Effects of loblolly pine tree age and wood properties on linerboard-grade pulp yield and sheet properties: Part 1 - Effects on pulp yield

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ABSTRACT: Results are reported on the relationships of loblolly pine tree age and wood characteristics and the yield of pulp obtained when the trees were chipped and pulped by the kraft process. Eighteen 13-year old and 18 22-year-old loblolly pine trees were selected to represent specified ranges of specific gravity and lignin content. The trees were further characterized by chemical analysis and near infrared spectroscopy before kraft pulping. The resulting pulps were characterized by measurements of yield and chemical analysis. Multiple regression analysis was used to identify wood characteristics that most influenced pulp yield and to derive equations relating pulp yield to tree age, specific gravity, and wood chemical composition. In addition, near-infrared spectroscopy calibrations were developed to allow prediction of pulp yield from analysis of wood. The results showed that wood specific gravity cannot be used to predict the yield of linerboard-grade pulp from trees of either age. The yield of pulp from 13-year-old trees can be predicted from the amounts of xylan and lignin present in the wood. The yield of pulp from 22-year-old trees decreased with increasing lignin content but was unrelated to xylan content. Thirteen-year-old trees had significantly higher xylan content and the excess xylan was lost during pulping. Regression equations were developed for estimating pulp yields from 13- and 22-year-old trees, given their xylan and lignin contents. Another equation derived from the combined data for trees of both age classes will be useful for predicting yields from trees of other ages, if it is assumed that the effect of tree age is linear.

Application: Changes in wood composition that are achievable through selective breeding programs or genetic engineering can significantly improve the value of the pulp and paper made from the wood, while simultaneously decreasing production costs.

Advances in forest genetics and forest management technology have put changes in wood composition within the reach of foresters. Given appropriate information on relationships among tree age, wood properties, pulp yield, and pulp properties, foresters will be in a position to grow trees for lower-cost production of pulp and paper with optimal performance characteristics. We undertook a project to identify and quantify such relationships, making use of instrumental methods (near infrared spectroscopy [NIR] and a wood scanning technology [SilviScan]) to predict pulp yield and paper quality characteristics. Traditionally, such predictions have required expensive and time-consuming physical testing of paper samples and elaborate microscopic examination of wood samples. In Part 1, we describe the work that focused on pulp yield. An upcoming Part 2 will report the results of studies to relate tree age and wood characteristics to fiber and sheet properties. We expect the combined results of the study to make it possible to identify superior planting and harvesting strategies for improved product quality and reduced paper production costs.

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. Logs cut from 13-year-old and 22-year-old trees were chipped and the chips were used to prepare kraft pulps in laboratory digesters. The pulping conditions were chosen to simulate commercial practice for production of linerboard-grade pulps with kappa nos. of 100–110. Samples of the chips and pulps were analyzed for chemical composition and pulp yields were determined.

The results reported here for the 13-year-old trees were included in an earlier publication that dealt only with trees in that age class [1]. Here, our emphasis is on the effect of tree age, as evaluated by comparison of the behaviors of 13- and 22-year-old trees.

BACKGROUND

Wood specific gravity and pulp yield

The current work was motivated, in part, by a desire to test the hypothesis that pulp yield increases with increasing wood specific gravity. Under that hypothesis, pulping wood of higher specific gravity is expected to significantly increase the net profit earned by a mill making linerboard from southern pine [2]. The literature contains frequent references to a positive link between specific gravity and pulp yield. In terms of pulp yield per unit volume of wood, there is little doubt that wood of higher specific gravity gives more pulp per unit of digester volume. However, there is less convincing evidence that increasing specific gravity increases the weight of pulp obtained by pulping a given weight of wood.

Perhaps the strongest evidence is provided by studies showing that, when late wood and early wood are separated before pulping to similar kappa numbers, the yield of pulp from late wood is often higher. Watson and Dadswell [3] pulped loblolly pine early wood (sp. gr. 0.329) and late wood (sp. gr. 0.558) to similar kappa numbers, obtaining yields of 50% and 58%, respectively. Barefoot, Hitchings, and Ellwood [4] separately pulped core wood (sp. gr. 0.34–0.46) and outer wood (sp. gr. 0.41–0.53) of four loblolly pine trees to a permanganate number of approximately 36. Three of the four trees

Tree No.	Tree Age yr	Wood Properties							
		Wood Sp. Gr.	Total Lignin (IPST) %	Cellulose (IP) %	Arabinan %	Galactan %	Glucan %	Xylan %	Mannan %
8	22	0.50	31.2	39.4	1.3	3.6	39.4	5.9	8.7
14	22	0.45	n.d.	41.2	n.d.	n.d.	n.d.	n.d.	n.d.
15	22	0.49	32.6	37.9	1.3	3.8	42.6	6.6	10.6
24	22	0.53	29.1	41.5	1.3	2.5	43.1	6.7	11.0
28	22	0.47	31.6	39.2	1.2	3.3	39.4	5.7	8.4
29	22	0.47	30.3	37.8	1.2	3.5	39.1	5.9	9.2
30	22	0.47	32.8	37.7	1.2	3.6	43.4	6.6	10.5
35	22	0.52	30.4	36.8	1.3	2.4	44.1	6.4	11.1
37	22	0.54	29.3	40.0	1.2	1.6	40.0	6.3	10.9
45	22	0.48	29.1	41.0	1.2	2.0	40.9	5.4	9.5
46	22	0.51	31.2	38.0	1.4	2.4	43.8	6.6	11.0
55	22	0.51	29.7	40.7	1.3	2.1	43.3	6.6	11.3
56	22	0.51	29.7	39.3	1.3	2.6	43.6	6.6	11.2
58	22	0.51	30.4	40.0	1.1	2.6	40.9	5.2	9.4
72	22	0.53	30.3	40.2	1.1	2.4	38.5	6.4	9.9
75	22	0.52	30.6	38.0	1.1	2.4	39.4	5.6	9.1
79	22	0.45	30.3	39.1	1.1	2.5	39.5	4.6	9.8
89	22	0.53	31.9	39.0	1.1	3.5	39.3	6.0	10.6
5	13	0.55	30.3	39.8	1.3	2.3	41.6	6.8	11.0
17*	13	0.50	30.2	39.1	1.4	2.1	40.0	7.1	10.2
16	13	0.46	28.9	39.5	1.3	2.0	39.5	6.6	10.5
19	13	0.49	28.8	37.9	1.2	2.4	40.0	6.4	10.0
20	13	0.41	31.3	39.1	1.3	2.1	41.0	7.0	8.7
23	13	0.42	30.3	40.0	1.2	2.7	39.4	6.2	9.4
27*	13	0.47	31.8	38.7	1.4	3.6	38.7	8.0	9.9
26	13	0.39	29.6	40.4	1.4	1.9	39.0	7.5	9.5
44	13	0.50	30.5	38.8	1.3	1.8	38.8	6.6	9.7
61*	13	0.39	30.6	39.7	1.4	2.0	38.6	7.1	9.1
63	13	0.46	31.7	37.2	1.4	2.7	39.1	7.1	9.8
64	13	0.47	30.4	39.3	1.3	2.2	39.1	6.6	9.2
67	13	0.48	30.4	39.2	1.3	2.1	40.2	6.6	9.0
71	13	0.44	32.1	37.2	1.3	2.7	38.3	7.4	8.0
84	13	0.47	29.4	39.1	1.5	2.1	41.8	7.3	9.4
92	13	0.43	29.7	39.7	1.3	2.0	37.6	6.4	9.7
99	13	0.43	32.6	37.7	1.4	2.6	35.4	7.6	7.5
102	13	0.49	29.3	37.8	1.4	2.4	38.5	7.1	9.3

IPST, IP: Labs where determined
Tree numbers marked with an asterisk have been incremented by 3 to avoid duplication of tree numbers.

I. Properties of wood.

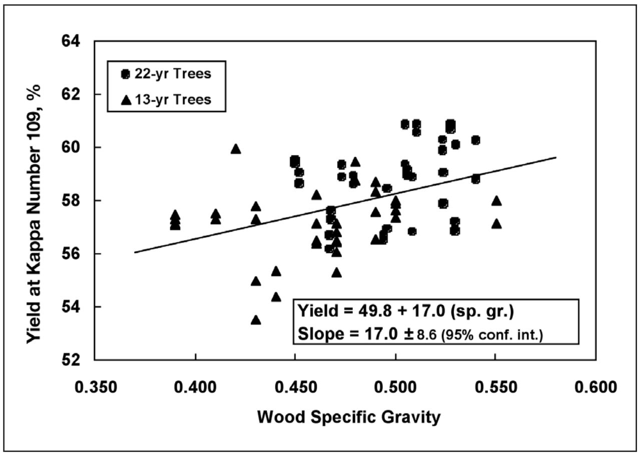
22-Year-Old Trees			13-Year-Old Trees		
Tree No.	Total Yield, %	Kappa Number	Tree No.	Total Yield, %	Kappa Number
8	57.59	101.8	5	56.45	103.1
	56.39	104.4		57.94	108.3
14	60.18	118.4	17*	57.99	114.1
	59.33	114.8		58.28	114.0
15	57.55	115.8	16	55.47	94.5
	57.40	116.4		55.98	89.8
24	60.10	108.8	19	58.46	110.0
	60.59	113.0		58.31	105.4
28	57.87	111.2	20	56.82	104.7
	57.86	113.7		57.16	105.8
29	60.85	121.7	23	58.74	98.3
	61.19	128.3		62.01	101.2
30	57.75	117.9	27*	56.42	118.0
	58.36	127.2		58.39	122.0
35	59.19	110.1	26	56.73	103.8
	58.96	117.9		57.21	109.3
37	61.22	117.0	44	57.70	106.8
	59.91	118.3		57.63	105.3
45	58.40	104.4	61*	55.69	97.0
	58.73	109.9		56.76	102.8
46	59.15	111.2	63	56.68	111.3
	58.30	121.4		55.52	100.4
55	61.14	113.7	64	56.48	108.7
	61.21	112.0		57.41	110.8
56	60.57	106.5	67	58.77	102.7
	59.21	107.6		58.06	103.0
58	58.21	102.7	71	55.19	107.1
	58.03	99.5		52.58	93.6
72	60.58	106.4	84	55.57	104.8
	60.43	106.8		54.72	94.1
75	60.81	113.2	92	56.04	98.0
	60.45	113.6		57.66	107.5
79	59.10	105.2	99	53.36	107.5
	59.01	106.0		55.05	109.4
89	59.39	127.3	102	55.79	93.8
	58.28	120.8		56.02	104.5

Tree numbers marked with an asterisk have been incremented by 3 to avoid duplicate tree numbers.

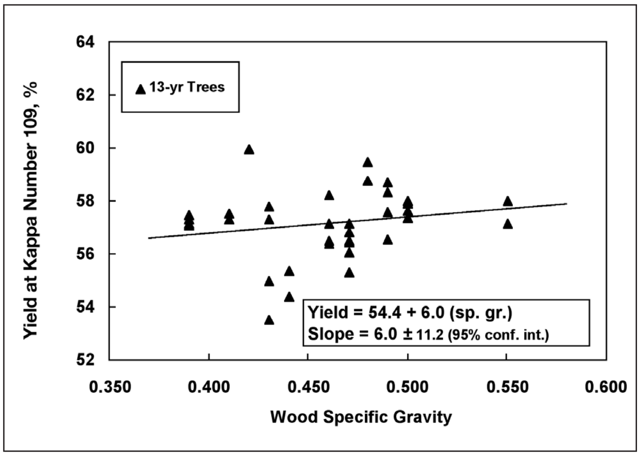
II. Pulp yields and kappa numbers.

exhibited a large yield advantage for outer wood over core wood. These authors later published data from experiments in which the same wood samples were pulped to lower permanganate numbers and the resulting yields were adjusted to kappa no. 25 [5]. The outer wood of one of the four trees gave a much higher yield than core wood from the same tree. Gladstone [6] evaluated kraft pulp yields from early wood and late wood of the mature zones of three loblolly pine trees. In the yield range 45%-55%, late wood fractions yielded 2%-7% more pulp than early wood fractions.

The picture is, however, not quite as clear as the above results would suggest. In the first of the studies cited [2], the authors also pulped early wood and late wood fractions of *P. radiata*. In that instance, the late wood yield was lower than that of the early wood, in spite of the late wood pulp's higher kappa number. Other studies cited previously [3,4] included several examples of outer (higher sp. gr.) wood affording lower pulp yields than low sp. gr. wood from the same tree. In another investigation, Kibblewhite and coworkers [7] pulped chips from 29 *Eucalyptus nitens* trees having wood densities ranging from 390 to 556 kg/m^3 and obtained yields that exhibited no significant correlation with wood density.



1. Apparent correlation of pulp yield with wood specific gravity. Combined data for 13- and 22-year-old trees.

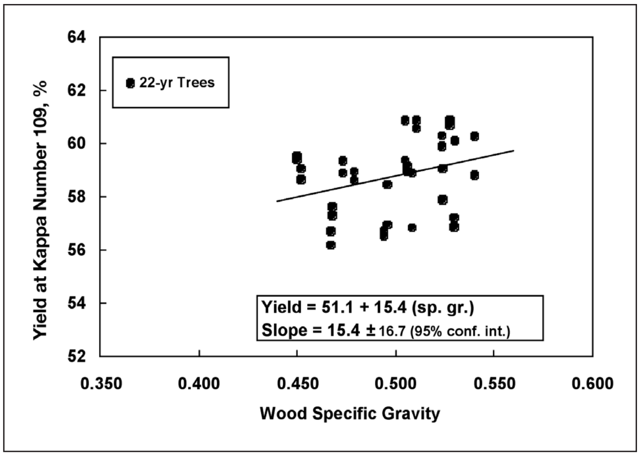


2. Pulp yield from 13-year-old trees versus specific gravity.

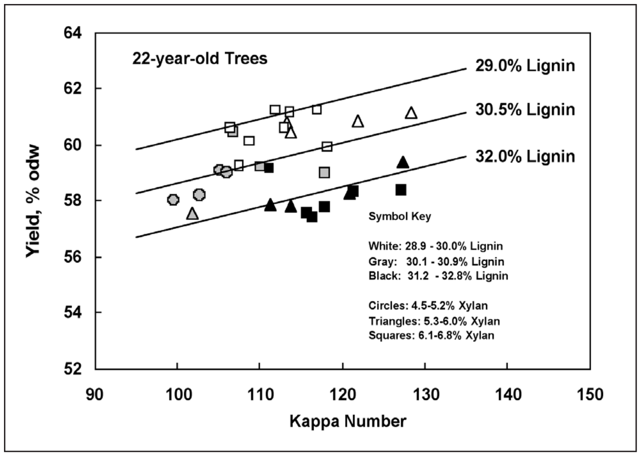
RESULTS AND DISCUSSION

The results of the wood analyses are shown in **Table I**. Chips from each of the 36 trees were subjected to laboratory kraft pulping, each cook being conducted in duplicate (see Experimental). The resulting pulp kappa numbers and yields are shown in **Table II**.

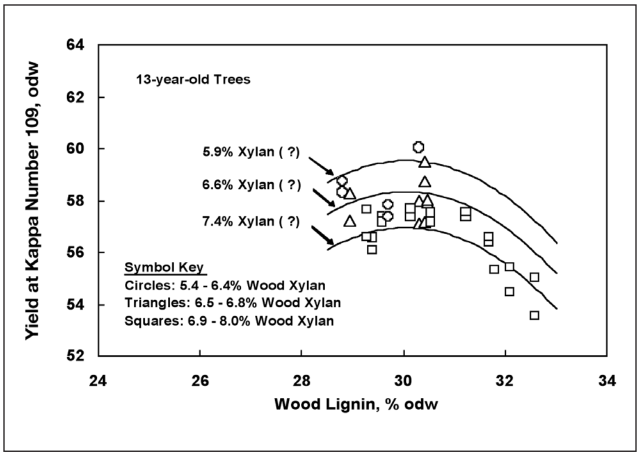
Analysis of the combined yield and kappa data from cooks of the 13- and 22-year-old trees at first seemed to verify the existence of a correlation between specific gravity and yield. Multiple regression analysis was used to obtain a coefficient describing the effect of kappa number on yield (common to



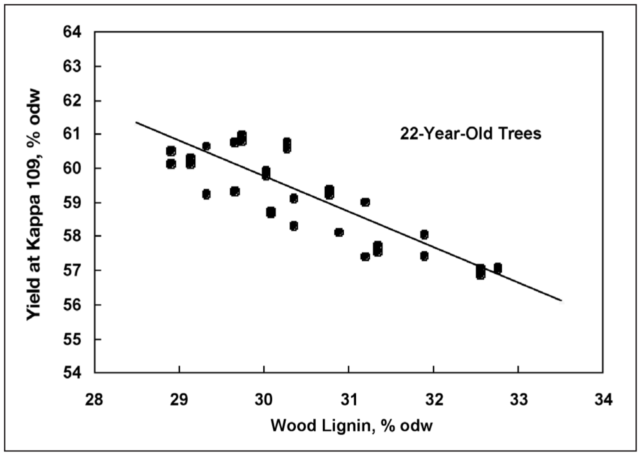
3. *Pulp yield from 22-year-old trees versus specific gravity.*



5. *Effect of wood lignin content and pulp kappa number on pulp yield from 22-year-old trees.*



4. *Effects of wood lignin and xylan content on the pulp yield from 13-year-old trees.*



6. *Effect of wood lignin content on pulp yield from 22-year-old trees at constant kappa number.*

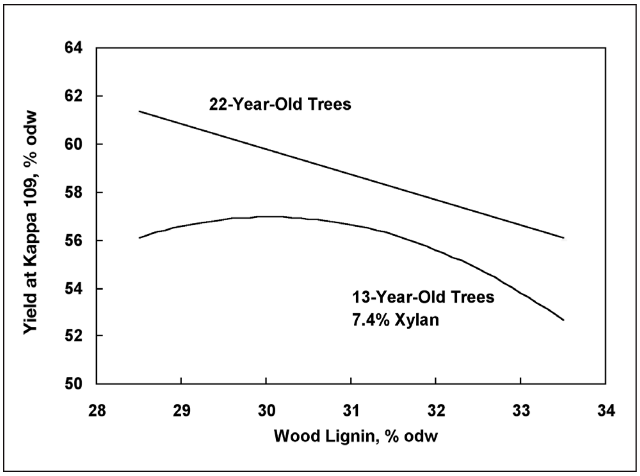
all trees). This coefficient was used to correct all yield measurements to a common kappa no., 109, which was close to the mean kappa number for all cooks. **Figure 1** plots this kappa-corrected yield against wood specific gravity. From a purely statistical standpoint, the correlation it suggests is significant, as evidenced by the fact the 95% confidence interval for the slope of the regression line, 17 ± 8.6, does not include zero. On the other hand, it must be recognized that the effect represented by this slope estimate may not be simply due to specific gravity but could represent the combined effects of tree age and specific gravity. When the data for the two tree age classes are plotted separately, as in **Figs. 2** and **3**, the 95% confidence intervals for both slope estimates include zero, showing that there is little or no effect of specific gravity per se.

Effects of wood composition on pulp yield

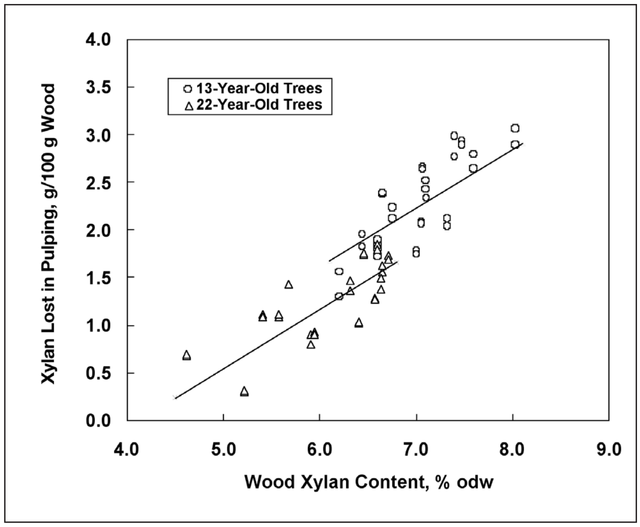
Further analysis showed that pulp yield differences between trees of a given age can be attributed to their differing contents of xylan and lignin. In the case of the 13-year-old trees, multiple regression analysis showed that the xylan effect predominates as long as the lignin content remains below a certain threshold value, as illustrated in **Fig. 4**. The curves shown are plots of the corresponding regression equation (Eq. [1]). The regression equation predicts that 13-year-old trees having a xylan content of only 5.9% may be expected to give a pulp yield that is more than 2% higher (based on o.d. wood weight) than trees having xylan content of 7.4%. For trees having lignin content higher than about 31%, the pulp yield is further reduced by about 1% for each 1% increase in lignin content.

Regression analysis also showed that the yield of pulp from the older trees was unrelated to their xylan content, which was generally lower than in the younger ones. The yield was, however, adversely affected by lignin content in the older trees, as illustrated in **Figs. 5** and **6**, where the lines are plots of the corresponding regression equation.

To facilitate interpretation of the regression equations, and to minimize the likelihood of encountering computational difficulties, the independent variables were coded, (i.e., normalized and scaled) so that the values of each coded variable ranged from approximately -1 to approximately +1. The re-



7. Yield versus wood lignin content: 22-year-old trees and 13-year old trees containing 7.4% xylan.



8. Xylan lost in pulping versus wood xylan content.

sulting equations were then decoded to give equations in terms of the original variables.

Equation (1) describes the pulp yield of 13-year-old trees:

$$Yield_{13} = -271.68 + 0.1323*Kappa - 1.72*Xylan - 21.762*Lignin - 0.3621*(Lignin)^2 \quad (1)$$

for which the coefficient of determination (R^2) = 0.82, s = 0.677, and n = 33. (Yields from tree 23, cook 2; tree 27, cook 2; and tree 64, cook 1 were shown statistically to be outliers and were consequently eliminated from the analysis.)

Equation (1) is an improved version of the corresponding equation reported earlier for the yield of 13-year-old trees [1]. The improvement resulted from the inclusion of a previously unrecognized quadratic lignin term.

Equation (2) for the yield of the 22-year-old trees is:

$$Yield_{22} = 83.26 + 0.0728*Kappa - 1.047*Lignin \quad (2)$$

for which R^2 = 0.70, s = 0.729, and n = 31. (Data from both cooks of tree 14 were excluded because of missing wood lignin content measurements. Also, yields from tree 8, cook 2, and both cooks of tree 45 were shown statistically to be outliers and were consequently eliminated from the analysis.)

Figure 7 uses plots of the previous regression equations, Eq. (1) and Eq. (2), to compare the pulp yields to be expected from trees of the two age classes, assuming that the wood xylan content is 7.4% in both instances. Higher pulp yields are obtained from 22-year-old trees, especially those having relatively low lignin content.

To the extent that the effect of tree age is linear, an equation that includes tree age as a variable could be useful in estimating the yield of pulp to be expected from trees of ages other than those included in this study. Regression analysis of the combined data from both age classes gave Eq. (3):

$$Yield_{13\text{-}22} = -189.75 + 0.779*Age - 0.0646*Age*Lignin + 0.203*Age*Xylan + 16.716*Lignin + 0.108*Kappa - 0.2653*(Lignin)^2 - 4.392*Xylan \quad (3)$$

for which R^2 = 0.87, s = 0.745, and n = 64.

To gain further insight into the reasons for the effect of tree age on pulp yield, the pulp samples were hydrolyzed; the hydrolyzates were then analyzed to determine their individual sugars contents. Inferences could then be drawn concerning the polysaccharide contents of the trees and the pulps made from them. **Table III** shows the results of these analyses.

The xylan content of the wood and pulps was of particular interest, given its effect on the yield of pulp from the younger trees and the lack of a corresponding effect on the yield from the older ones. For each cook, the amount of xylan lost during pulping was calculated and related to the wood xylan content and tree age. The relationship is described by the following regression equation, Eq. (4):

$$Xylan\ Loss = -1.45 - 0.050*Age + 0.618*Xylan \quad (4)$$

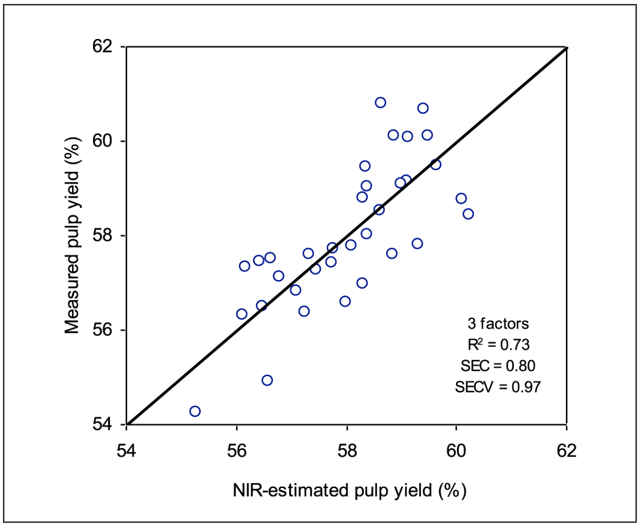
for which R^2 = 0.84, s = 0.28, and n = 62. (Data for four of the 36 trees – trees 15, 16, 19, 89 – were statistically shown to be outliers and were not included when deriving this relationship.)

As illustrated in **Fig. 8**, the younger trees had significantly higher xylan content and the excess xylan was lost during pulping. This accounts for much of the observed effect of tree age on yield. Even in instances where 13-year-old trees had xylan content similar to those of the older trees, the younger trees lost more xylan during pulping than the older ones. This suggests the existence of two different types of xylan, one of

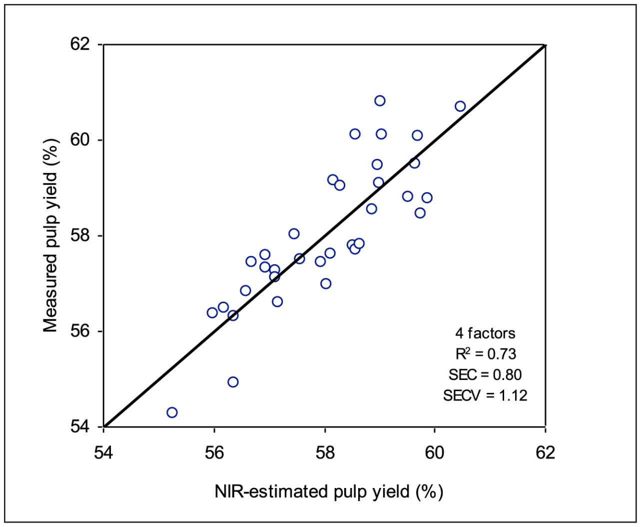
Tree No.	Age	Pulp Properties								
		Total Yield %	Kappa No.	Total Lignin (IPST) %	Cell-ulose (IPST) %	Arabinan %	Galactan %	Glucan %	Xylan %	Mannan %
8	22	57.6	101.8	17.6	61.5	1.1	1.3	63.5	8.9	6.1
14	22	60.2	118.4	20.3	61.7	1.0	1.1	63.4	8.5	5.2
15	22	57.6	115.8	20.6	55.9	0.9	1.3	57.2	6.9	3.8
24	22	60.1	108.8	18.9	62.0	1.1	1.0	63.9	8.3	5.7
28	22	57.9	111.2	19.2	55.1	1.1	1.1	56.6	7.3	4.7
29	22	60.9	121.7	20.0	55.6	0.9	1.2	57.5	8.2	5.5
30	22	57.8	117.9	20.4	59.6	1.0	1.6	61.3	8.2	5.2
35	22	59.2	110.1	18.5	60.6	1.0	0.9	62.4	7.9	5.5
37	22	61.2	117.0	21.1	58.0	1.1	0.8	60.3	8.1	6.7
45	22	58.4	104.4	17.7	57.2	0.9	0.8	58.9	7.3	5.3
46	22	59.2	111.2	18.6	59.7	1.1	1.1	61.5	8.6	5.4
55	22	61.1	113.7	18.3	58.9	1.0	0.8	60.9	8.6	5.8
56	22	60.6	106.5	18.5	62.8	1.0	1.0	64.7	8.7	5.7
58	22	58.2	102.7	18.2	63.8	1.1	1.2	65.8	8.4	6.1
72	22	60.6	106.4	19.5	65.0	1.2	1.2	67.2	8.9	6.7
75	22	60.8	113.2	18.7	54.7	0.9	0.9	56.3	7.4	4.7
79	22	59.1	105.2	21.1	55.6	0.9	1.3	57.1	6.6	4.5
89	22	59.4	127.3	21.0	52.0	0.9	1.5	53.4	7.1	4.1
5	13	56.5	103.1	16.8	59.6	1.2	1.0	61.5	8.0	5.6
15*	13	58.3	114.0	17.8	55.3	1.1	0.9	57.0	7.6	5.2
16	13	55.5	94.5	14.2	60.4	1.1	0.9	62.6	8.0	6.6
19	13	58.3	105.4	16.2	60.7	1.4	1.2	62.5	9.0	5.5
20	13	56.8	104.7	16.6	59.9	1.3	1.3	61.6	9.2	5.1
23	13	58.7	98.3	16.5	58.2	1.2	1.0	60.1	7.9	5.6
25*	13	58.4	122.0	21.0	56.4	1.4	1.3	58.2	8.8	5.5
26	13	56.7	103.8	16.3	57.1	1.0	1.1	58.8	8.0	5.2
44	13	57.6	105.3	17.0	55.0	1.2	0.8	56.8	7.4	5.4
59*	13	56.8	102.8	17.8	59.7	1.3	1.0	61.4	8.4	5.0
63	13	55.5	100.4	17.0	60.7	1.3	1.2	62.5	8.3	5.4
64	13	56.5	108.7	16.6	55.5	1.2	1.0	57.1	8.5	4.7
67	13	58.1	103.0	16.2	60.6	1.2	1.0	62.3	8.1	5.1
71	13	55.2	107.1	16.0	59.3	1.2	1.3	60.8	8.4	4.6
84	13	55.6	104.8	14.4	61.8	1.4	1.1	63.6	9.5	5.5
92	13	56.0	98.0	14.5	60.6	1.3	1.1	62.5	8.0	5.6
99	13	55.1	109.4	16.6	56.4	1.3	1.3	57.9	9.0	4.4
102	13	56.0	104.5	14.4	57.1	1.2	1.2	58.8	8.9	5.1

FQA = Fiber Quality Analyzer; STFI = Trade name for short-span compressive strength tester; TEA = Tensile energy absorption
Tree numbers marked with an asterisk have been incremented by 1 to avoid duplicate tree numbers.

III. Properties of pulp.



9. Measured versus near infrared spectroscopy (NIR)-estimated pulp yield. The calibration used to derive the estimated yields was based on NIR spectra collected from milled wood chips. (SEC = standard error of calibration; SECV = standard error of cross-validation.)



10. Measured versus NIR-estimated pulp yield. The calibration used to derive the estimated yields was based on NIR spectra collected from dried wood chips.

which is more easily removed and which occurs to a greater extent in younger trees.

A similar analysis of lignin loss during pulping gave Eq. (5):

$$\text{Lignin loss} = -0.728 - 0.170{*}\text{Age} + 1.00{*}\text{Lignin} - 0.0614{*}\text{Kappa} \quad (5)$$

for which $R^2 = 0.81$, $s = 0.680$, and $n = 70$.

An unexpected feature of this equation is the inclusion of a statistically significant age term. Because the kappa number test was designed to result in a linear relationship between kappa number and pulp lignin content, the amount of lignin lost during pulping (as calculated from the wood lignin content, the yield, and the pulp lignin content) should be a function of only wood lignin content and pulp kappa number. The appearance of a significant tree age term implies that the proportionality constant relating kappa number to pulp lignin content is different for pulps from trees of the two age classes. This prompted a re-examination of the lignin and kappa number data of Table III. Although plots of kappa number versus pulp lignin content exhibited considerable scatter, zero-intercept regressions of kappa number versus total lignin content for the two different age classes had slopes that differed significantly. The 95% confidence intervals for the slopes of the kappa versus lignin plots were 6.36 ± 0.21 and 5.80 ± 0.15 for the 13-year-old and 22-year-old trees, respectively. For pulps from 13-year-old trees, the average ratio of lignin content to kappa number was 0.157; for pulps from 22-year-old trees, it was 0.172. A *t*-test showed that these two ratios were significantly different at the 99% confidence level. The difference was not attributable to differences in acid-soluble lignin content between the two age classes (0.3%–0.4% for the 13-year-old trees and 0.5%–0.7% for the 22-year-old trees). The ratios of Klason (acid-insoluble) lignin content to kappa number also differed significantly between the two age classes.

NIR calibrations

A subsample of the chips used for pulping was analyzed using NIR spectroscopy, and calibrations for pulp yield (at kappa no. 109) based on dried and milled chips were obtained using partial least squares (PLS) regression. Estimated yields based on the NIR calibration versus measured yields are shown in **Figs. 9** and **10** for the milled and dried wood chips, respectively. The R^2 for both calibrations was 0.73, with the chip calibration having one more factor and a higher standard error of cross-validation (SECV). It is likely that the additional factor accounted for the irregular surface of the chips.

Statistics for the calibrations were reasonable but not as strong as those reported by White and others [1]. A possible explanation is that the calibrations included samples of two different ages, which as demonstrated in this study behave quite differently when pulped. Despite the weaker statistics, the calibrations demonstrate that it is possible to use NIR spectroscopy to estimate the pulp yield of plantation-grown loblolly pine of various ages. It is also interesting that the calibrations based on milled and dried chips had very similar statistics, indicating that both could be used for the estimation of wood properties by NIR spectroscopy.

EXPERIMENTAL

Tree selection and sample preparation

Eighteen 13-year-old and 18 22-year-old *P. taeda* trees were selected from a half-sib progeny trial planted on similar sites at International Paper's (IP's) Southlands facility (Bainbridge, GA, USA). Increment cores removed at breast height were used for the measurement of basic specific gravity, cellulose, and lignin content. Cellulose and lignin content were both estimated by IP using NIR spectroscopy. The trees were selected to represent a range of combinations of specific gravity and lignin content, as Table I shows. Four 0.75-m bolts from each tree were chipped in a 1.2-m Carthage chipper. A composite chip sample from each tree was analyzed for sugar composition by high-performance anion exchange chromatography with pulsed amperometric detection. Klason lignin was determined by TAPPI T222 "Acid-insoluble lignin in wood and pulp" and acid-soluble lignin by TAPPI UM 250 "Acid-soluble lignin in wood and pulp." A subsample was removed for NIR analysis at the University of Georgia.

Collection of NIR spectra

NIR spectra were collected from the whole-tree chip samples in two different ways. Each method is described as follows:

- NIR spectra over the wavelength range 400–2500 nm were collected from each dried whole-tree chip sample using a Foss NIRSystems 6500 (Foss NIRSystems; Laurel, MD, USA) (wavelength range 400–2500 nm) fitted with a large transport module.
- The chips were then milled and NIR spectra of the ground samples were collected using a FOSS NIRSystems 5000 (wavelength range 1100–2500 nm) fitted with a spinning sample module.

Pulping

Chip samples were screened on a Rader trademarked thickness screen. The 2- to 8-mm thickness fraction was retained. Chips were air-dried to provide consistent moisture content and facilitate yield measurements.

Pulping was done with a 1.0 o.d. kg chip charge in a 10L M/K digester (M/K Systems; Peabody, MA, USA) with external liquor circulation and computer control. Duplicate cooks were made for each tree sample. The pulping conditions were 15.7% active alkali, 30% sulfidity, and 500 H-factor. The cooked chips were refined in a 30.5-mm Sprout Waldron refiner at 0.30 mm clearance. The refined pulp was then screened on a flat screen. The total and screened yields were calculated from the screen accept and reject pulp weights. Concentrations of individual sugars (arabinan, galactan, glucan, mannan, and xylan), cellulose, hemicellulose, Klason lignin, and acid soluble lignin were measured for each pulp sample [8]. The carbohydrate compositions were determined by high performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) analysis, while lignin concentrations were determined by the Klason method [9]. Cellulose concentration was determined by calculation as cellulose = glucan – 1/3*mannan [10]. Hemicellulose concentration was calculated as the sum of arabinan, galactan, xylan, and glucomannan, plus (glucan – cellulose).

ABOUT THE AUTHORS

The topic of this paper was identified as a research need by the State of Georgia Traditional Industries Pulp and Paper Program. The study adds to the literature on the relationship between wood type and pulp yield. It differs from previous research by being specific to a given species and to trees of specified age, specific gravity, and lignin content and by employing a modern instrumental technique, NIR spectroscopy.

A challenge for us was to identify which variables were important because there were so many possibilities. We addressed the problem by using an all-possible-regressions approach to multiple regression analysis. The importance of the xylan component was new and unexpected. The absence of a specific gravity effect was also a surprising finding.

Mills could benefit from this information by having access to trees with lignin and xylan content in the desired ranges that result from newly instituted forestry programs. Our next step is to determine the dependence of linerboard physical properties on tree age and wood properties.

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NIR calibrations

Calibrations for screened and total kraft pulp yield (at kappa nos. 30 and 100) were developed using PLS regression with full cross-validation and a maximum of 10 factors (Unscrambler, version 9.2, CAMO AS; Oslo, Norway). Calibrations were developed using second derivative spectra; left and right gap widths of 8 nm were used for the conversion. For the Foss instruments, this limited the wavelength range available for calibration development to 1108–2492 nm (wavelengths in the 400–1100 nm range collected by the FOSS 6500 were not used). The SECV (determined from the residuals of each cross-validation phase), the standard error of calibration (SEC) (determined from the residuals of the final calibration), and R^2 were used to assess calibration performance.

CONCLUSIONS

The specific gravity of wood sampled from 13- or 22-year-old loblolly pine trees cannot be used to predict the yield of linerboard-grade pulp from those trees. The yield of pulp from 13-year-old trees can be predicted from the amounts of xylan and lignin present in the wood. Thirteen-year-old trees having a xylan content of only 5.9% may be expected to give a pulp yield that is more than 2% higher (based on o.d. wood weight) than trees having a xylan content of 7.4%. For trees having lignin content higher than about 31%, the pulp yield is further reduced by about 1% for each 1% increase in lignin content.

The yield of pulp from 22-year-old trees is unrelated to their xylan content, which is generally lower than in the younger ones. Pulp yield is, however, lower for trees of higher lignin content. Thirteen-year-old trees had significantly higher xylan content, and the excess xylan was lost during pulping. This accounted for much of the observed effect of tree age on yield. However, even in instances where 13-year-old trees had xylan content similar to those of the older trees, the younger ones lost more xylan during pulping. This suggests the existence of two different types of xylan, one of which is more easily removed, and the occurrence of a larger amount of the easily removed type in younger trees.

Regression equations have been developed that will allow prediction of pulp yields that can be expected from 13- and 22-year-old trees, given their xylan and lignin contents. Another equation will allow prediction of yields that can be expected from trees of other ages, to the extent that the tree age effect can be assumed to be linear.

The proportionality constants relating kappa number to pulp lignin content were different for pulps from trees of the two age classes. For pulps from 13-year-old trees, the average ratio of lignin content to kappa number was 0.157 ± 0.005; for pulps from 22-year-old trees, it was 0.172 ± 0.004 (95% confidence intervals).

NIR calibrations demonstrate that it is possible to use NIR spectroscopy to estimate the pulp yield of plantation grown loblolly pine of various ages. Calibrations based on milled and dried chips had very similar statistics, indicating that both could be used for the estimation of wood properties by NIR spectroscopy. **TJ**

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