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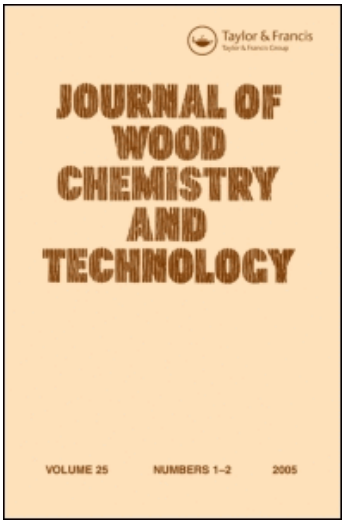

Journal of Wood Chemistry and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597282

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Online publication date: 04 December 2010

To cite this Article Chen, Qing-min , Hu, Zhoujian , Chang, Hou-min and Li, Bailian(2007) 'Micro Analytical Methods for Determination of Compression Wood Content in Loblolly Pine', Journal of Wood Chemistry and Technology, 27: 3, 169 — 178
To link to this Article: DOI: 10.1080/02773810701700810
URL: http://dx.doi.org/10.1080/02773810701700810

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Journal of Wood Chemistry and Technology, 27: 169–178, 2007
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ISSN 0277-3813 print/1532-2319 online
DOI: 10.1080/02773810701700810

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Micro Analytical Methods for Determination of Compression Wood Content in Loblolly Pine

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Abstract: All loblolly pine trees, especially the juvenile portion, contain various amounts of compression wood. The morphological, chemical, and papermaking properties of compression wood are distinctively different from those of normal juvenile wood and mature wood. Compression wood has higher lignin and galactan, but lower cellulose and mannan content, shorter average fiber length, lower fiber width but thicker cell wall, higher fiber coarseness and higher microfibril angles as compared with the corresponding normal wood. Micro analytical methods have been developed to quantitatively determine the percentage of compression wood in an incremental core so as to eliminate the effects of compression wood on the aforementioned properties. This enables accurate quantitative genetic analyses of these properties for tree breeding programs.

Keywords: Chemical composition, compression wood, opposite wood, micro analyses, near infrared spectra

The authors gratefully acknowledge USDA-CSREES (IFAFS grant 2001-52104-11224) and NCSU Forestry Biotechnology Industrial Research Consortium (FORBIRC) for financial support.

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INTRODUCTION

Compression wood is the abnormal wood formed in gymnosperms when it is grown under stress. Formation of compression wood may be stimulated by a combination of several factors such as light, gravity, auxin, compressive stress, and wind action.[1] The morphological and chemical properties of compression wood have been extensively studied and are well known to be distinctively different from those of normal wood.[2] Juvenile wood is the wood formed near the stem center and is formed by a young vascular cambium.[3] Compared to mature wood, juvenile wood has lower wood density, shorter fiber length, higher lignin content, lower cellulose content, and higher compression wood content.[3,4] The percentage of compression wood in juvenile pine is on average about 18% by volume,[5] but can be as high as 44%.[6] Compression wood in softwood is often associated with juvenile wood.[3]

It is generally considered that the juvenile wood is the same as compression wood because both have similar properties: short fibers, larger microfibril angles, higher longitudinal shrinkage, higher lignin content, lower cellulose content, lower cellulose crystallinity, and low pulp yield.[3,7] Our recent studies clearly demonstrate that compression wood is distinctively different from juvenile wood, and that the differences in chemical composition between juvenile wood and mature wood are relatively small.[8–10]

Increasing utilization of rapidly growing plantations in the southeast U.S. is shifting the raw material for solid wood and pulp from predominately mature wood to a greater proportion of short-rotation juvenile wood. Because both juvenile wood and compression wood are generally considered inferior for both pulp and solid wood products, increased usage of juvenile wood (and compression wood) from fast-growing plantation forests can significantly impact industrial production cost and product quality. Consequently, the loblolly pine breeding program at NCSU has started to include wood properties, such as α-cellulose and lignin content, microfibril angle, average fiber length, and so on, in addition to the traditional breeding parameters in the breeding program. However, the impact of compression wood must be eliminated for a meaningful breeding program because the content of compression wood in any sample greatly affects the analytical data of these properties. Thus, there is an urgent need to develop rapid micro analytical methods to determine the compression wood content in each ring of an incremental core.

EXPERIMENTAL

Materials

Three loblolly pine (*Pinus taeda*) trees with significant bends in the stem were harvested from the plantations in North Carolina. The ages of the trees are 35

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years (tree 1), 21 years (tree 2), and 18 years (tree 3). Several 2-inch disks were cut from each stem at the bent section to obtain the maximum amount of compression wood (CW) and opposite wood (OW). Wood meal (40-mesh) was prepared from each ring, using a Wiley mill.

Removal of Extractives

Extractive-free wood meal was prepared by wrapping the wood meal with filter paper and extracting with 2:1 benzene:ethanol overnight in a Soxhlet extractor. After extraction, the wood meal was first air-dried before being dried in a desiccator under vacuum over phosphorus pentoxide.

Determination of Lignin and α-Cellulose

The total lignin content was determined by the modified Klason lignin method,[11] combining both the Klason lignin and acid soluble lignin. For α-cellulose determination, holocellulose was prepared by a micro analytical method modified from that of Browning.[12] Extracted wood meal (100 mg, 40 mesh) was soaked with 4 ml of water in a 10-ml flask (with stopper) placed in a 75°C water bath. To the flask, 0.5 ml 10% sodium chlorite solution and 0.2 ml acetic acid were added 4 times at a 30 min interval. At the end of the reaction, the holocellulose was obtained by filtering and washing the residue in a coarse sintered glass filter with water followed by acetone. The air-dried holocellulose (50 mg OD equivalent) was extracted with 4 ml of 17.5% NaOH solution at room temperature for 30 min, diluted with 4 ml of water and left for another 30 min. The α-cellulose was obtained by filtration using a coarse sintered glass filter, the isolated residue was washed with water, followed by 1% acetic acid, and finally water. The α-cellulose was oven-dried to constant weight and the α-cellulose content determined gravimetrically.

Determination of Monosaccharides

Sugar determination was performed using an aliquot (20 μl) of the filtrate from the Klason lignin using a Dionex A550 Ion Chromatograph equipped with GP 40 gradient pump, EP 40 electrochemical detector, and a post column injection system (Dionex Corporation, Sunnyvale, CA, USA). The system used a Dionex PA1 column and operated at 18°C with an eluent (water) flow rate of 1.2 ml/min and a post column injection of 400 μm of NaOH at a flow rate of 0.15 ml/min. Fucose was added as an internal standard. All sugars were determined as anhydrous sugars based on weight of wood, that is, as glucan, galactan mannan, and so on.

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Determination of Lignin, α-Cellulose, and Sugar Contents using Transmittance Near Infrared (NIR) Spectroscopy

Lignin, α-cellulose and sugar contents were determined using Transmittance Near Infrared (NIR) Spectroscopy according to protocols established previously in our laboratory.[13–15] Extracted wood meal was ground in a Wily mill to pass a 60-mesh screen and only wood meal retained by an 80-mesh screen (80 mg) was pressed into a pallet using a standard 13 mm KBr press.

A Foss NIRSystems Inc. (Silver Spring, MD, USA) near infrared spectrometer equipped with an InTact Single Tablet Module (NR-1650) and a monochromator (NR-6500-V/H) from was used in this study. Absorbance spectra, 32 scans, were collected at 2.0 nm intervals over the range 600–1,900 nm.

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RESULTS AND DISCUSSION

Chemical Composition

Lignin contents and sugars compositions of compression wood and opposite wood were determined for several selected rings as shown in Table 1. All data are expressed as % of extractive-free wood. For opposite wood analyses, rings 8 and 9 and rings 19–21 were combined to give a sufficient amount of material for analysis. As seen in Table 1, the compression wood has much higher lignin and galactan content, and lower glucan and mannan content than the opposite wood. These results are in total agreement with those reported in the literature.[2,8–9]

Table 1. Lignin content and sugar composition of compression wood and opposite wood of tree 1

Sample name	Lignin%	Galactan%	Glucan%	Mannan%
Compression, ring 27	35.8	6.73	40.60	8.17
Compression, ring 26, latewood	37.8	6.62	38.75	7.38
Compression, ring 26	36.2	6.36	38.45	6.67
Compression, ring 21	37.5	6.95	40.17	7.91
Compression, ring 20	36.5	7.44	40.42	7.66
Compression, ring 19	36.1	6.99	38.75	7.30
Compression, ring 9	35.5	6.25	41.38	8.37
Compression, ring 8	34.1	5.55	42.64	8.95
Normal, ring 27	28.0	1.56	50.67	14.16
Opposite, rings 19–21	27.6	1.60	49.88	13.53
Opposite, ring 9	28.3	1.73	48.90	12.52

Correlation between Lignin and Sugar Contents

Because compression wood and opposite wood are distinctively different in their lignin and sugar content, these properties from selected rings in the transition zones were also determined. Individual sugar content was plot against lignin content as shown in Figure 1. Relatively good correlations exist, which suggests that compression wood content in an increment core could be estimated by determination of its lignin and sugar content. The correlations

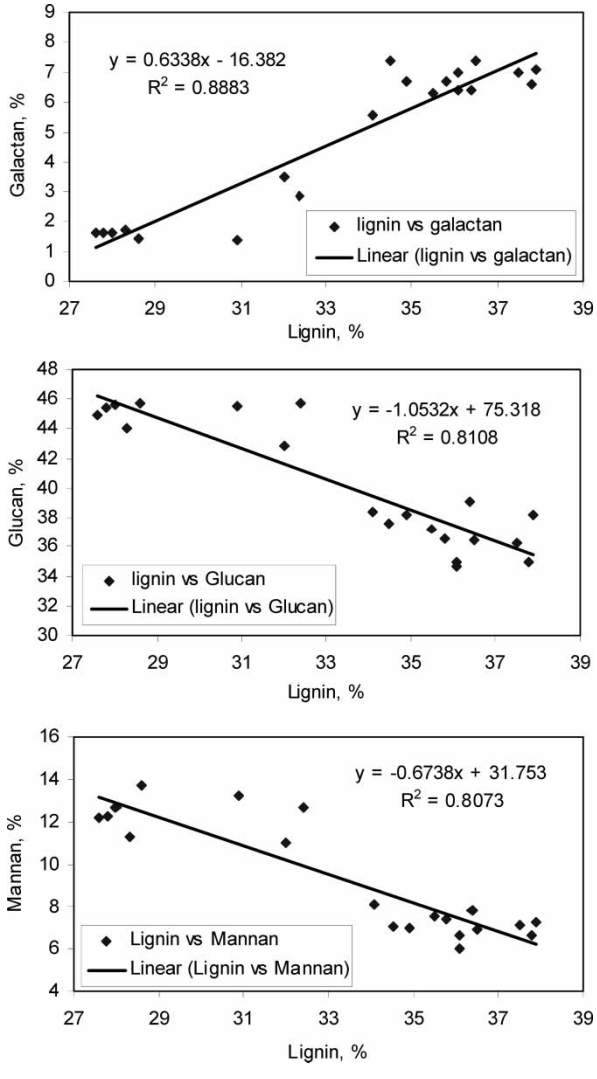

Figure 1. Correlation between sugar and lignin content in loblolly pine.

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between galactan and lignin and appear to be better than that between glucan and lignin and mannan and lignin, with R^2 values of 0.88, 0.81, and 0.81, respectively.

Correlation between α-Cellulose and Lignin Contents

Because glucan and lignin content show a good correlation for compression wood content, shown earlier, we felt that α-cellulose and lignin content

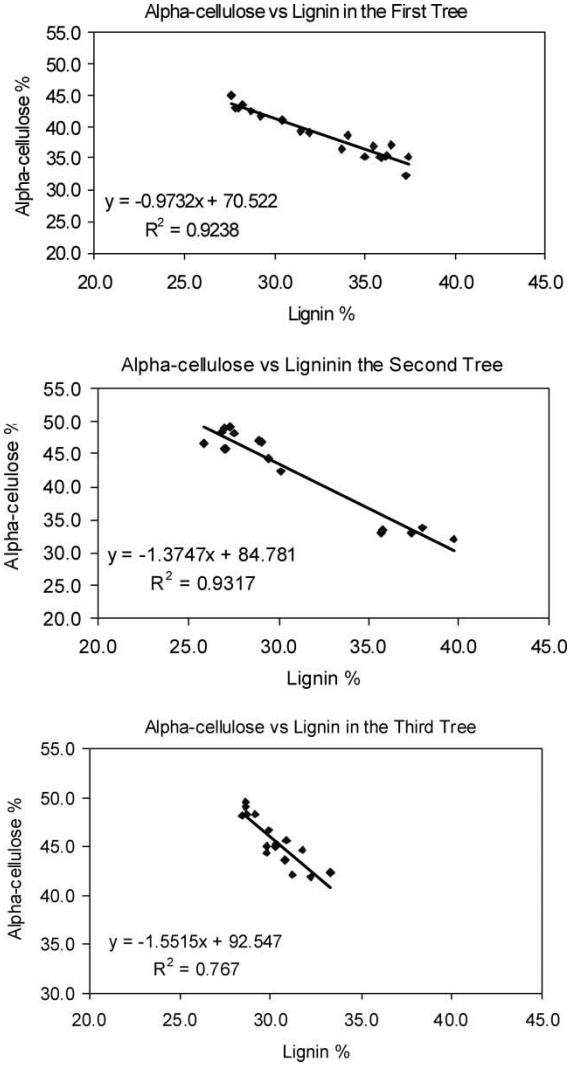

Figure 2. Correlation between α-cellulose and lignin content of trees 1–3.

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should also give a good correlation. In many laboratories, α-cellulose is easier and less expensive to determine than sugar content: therefore, we determine both lignin and α-cellulose contents on selected rings from all three wood disks.

Samples from different wedges were taken from several individual rings of each disk. Special attention was taken to ensure that samples with a wide range of compression wood contents were selected. The results are shown in Figure 2 for each individual tree. As can be seen in Figure 2, the normal wood (opposite wood) of trees 2 and 3 has higher α-cellulose content than that of tree 1. The normal wood (opposite wood) of tree 2 has lower lignin content than that of tree 1 and 3. These results point to the possibility that there may be variations in these chemical compositions among trees. However, this needs to be verified by future studies involving more trees. Also visible in Figure 2 is that the slopes of the α-cellulose versus lignin content plots are different between the three trees, suggesting that the effects of compression wood on α-cellulose and lignin content may differ from tree to tree. Again, this needs to be confirmed by later studies with a larger sample set.

Tree 3 apparently has lower content of compression wood and has a narrower range of α-cellulose and lignin content. This factor presumably accounts for the poorer correlation of tree 3 as compared with those of trees 1 and 2. The R^2 value for the linear regression line for tree 3 was 0.77 as compared to $\sim$0.92 for tree 1 and 2. Nevertheless, when data from all the three trees were plotted together, a good correlation was obtained as shown in Figure 3. The error for both α-cellulose and lignin content are $\pm$1% at a 95% confidence interval. Moreover, the wider spread in α-cellulose content ($\pm$3%) suggest that there is natural variation in α-cellulose among the three trees.

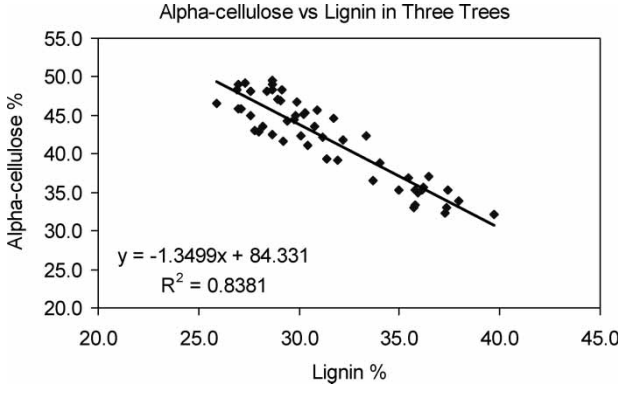

Figure 3. Correlation between α-cellulose and lignin content of all data from trees 1–3.

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NIR Prediction of Compression Wood

Applications of transmittance NIR for estimation of α-cellulose, lignin, and xylan contents as well as Syringyl/Guaiacyl ratio (S/G) in hardwood lignin have recently been developed in our laboratory.[13–15] The main advantage of transmittance NIR over traditional reflectance NIR is that the former requires a sample weight of only 80 mg, as opposed a minimum of 500 mg

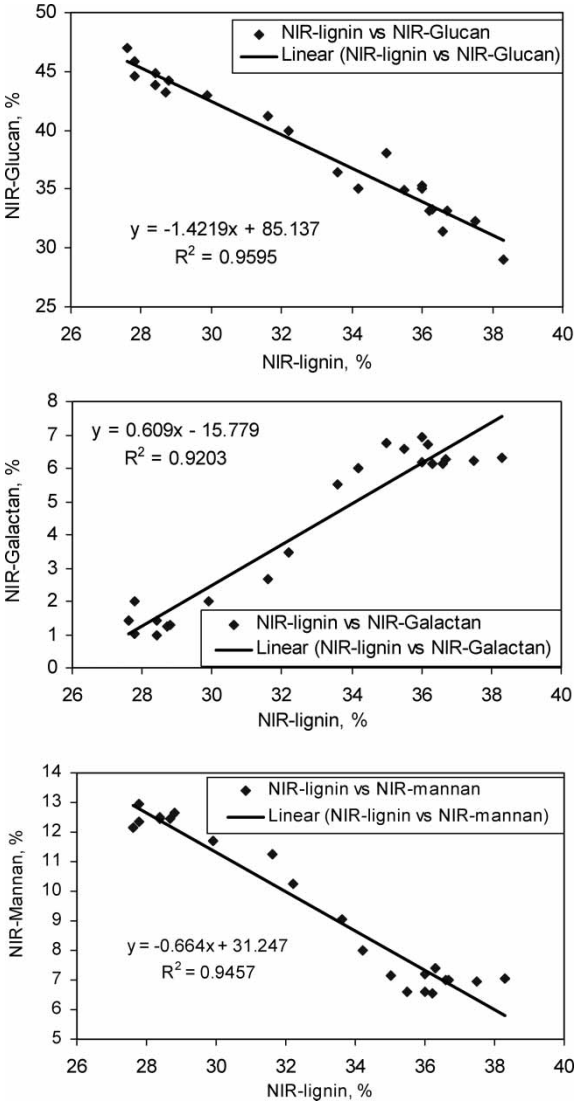

Figure 4. NIR correlations of lignin and sugar content.

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for the latter. Thus, transmittance NIR made it possible to determine the ring by ring chemical compositions of an incremental core. Therefore, we explored the possibility of using transmittance NIR to predict glucan, galactan, and mannan contents, in addition to α-cellulose and lignin. Excellent correlations were obtained between the NIR prediction and lab data for all these sugars (calibration lines not shown). Figure 4 shows the correlations of each sugar and lignin content predicted by NIR.

Although it can be also easily determined by NIR, α-cellulose is often a trait for tree breeding, and it may vary quite widely among families.[16–17] Thus, NIR determination of galactan and mannan provide rapid, independent, and micro analytical methods of accessing the impact of compression wood in tree breeding for α-cellulose. Of particular interest is the correlation of galactan and lignin contents. It has been recently demonstrated in a progeny study that lignin content of loblolly pine varies within a relatively small range with little genetic variations among families.[16] High lignin content along with high galactan content is specific to compression wood.[2,8,9] Therefore, the correlation between galactan and lignin content is probably the best parameter for determining compression wood content.

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CONCLUDING REMARKS

Compression wood content in an increment core can be accessed by plotting galactan, glucan, mannan, or α-cellulose content against lignin content. These chemical properties can be determined either by micro chemical analyses or by transmittance NIR spectroscopy.

However, more studies are needed before the tree improvement program can take advantage of the current results. The extent of natural variation in various traits of interest and the impact of compression wood on those traits need to be investigated. Additional traits of interest include microfibril angle, average fiber length, average fiber width, coarseness, and so on.

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