# CHEMICAL VARIATION IN LODGEPOLE PINE WITH SAPWOOD/HEARTWOOD, STEM HEIGHT, AND VARIETY

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### ABSTRACT

The chemical variation in lodgepole pine over its geographical range with sapwood/heartwood, stem height, and variety was investigated. In both varieties, *latifolia* and *murrayana*, the heartwood was significantly higher in extractives than the sapwood, whereas the sapwood was significantly higher in holocellulose and alpha-cellulose than the heartwood. The sapwood and heartwood did not differ in pH and lignin content.

Ash, lignin, and holocellulose content were positively correlated with stem height in *latifolia* and *murrayana*. Extractive and alpha-cellulose content were negatively correlated with stem height. The stem height variations appeared to be related to the heartwood/sapwood ratio, proportion of mature and juvenile wood, and the specific gravity.

The variation between *latifolia* and *murrayana* in ash, extractive, and lignin content and pH was small. In contrast, the holocellulose and alpha-cellulose content was 2% higher in *murrayana* than in *latifolia*. The polysaccharide variation may be influenced by climate and site conditions as well as genetic differences.

Keywords: Pinus contorta, latifolia, murrayana, chemical variation, heartwood, sapwood, stem height, ash, pH, extractives, lignin, holocellulose, alpha-cellulose.

### INTRODUCTION

Lodgepole pine is an abundant but underutilized timber resource in the United States and Canada. The distribution range of *Pinus contorta* Dougl. ex Loud. extends from Colorado to California and then north to Alaska. *Latifolia*, the major lodgepole pine variety, grows along the Rocky Mountains from 40–60° north latitude; while *murrayana*, a less important variety, grows in California and Oregon extending from 35–45° north latitude.

Lodgepole pine is used for lumber, poles, railroad ties, construction timbers, and structural flakeboard. Potential future uses include cementboard (Moslemi et al. 1983), underlayment grade particleboard, furniture grade particleboard, hard-

Wood and Fiber Science, 22(1), 1990, pp. 22–30 © 1990 by the Society of Wood Science and Technology board, and medium density fiberboard (Maloney 1982). Lodgepole pine is also a highly acceptable species for pulp with good yield, bursting strength, folding endurance, and tensile strength (McGovern 1951). Mechanical pulps made from lodgepole pine have excellent color and satisfactory strength (Guernsey and Dobbie 1966).

In the future, pulp mills and wood conversion processes will face a wood supply shortage (McMichael 1982), which will necessitate the use of whole tree chips as a raw material for pulping, composite products, and as a fuel. Because lodgepole pine is abundant and typically small in diameter, it will likely be used as whole tree chips. In order to utilize this resource more fully, basic knowledge is needed on the physical and chemical properties that could affect utilization.

This project was designed to measure the chemical variation in lodgepole pine with sapwood/heartwood, stem height, and between varieties, *latifolia* and *murrayana*. These results complement a similar study on the chemical variation in lodgepole pine with latitude, elevation, and diameter class (Kim 1988; Kim et al. 1989).

This research project was unique because it measured the chemical variation in a tree species, including two varieties, over an extremely broad latitudinal and elevational range in the Northwestern United States and Canada. In addition, the large number of trees sampled and analyzed strengthened the statistical significance of the results.

#### MATERIALS AND METHODS

The *latifolia* and *murrayana* samples were collected during the summer months of 1983 and 1984 by the Forestry Sciences Laboratory at the Intermountain Station in Missoula, Montana (Koch 1987). Twenty-seven *latifolia* trees were collected at nine different latitudes at 2.5° intervals (40–60° north latitude) to make a total of 243 trees (3 diameters  $\times$  3 elevations  $\times$  3 replications  $\times$  9 latitudes). Nine *murrayana* trees were taken from mid-elevation at four latitudes (37.5–45.0°) to make a total of 36 *murrayana* trees (3 diameters  $\times$  1 elevation  $\times$  3 replications  $\times$  4 latitudes). The trees were cut into ten 2.5-inch-thick disks representing tenth points up each of the 279 trees, and they were then sent to the University of Idaho for chemical analysis. For sapwood/heartwood analyses, 279 disks representing 20% stem height from each tree were sent separately.

The disks were split into either 90 or 180° wedges with a chisel. The large, visible portions of bark, knots, decay, and other abnormalities were removed. The wedges for the sapwood/heartwood analyses were stained with 10% ferric chloride to determine the transition point from sapwood to heartwood (Kutscha and Sachs 1962). The wood was then split with a chisel to separate the sapwood from heartwood, and then the stained edges were removed with a band saw.

The sapwood/heartwood and stem height samples were chiseled into matchstick-size pieces and Wiley-milled to yield samples for analysis of 40–60 mesh (TAPPI M-59). Weighted samples from low, medium, and high elevations were pooled, blended in a rotary mixer for 5 minutes, and then stored in polyethylene bags to form average elevation samples. Sapwood and heartwood analyses for *latifolia* [2 diameters  $\times$  9 latitudes (or 4 latitudes for *murrayana*)  $\times$  3 tree replications] were determined only in the 152- and 228-mm diameter trees, as the 76-mm trees did not yield enough material for analysis. The 152- and 228-mm *latifolia* trees averaged 91 and 107 years of age, respectively, while the *murrayana* trees averaged 84 and 91 years of age as found by Koch (1987).

For the analyses at different stem heights, each of the latitude samples were combined to give an average latitude and elevation sample. Analyses at different stem heights (2 diameters  $\times$  9 stem heights  $\times$  3 tree replications) were determined only in the 152- and 228-mm diameter trees. The chemical variation between *latifolia* and *murrayana* was compared at medium elevation and at the 40.0, 42.5, and 45.0° latitudes which they had in common.

The moisture (ASTM D 2016-74), ash (ASTM D 1102-56), ethanol-toluene extractives (ASTM D 1107-56), lignin (ASTM D 1106-56), and alpha-cellulose (ASTM D 1103-60) were measured following standard procedures. Toluene was substituted for benzene in the extractive analyses (Goetzler 1982). The pH and holocellulose content were measured using the Forest Products Laboratory Methods 67-033 and 67-018, respectively. The ultimate analyses were performed by the Analytical Services Laboratory in the College of Agriculture at the University of Idaho. The proximate analyses were performed by Huffman Laboratories, Inc. in Golden, Colorado. The average chemical composition was calculated on an oven-dry wood basis. The data were statistically analyzed using SAS GLM, SAS CORR, and SAS REG from which analyses of variance and correlation coefficients were computed. Statistically significant correlations were at the 95% confidence level.

#### **RESULTS AND DISCUSSION**

### Chemical variation with sapwood/heartwood at 20% stem height

With latitude and diameter data pooled, the average ash content at 20% stem height varied significantly between heartwood and sapwood in *latifolia* but not in *murrayana* (Table 1). In a related study with loblolly pine, McMillin (1970) found that the mineral content was highest in the pith, lower in the heartwood, and then lowest in the sapwood.

The average pH of *latifolia* heartwood was slightly higher than sapwood at most latitudes, especially in the 228-mm trees. There was no apparent pH variation in *murranaya*. Previous studies with other species by Sandermann (1959), Gray (1958), and McNamara (1970) found no distinctive variation in pH between sapwood and heartwood.

The extractive content of heartwood was significantly higher than sapwood in *latifolia* (3.30% vs. 2.03%) and *murrayana* (2.81% vs. 1.88%) as shown in Table 1.

Analyses	latifolia		murrayana	
	Sapwood	Heartwood	Sapwood	Heartwood
pH	4.52	4.55	4.58	4.59
Ash (%)	0.25	0.30	0.30	0.31
Extractives (%)	2.03	3.30	1.88	2.81
Lignin (%)	26.02	25.77	25.71	26.13
Holocellulose (%)	80.02	76.99	79.51	76.09
Alpha-cellulose (%)	49.38	46.02	49.52	44.17

**TABLE 1.** Variation in chemical composition of sapwood and heartwood in latifolia and murrayana with latitude and diameter data pooled.

Each value is an average of either 54 analyses for latifolia or 24 analyses for murrayana.

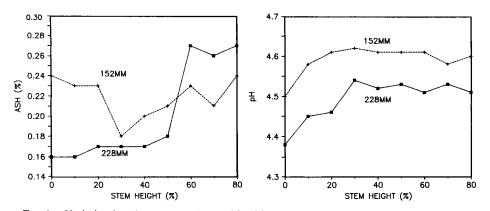


FIG. 1. Variation in ash content and pH of latifolia as a function of stem height and diameter.

McMillin (1970) and Peter et al. (1979) also showed that the extractive content of loblolly pine and lodgepole pine heartwood was higher than sapwood.

The lignin content of sapwood and heartwood at 20% stem height was not significantly different in *latifolia* and *murrayana*. Previous studies have come to contradictory conclusions concerning the variation in lignin content in sapwood and heartwood of different tree species. Ritter and Fleack (1926) reported that loblolly pine sapwood contained more lignin than heartwood, while Peter et al. (1979) found that lodgepole pine sapwood had less lignin than heartwood. Uprichard (1965) found that there was in general no marked lignin variation within radiata pine trees, although growth rings adjacent to the pith had the highest lignin content. Therefore, based on the literature and our results, the variation in lignin content between heartwood and sapwood does not appear to be significant.

The holocellulose content of sapwood was significantly higher than that of heartwood in both *latifolia* and *murrayana* as shown in Table 1. These results agreed with those of Zobel and McElwee (1958) and Byrd and Elwood (1965), who reported that loblolly pine outer wood (sapwood) contained 1–3% more holocellulose than corewood (heartwood).

The alpha-cellulose content of sapwood was also significantly higher than that of heartwood in *latifolia* (49.38% vs. 46.02%) and *murrayana* (49.52% vs. 44.17%). Previous researchers found that loblolly pine (Zobel and McElwee 1958; Byrd

	Stem height		
Analyses	latifolia	murrayana	
pН	+	0	
Ash	+	+	
Extractives	_	_	
Lignin	+	-+	
Holocellulose	+	+	
Alpha-cellulose	_	_	
Specific gravity	_		

**TABLE 2.** Statistical correlations in latifolia and murrayana as a function of stem height. Correlations were either positive (+), negative (-), or not significant (0) at the 95% confidence level.

The specific gravity relationships were determined by Koch (1987).

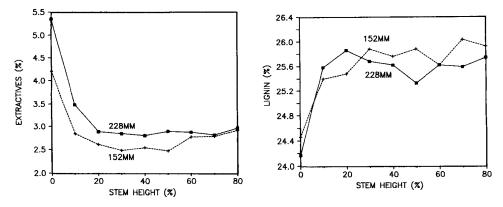


FIG. 2. Variation in extractive and lignin content of *latifolia* as a function of stem height and diameter.

and Elwood 1965) and radiata pine (Uprichard 1976) outer wood contained 4– 7% more alpha-cellulose than corewood. The variation in alpha-cellulose content between sapwood and heartwood is probably related to the proportion of mature and juvenile wood and to the extractive concentration.

### Chemical variation with stem height

With diameter data pooled, the ash content in *murrayana* was positively correlated with stem height (r = 0.42), ranging from 0.17% at 0% stem height to 0.27% at 80% stem height (Fig. 1 and Table 2). The ash content in *latifolia* was also positively correlated with stem height (r = 0.30), ranging from 0.17% to 0.26%. These results agreed with previous research by Clermont and Schwarz (1951), which showed that the ash content of black spruce increased with stem height. The increase in ash content is probably related to the higher proportion of juvenile wood and the more frequent knot and branch bases at the top of the tree stem.

The pH was positively correlated with stem height in *latifolia* (r = 0.37) but not in *murrayana*. The pH range in both varieties was small; *latifolia* ranged from 4.44 to 4.58 while *murrayana* ranged from 4.48 to 4.61. As shown in Fig. 1, the pH increased from 0% to 30% stem height and then remained fairly constant. Sandermann (1959) found substantial variation in pH from the base of a Douglas fir tree (4.22) to the top (5.83). The variation in pH with stem height and diameter class is probably related to the proportion of heartwood and ash in the stem.

The extractive content was negatively correlated with stem height in both *la-tifolia* (r = -0.51) and *murrayana* (r = -0.39). As shown in Fig. 2, the average extractive content decreased from 4.60% at 0% to 2.66% at 30% stem height in *latifolia* and from 3.06% at 0% to 2.09% at 60% stem height in *murrayana*. The extractive content declined sharply from 0% to 20% stem height and then remained fairly constant to the top of the tree. The variation in extractive content with stem height and diameter class is closely related to the proportion of mature heartwood in the stem.

The lignin content of both *latifolia* (r = 0.53) and *murrayana* (r = 0.43) was positively correlated with stem height (Table 2). In *latifolia* the average lignin

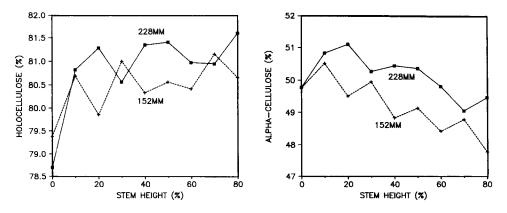


FIG. 3. Variation in holocellulose and alpha-cellulose content of *latifolia* as a function of stem height and diameter.

content ranged from 24.32% at 0% to 25.83% at 80% stem height. The lignin content increased by approximately 1.5% from 0% to 20% stem height and then appeared to remain constant at higher stem heights (Fig. 2). The increase in lignin with stem height is probably related to the higher proportion of juvenile wood and thus lignin in the upper stem.

The holocellulose content of both *latifolia* and *murrayana* was also positively correlated (r = 0.48) with stem height (Table 2). *Latifolia* ranged from 79.04% at 0% to 81.13% at 80% stem height, as shown in Fig. 3. The holocellulose increased to approximately20–30% stem height and was then reasonably constant. Zobel and McElwee (1958) and Byrd and Elwood (1965) found that the holocellulose content in loblolly pine increased 1–3% with stem height. This increase is probably related to the higher proportion of hemicellulose in the juvenile wood in the top of the tree.

In contrast, the alpha-cellulose content of both *latifolia* (r = -0.51) and *murrayana* (r = -0.34) was negatively correlated with stem height. The average alphacellulose content gradually decreased from 50.67% at 10% to 48.61% at 80% stem height in *latifolia*, as shown in Fig. 3. This negative correlation between alphacellulose and stem height has been shown in earlier studies on black spruce (Clermont and Schwarz 1951) and loblolly pine (Zobel and Stonecypher 1966). The higher percentage of alpha-cellulose in the tree base is likely due to the higher proportion of slow-grown mature wood. Since specific gravity decreased as stem

	murrayana	latifolia
pH	4.57	4.63
Ash (%)	0.29	0.28
Extractives (%)	2.78	2.37
Lignin (%)	25.91	26.21
Holocellulose (%)	83.10	80.76
Alpha-cellulose (%)	51.95	48.82

**TABLE 3.** Chemical composition of latifolia and murrayana with latitude (40–45°) and diameter data pooled.

	murrayana	latifolia
Ultimate analysis		
Carbon (%)	50.06	50.21
Hydrogen (%)	6.17	6.28
Sulfur (%)	0.02	0.02
Oxygen (%)	43.64	43.38
Nitrogen (%)	0.01	< 0.01
Ash (%)	0.1	0.1
Heating value (Btu/lb)	8,577	8,705
Proximate analysis		
Volatile matter (%)	79.94	82.58
Fixed carbon (%)	19.73	17.00
Ash (%)	0.33	0.42

TABLE 4. Proximate and ultimate analyses for latifolia (40-60°) and murrayana (37.5-45.0°).

height increased in *latifolia* and *murrayana* (Koch 1987), the alpha-cellulose should also decrease with stem height.

## Chemical variation with variety

The chemical variation between *latifolia* and *murrayana* in pH, ash, extractive, and lignin content with latitude and diameter data pooled was very small as shown in Table 3. In contrast, the holocellulose and alpha-cellulose content of *murrayana* was approximately 2% higher than *latifolia*.

The pH, ash, extractive, and lignin content varied more with latitude and diameter than with variety. In contrast, holocellulose and alpha-cellulose content varied more with variety than with latitude or diameter. The variation in carbohydrate content may be due to genetic differences between the varieties, or it may be due to climactic or site conditions. For example, *murrayana* grows closer to coastal regions with a higher rainfall than *latifolia*, which grows in more mountainous arid regions.

Ultimate and proximate data for *latifolia* and *murrayana* are also very similar as shown in Table 4.

#### Chemical variation as related to utilization

The chemical variation with sapwood/heartwood, stem height, and variety in lodgepole pine is relatively small and follows trends found in other studies. Heartwood contains approximately 1% more extractives than sapwood, while sapwood contains approximately 3% more holocellulose and alpha-cellulose than heartwood. The larger diameter trees on the average contain more extractives and more holocellulose and alpha-cellulose than smaller diameter trees. Sawmill residue chips from larger trees will give higher pulp yields than whole tree chips from logging residue, since sawmill residues consist largely of sapwood.

The quality of lodgepole logs and fiber diminishes with tree height because of the higher proportion of juvenile wood and the lower specific gravity in the upper tree. The juvenile wood is weaker, more dimensionally unstable, and more difficult to pulp than mature wood.

#### CONCLUSIONS

Heartwood contained significantly more extractives than sapwood, whereas the sapwood contained significantly more holocellulose and alpha-cellulose than heartwood. The pH and lignin content did not differ significantly between sapwood and heartwood.

Ash, lignin, holocellulose, and pH were positively correlated with stem height, whereas the extractive and alpha-cellulose content were negatively correlated with stem height. The decrease in alpha-cellulose with stem height is correlated with an increase in hemicellulose, an increase in juvenile wood, and a decrease in specific gravity.

The variation in ash, extractive, and lignin content and pH between *latifolia* and *murrayana* was not significant. In contrast, holocellulose and alpha-cellulose were approximately 2% higher in *murrayana* than *latifolia*. This variation in polysaccharide content between the two varieties may be related to climate and site conditions as well as genetic differences.

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#### REFERENCES

- AMERICAN SOCIETY OF TESTING MATERIALS. ASTM Standards D 2016-74, D 1102-56, D 1107-56, D 1106-56, and D 1103-60.
- BYRD, L., AND E. L. ELWOOD. 1965. Wood characteristics and kraft paper properties of four selected loblolly pines. Forest Prod. J. 15(5):313-320.
- CLERMONT, L. P., AND H. SCHWARZ. 1951. The chemical composition of Canadian wood. Pulp Paper Mag. Canada 52(13):103–105.
- FOREST PRODUCTS LABORATORY METHODS. Madison, WI. Methods 67-033 and 67-018.
- GOETZLER, M. 1982. Final inter-committee report on toluene substitution for benzene in TAPPI analytical procedures. TAPPI 65(3):149–150.
- GRAY, R. 1958. pH in wood. J. Ind. Wood Sci. 1:58.
- GUERNSEY, F., AND J. DOBBIE. 1966. Properties and utilization of lodgepole pine in western Canada. Department of Forestry Publication No. 1143.
- KIM, W.-J. 1988. Chemical characterization of lodgepole pine in North America for use as an industrial raw material. Ph.D. dissertation, University of Idaho, Moscow, ID.
- -----, A. G. CAMPBELL, AND P. KOCH. 1989. Chemical variation in lodgepole pine with latitude, elevation, and diameter class. Forest Prod. J. 39(3):7–12.
- KOCH, P. 1987. Gross characteristics of lodgepole pine trees in North America. USDA Forest Service, General Technical Report INT-227.
- KUTSCHA, N., AND I. SACHS. 1962. Color tests for differentiating heartwood and sapwood in certain softwood tree species. USDA Forest Service, Forest Product Laboratory Report No. 2246.
- MALONEY, T. M. 1982. Particle and fiber building products from residue raw material. USDA Forest Service, Intermountain Forest and Range Exp. Sta. General Technical Report INT-132:171-179.
- MCGOVERN, J. 1951. Pulping of lodgepole pine. USDA Forest Service, Forest Products Laboratory Report No. R1792.
- MCMICHAEL, M. 1982. Utilizing residue material in pulping. USDA Forest Service, Intermountain Forest and Range Exp. Sta. General Technical Report INT-132:194–196.

- McMILLIN, C. 1970. Mineral content of loblolly pinewood as related to specific gravity, growth rate, and distance from pith. Holzforschung 24(5):152–157.
- MCNAMARA, W. S. 1970. pH measurement on northeastern wood. Wood Sci. 3(2):48-51.
- MOSLEMI, A. A., F. GARCIA, AND A. D. HOFSTRAND. 1983. Effect of various treatments and additives on wood-Portland cement-water systems. Wood Fiber Sci. 15(2):164-176.
- PETER, J., G. LIEU, R. KELSEY, AND F. SHAFIZADEH. 1979. Some chemical characteristics of green and dead lodgepole pine and western white pine. USDA Forest Service Note INT-256.
- RITTER, J., AND C. FLEACK. 1926. Chemistry of wood, springwood and summerwood. Ind. Eng. Chem. 18(8):608-609.
- SANDERMANN, W. 1959. On the determination of pH value in timber and their practical importance. Holz Roh- Werkst. 17(8):433.
- TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY. Standard T M-59.
- UPRICHARD, M. 1965. The alpha-cellulose content of wood by the chlorite procedure. APPITA 19(1): 36–39.
- ZOBEL, J., AND L. MCELWEE. 1958. Variation of cellulose in loblolly pine. TAPPI 41(4):167–170.
  —, AND R. STONECYPHER. 1966. Variation and inheritance of cellulose in the southern pine. TAPPI 49(8):383–387.

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