**

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ABOUT THE AUTHORS

We choose this topic to research after we identified the need to understand the effect of wood properties (lignin content and specific gravity) on pulp yield and sheet properties. Earlier, we had reported the results of a study of the effect of wood specific gravity and chemical composition on the yield of pulp from loblolly pine.

The multiple linear regression analysis was challenging, but Tom McDonough and Chuck Courchene were able to use a good statistical approach to identify significant variables. We found that a larger database would be needed to fully sort out some of the parametric effects. The lack of correlation of some specific wood properties (e.g., zero span tensile strength and MFA) with sheet properties was unexpected.

With the information from this study of 13-year-old trees, in combination with similar information about trees of different ages, forest geneticists can make better decisions about which traits to focus on in breeding programs that aim to increase kraft pulp yields while obtaining superior fiber properties – crucial determinants of mill profitability.

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