

THE EFFECTS OF SILVICULTURAL TREATMENTS ON THE
CHEMICAL COMPOSITION OF PLANTATION-GROWN
LOBLOLLY PINE WOOD¹

Todd F. Shupe

Graduate Research Instructor

Elvin T. Choong

Professor

Louisiana Forest Products Laboratory
School of Forestry, Wildlife, and Fisheries
Louisiana State University Agricultural Center
Baton Rouge, LA 70803-6202

and

Chun H. Yang

Former Graduate Research Assistant
School of Forestry, Wildlife, and Fisheries
Louisiana State University Agricultural Center
Baton Rouge, LA 70803-6202

(Received November 1995)

ABSTRACT

The influence of silvicultural treatments (fertilization, stand density, and pruning) on the chemical composition (hot-water extractives, alcohol-benzene extractives, ether extractives, Klason lignin, holocellulose, and alpha-cellulose) of outerwood and innerwood of plantation-grown 12-year-old loblolly pine (*Pinus taeda* L.) was investigated. Plots located near Bogalusa, in southeastern Louisiana, were maintained at four levels of stand density (2,470; 1,976; 1,482; and 988 trees per hectare) and exhibited varied effects on wood chemical properties. The highest mean extractive contents occurred in the plots with 2,470 residual trees per hectare. Stand densities did not appear to be consistently related to Klason lignin, holocellulose, and alpha-cellulose contents. Fertilization caused a significant reduction in alcohol-benzene extractive content, ether extractive content, and Klason lignin. There was no significant effect in any chemical property attributable to the pruning treatment, except in alcohol-benzene extractives, which decreased significantly in the pruned trees. Innerwood yielded significantly greater extractive contents for the alcohol-benzene and hot-water methods of extractive content determination, and outerwood yielded significantly higher values for Klason lignin, holocellulose, and alpha-cellulose.

Keywords: Alpha-cellulose, extractives, fertilization, holocellulose, loblolly pine, pruning, Klason lignin, thinning.

INTRODUCTION

There has been much research on the future uses of forest biomass from short-rotation in-

tensive culture (SRIC) plantations. The thrust of this effort has focused on the potential profitability of hardwood woody biomass as a raw material for energy purposes (Fege et al. 1979; Howlett and Gamachie 1977; Inman et al. 1977) and the characterization and identification of favorable species (Chow et al. 1987; Bendtsen 1978; Blankenhorn et al. 1985; Shupe 1993; Shupe et al. 1993).

¹ This paper (No. 95-22-9253) is published with the approval of the Director of the Louisiana Agricultural Experiment Station. The research was supported in part by the McIntire-Stennis Cooperative Forest Research Program.

SRIC research has traditionally been justified by linking the increased future demand of wood and wood products and an ever-increasing global population. As the global population continues to increase, total wood demand is also predicted to increase (Sutton 1994). In addition to solid wood products and wood composite panels, there will certainly be an increased demand for both traditional and novel products that can be derived from wood's organic constituents.

Extractives are directly related to permeability, specific gravity, hardness, and wood compressive strength (Panshin and deZeeuw 1980) and can also be used to predict extracted specific gravity values from specific gravity values determined on unextracted material (Taras and Saucier 1967). The alpha-cellulose and holocellulose content of wood is critical in determining the acceptability of a particular species for pulp and paper. Alpha-cellulose content is also critical in terms of rayon production for the textile industry. The lignin component of wood can be used to derive end-products such as benzene and vanillin. Lignin also can potentially be used as an additive or supplement in wood composite adhesive technology (Cyr and Ritchie 1989).

While other researchers have studied the effects of an individual treatment, i.e., fertilizer, on alpha-cellulose and holocellulose content (Zobel et al. 1961), research is lacking on the effects of multiple silvicultural treatments on short-rotation loblolly pine wood chemical properties. The objectives of this study were to determine the effect of fertilization, thinning, and pruning on the alcohol-benzene, hot-water, and ether extractive contents along with Klason lignin, holocellulose, and alpha-cellulose contents of 12-year-old loblolly pine innerwood and outerwood. All references to plot ages are actual tree ages, which include the year before lifting and outplanting of seedlings.

PROCEDURES

Plantation sampling

The 12-year-old loblolly pine plots used in this study are located on a previous agricul-

tural pasture at the Louisiana State University Lee Memorial Forest in southeastern Louisiana's Washington Parish. The site is located at approximately 30°52'N latitude and 89°59'W longitude and is a good site for growth of loblolly pine because of the milder winters, more fertile soil, and longer growing seasons. The soil on this particular plot is a Ruston fine sandy loam (fine-loamy, siliceous, thermic Typic Paleudults) (USDA Soil Conservation Service 1991). The soil is well drained with a pH of 4.5-4.9 (Burns et al. 1985). The site index is 100 feet at age 50 (Burns 1982). Genetically unaltered (1-0) seedlings were grown using seed collected from natural loblolly stands located in Louisiana and were planted on each one-hectare (ha) plot at 1.82 m × 1.82 m spacings.

The study consisted of four main plots, each comprising 1 ha. The four main plots were randomly assigned one of four levels of stand density at the time of establishment (2,470; 1,976; 1,482; and 988 trees per ha). At a stand age of 9 years, silvicultural treatments were assigned to 0.50-ha subplots on all main plots. The fertilizer treatments (222 kg per ha of granular urea (45% nitrogen), superphosphate (53.4% available P₂O₅), and muriate of potash (60% available K₂O) were randomly assigned to either the east or west half of each subplot, and the pruning treatment was randomly selected to be applied to either the north or south half of each subplot (Fig. 1). Pruning treatments were performed simultaneously with the fertilization treatments and simply consisted of manually removing all live branches along the bole up to 10 m below the live crown. The cultural treatments in the ninth year were all performed within a 30-day period.

From each experimental unit (0.25 ha subplot) (Fig. 1), 10 trees were randomly selected, and an over-sized increment borer 10 mm in diameter was used to take a sample at breast height. Increment cores were immediately wrapped in plastic to prevent moisture loss. At the laboratory, the increment cores were air-dried before being debarked and divided into innerwood (growth rings 1-6) and outerwood (growth rings 7-12).



FIG. 1. Split-plot arrangement of fertilization and pruning treatments for each main plot. There was one main plot for each level of stand density (i.e., 2,470; 1,976; 1,482; and 988 trees per ha).

Laboratory experimentation

Chemical constituent values were obtained using the following test procedures: 1) alcohol-benzene extractive content (ASTM D 1105-84), 2) hot-water extractive content (ASTM 1110-84), 3) ether extractive content (ASTM 1108-84), Klason lignin content (D 1106-84), holocellulose content (ASTM D 1104-56), alpha-cellulose content (ASTM D 1103-60) (ASTM 1982, 1993).

The statistical analysis was conducted using SAS programming methods (SAS 1989) in conjunction with analysis of variance (AOV) techniques (Steel and Torrie 1980; Box et al. 1978). The significance of each factor and factor interactions were determined at the $\alpha = 0.05$ level using Type III Sum of Squares. In order to test the treatment effects on the chemical composition of wood, a split-plot arrangement was established to provide four thinning treatments (i.e., 2,470; 1,976; 1,482; and 988 trees per ha) as whole plot factors with levels of fertilizer (fertilized and unfertilized) and pruning (pruned and unpruned) as subplot factors.

RESULTS AND DISCUSSION

Extractives (extraneous material)

Chemical property values of 12-year-old loblolly pine wood are summarized in Tables

TABLE 1. Summarized means of individual treatments on the extractive contents of 12-year-old loblolly pine wood.

Treatments	Extractive content (%)			
	Alcohol-benzene	Ether	Hot-water	Total
Unfertilized	3.09 A ¹	0.50 A	3.25 A ¹	6.84 A
Fertilized	2.66 B	0.32 B	3.11 A	6.09 B
Unpruned	3.25 A	0.38 A	3.24 A	6.87 A
Pruned	2.50 B	0.45 A	3.12 A	6.07 B
Thinning ²				
2,470	3.40 A	0.47 A	3.39 A	7.26 A
1,976	2.42 C	0.40 AB	2.98 B	5.80 BC
1,482	3.04 BC	0.35 AB	3.27 A	6.66 B
988	2.66 C	0.18 C	3.09 B	5.93 BC
Outerwood	2.90 B	0.41 A	3.18 B	6.49 B
Innerwood	6.83 A	0.41 A	4.10 A	11.34 A

¹ Means with the same letter are not statistically different at $\alpha = 0.05$. Duncan's multiple range multiple comparisons for each method of extractive content determination were made within each column for each cultural treatment.

² Number of trees per ha.

1–2. The extractive contents for each method of determination are summarized for each cultural treatment in Table 1. Duncan's mean separation letters associated with levels of significance are listed in all tables.

Fertilization resulted in a significantly lower alcohol-benzene and ether extractive content but did not significantly affect hot-water extractives (Table 1). Our values are near those reported by Max (1945) for alcohol-benzene (2.76%) and hot-water (1.24%) on green loblolly pine wood. However, Max (1945) found a much greater value for ether extractives of 1.83%. It is interesting to note that our value for innerwood (6.83%) is much greater than that reported by Max (1945) even though our wood was seasoned (air-dried) prior to extraction, which typically reduces the amount of extractives removed by alcohol-benzene or ether. Pettersen (1984) reported the following mean extractive contents for loblolly pine: 1% NaOH (11%), hot-water (2%), and alcohol-benzene (3%).

The results for the pruning treatment were mixed. Pruning resulted in insignificantly less hot-water extractives and significantly less alcohol-benzene extractives. However, pruning led to insignificantly more ether extractives (Table 1).

A slight association between trees per ha and extractive content is evident in the stand density treatments. For ether extractives, there is a continuous decrease in extractives with decreasing trees per ha (Table 1). However, the range of mean values is small (0.18–0.47). The hot-water and alcohol-benzene extractives both showed the following rank for thinning effect (2,470 > 1,482 > 988 > 1,976) (Table 1). These results are interesting since Kramer and Kozłowski (1979) found extractives to be products of metabolic growth. Hence, treatments that increase tree growth and vigor should increase extractive content. However, this hypothesis was not proven in this study. In general, plots of 2,470 trees per ha yielded wood with the highest extractive content (Table 1).

As expected, the hot-water and alcohol-benzene extractive contents were significantly greater in innerwood than outerwood. The difference in southern pine heartwood and sapwood extractive content has been well documented (Ritter and Fleck 1926; Wahlenberg 1960; Posey and Robinson 1969; McMillin 1968). Surprisingly, the values were the same (0.41) for ether extractives.

Non-extraneous material

Fertilization served to significantly lower the mean Klason lignin content from 75.52% for unfertilized to 73.30% for the fertilized plots. There was not a significant difference between fertilized and unfertilized plots for holocellulose and alpha-cellulose (Table 2). Zobel et al. (1961) also failed to find a significant difference between heavily and moderately fertilized 25-year-old loblolly pine plantations and a control plantation with regards to both water-resistant carbohydrates, "holocellulose," and alpha-cellulose. Pettersen (1984) reported the following mean carbohydrate contents: holocellulose (68%), alpha cellulose (45%), and Klason lignin (27%).

There was not a significant difference between pruned and unpruned plots for Klason lignin, holocellulose, or alpha-cellulose content. The thinning treatment showed mixed results with regards to Klason lignin, holocel-

TABLE 2. Summarized means of individual treatments on Klason lignin, holocellulose, and alpha-cellulose contents of 12-year-old loblolly pine wood.

Treatments	Klason lignin (%)	Holocellulose (%)	Alpha-cellulose (%)
Unfertilized	75.52 A ¹	27.48 A	45.69 A
Fertilized	73.30 B	26.70 A	46.17 A
Unpruned	73.77 A	27.23 A	45.77 A
Pruned	72.56 A	27.44 A	45.60 A
Thinning ²			
2,470	73.40 A	26.60 B	46.46 A
1,976	72.56 A	27.44 A	44.98 AB
1,482	73.32 A	27.63 A	45.38 B
988	73.32 A	26.68 B	46.90 A
Outerwood	72.91 A	28.82 A	45.93 A
Innerwood	71.18 B	27.09 B	41.60 B

¹ Means with the same letter are not statistically different at $\alpha = 0.05$. Duncan's multiple range multiple comparisons were made for a specific treatment for each chemical property.

² Number of trees per ha.

lulose, and alpha-cellulose (Table 2). There appears to be no association between stand density and Klason lignin, holocellulose, and alpha-cellulose values. The range of values for all three chemical properties was very small, and no single thinning treatment was significantly superior for improving any chemical property.

As expected, outerwood values were significantly higher than innerwood for Klason lignin, holocellulose, and alpha-cellulose. There is a relationship between wood density and the structural cell-wall material (i.e., holocellulose and alpha-cellulose) (Panshin and deZeeuw 1980). Also, in addition to forming a majority of the middle lamella, lignin is intimately associated with cellulose and adds rigidity to the cell (Haygreen and Bowyer 1989).

Koch (1972) attributed the changes in polysaccharide content across tree diameter to the presence of juvenile wood. Many researchers have also found that loblolly pine outerwood possesses more holocellulose and alpha-cellulose than innerwood (Byrd et al. 1965; McMillin 1968; Stamm and Sanders 1966; Zobel and McElwee 1958; Zobel et al. 1966).

CONCLUSIONS

(1) Fertilization was found to result in a significant decrease in alcohol-benzene and ether

extractive contents and Klason lignin contents. However, fertilization did not affect hot-water extractive, holocellulose, or alpha-cellulose contents.

(2) Pruning had an insignificant effect on all chemical properties except alcohol-benzene extractive content, which decreased significantly in the pruned trees.

(3) Plots with the most trees per ha (2,470) gave the highest mean extractive content for all three methods of determination, but this level was uniquely and significantly greater for only the alcohol-benzene test. Stand density did not appear to be consistently related to lower or higher Klason lignin, holocellulose, and alpha-cellulose mean values.

(4) In regards to outerwood/innerwood differences, this study has shown an increased amount of alcohol-benzene and hot-water extractive contents in innerwood and a greater amount of Klason lignin and polysaccharides in the outerwood region.

REFERENCES

- AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM). 1982. Annual book of ASTM standards. Part 22, Wood; adhesives. Philadelphia, PA. 1204 pp.
- . 1993. Annual book of ASTM standards. Section 4, Vol. 04.09—Wood. Philadelphia, PA. 624 pp.
- BENDTSEN, B. A. 1978. Properties of wood from improved and intensively managed trees. *Forest Prod. J.* 28(10):61-72.
- BLANKENHORN, P. R., T. W. BOWERSOX, K. M. KUKLEWSKI, AND G. L. STIMLEY. 1985. Effects of rotation, site, and clone on the chemical composition of *Populus* hybrids. *Wood Fiber Sci.* 17(3):351-360.
- BOX, G.E.P., W. G. HUNTER, AND J. S. HUNTER. 1978. Statistics for experimenters. John Wiley & Sons, Inc., New York, NY. 653 pp.
- BURNS, P. Y. 1982. Unpublished data. Louisiana State University, School of Forestry, Wildlife, and Fisheries. Baton Rouge, LA.
- , S. C. HU, AND D. P. REED. 1985. Intensive culture of loblolly pine in southeastern Louisiana: Results through age 21. Unpublished manuscript.
- BYRD, V. L., E. L. ELWOOD, R. G. HITCHINGS, AND A. C. BAREFOOT. 1965. Wood characteristics and kraft properties of four selected loblolly pines. II. Wood chemical constituents and their relationship to fiber morphology. *Forest Prod. J.* 15:313-320.
- CHOW, P., G. L. ROLFE, W. K. MOTTER, AND K. A. MAJERUS. 1987. Site, spacing, tree portion, and species influence ash and extractives content of five juvenile hardwoods. VI:24-26 Proc. Central Hardwood Forest Conference, Feb. 24-26, 1987, Knoxville, TN.
- CYR, N., AND R.G.S. RICHIE. 1989. Estimating the adhesive quality of lignins for internal bond strength. Pages 5-11 W. G. Glasser and S. Sarkanen, eds., Lignin properties and materials. 195th National Meeting of the American Chemical Society, June 5-11, 1988 Toronto, Ontario, Canada.
- FEGE, A. S., R. E. INMAN, AND D. J. SALO. 1979. Energy farms for the future. *J. Forestry* 77:358-360.
- HAYGREEN, J. G., AND J. L. BOWYER. 1989. Forest products and wood science. 2nd ed. Iowa State Univ. Press, Ames, IA. 500 pp.
- HOWLETT, K., AND A. GAMACHE. 1977. Silvicultural biomass farms, Vol. VI: Forest and mill residues as potential sources of biomass. National Technical Information Service Rep. MITRE-TR-7347-V6/LL, Springfield, VA. 124 pp.
- INMAN, R. E., D. J. SALO, AND B. MCGURK. 1977. Site-specific production studies and cost analyses. National Technical Information Service Rep. MITRE-TR-7347-V4. McLean, VA. 123 pp.
- KOCH, P. 1972. Utilization of the southern pines. Vol. 1. The raw material. USDA Agric. Handb. No. 420. 734 pp.
- KRAMER, P. J., AND T. T. KOZLOWSKI. 1979. Physiology of woody plants. Academic Press, Inc. Orlando, FL. 811 pp.
- MAX, K. W. 1945. Chemical analysis of green loblolly pine. *South. Pulp Pap. J.* 7(8):36.
- McMILLIN, C. W. 1968. Chemical composition of loblolly pine wood as related to specific gravity, growth rate, and distance from pith. *Wood Sci. Technol.* 2(3): 233-240.
- PANSHIN, A. J., AND C. DEZEEUW. 1980. Textbook of wood technology. 4th ed. McGraw-Hill Book Co., New York, NY. 705 pp.
- PETERSEN, R. C. 1984. The chemical composition of wood. Pages 57-126 in R. M. ROWELL, ED. Chemistry of solid wood. Based on a symposium sponsored by the Division of Cellulose, Paper, and Textile at the 185th Meeting of the American Chemical Society, Seattle, WA, March 20-25, 1983. American Chemical Society, Washington, DC. 614 pp.
- POSEY, C. E., AND D. W. ROBINSON. 1969. Extractives of shortleaf pine—an analysis of contributing factors and relationships. *Tappi* 52:110-115.
- RITTER, G. J., AND L. C. FLECK. 1926. Chemistry of wood. IX. Springwood and summerwood. *Ind. Eng. Chem.* 18:608-609.
- SAS INSTITUTE, INC. 1989. SAS/STAT User's Guide, Version 6, 4th ed, Vol. 2. Cary, NC. 846 pp.
- SHUPE, T. F. 1993. An assessment of some of the chemical properties of ten-year-old short-rotation hardwood biomass grown in Illinois. M.S. thesis, Univ. of Illinois at Urbana-Champaign, IL. 202 pp.
- , P. CHOW, G. L. ROLFE, M. RAHEEL, AND P. H. GEIL. 1993. Some chemical properties of hardwood