EFFECT OF EXTRACTION ON WOOD DENSITY OF WESTERN HEMLOCK (*TSUGA HETEROPHYLLA* (RAF.) SARG.)

Ryan Singleton

Faculty Research Assistant
Department of Wood Science and Engineering
Oregon State University
Corvallis, OR 97331

Dean S. DeBell

Research Forester (retired)
Pacific Northwest Research Station
3625-93rd Avenue SW
Olympia, WA 98512

and

Barbara L. Gartner†

Associate Professor
Department of Wood Science and Engineering
Oregon State University
Corvallis, OR 97331

(Received September 2002)

ABSTRACT

Extractives can account for between 1 to 20% of the oven-dry weight of wood of various tree species and can influence wood density values appreciably. Removing these chemical deposits (extraction) in wood samples can help establish a consistent baseline for comparing wood densities where extractives are expected to differ between sample parameters. Although western hemlock is a very important timber species in the Pacific Northwest, laboratories that determine wood density may or may not remove extractives prior to density assessment. Wood density values were compared before and after extraction for 19 young-growth western hemlock samples. Extraction was performed using 95% ethyl alcohol-toluene solutions. Ring density values averaged 0.045 g/cm^3 lower for extracted samples compared to unextracted samples across rings. Slightly higher amounts of extractives were found at rings near the pith; however, a general consistency in extractive content existed among samples and along the radial profile.

Keywords: Western hemlock, *Tsuga heterophylla*, extraction, wood density, X-ray densitometry.

INTRODUCTION

Nearly all softwood and many hardwood tree species contain chemical deposits (resins, tannins, gums, alkaloids, etc.) within and between wood cells. These materials (termed 'extractives') tend to be unevenly distributed from pith to bark. Many factors can influence their presence and quantity throughout the woody stem, including but not limited to heartwood/sapwood boundaries, tree ages, growth rates, genetics, and site conditions. Because extractives can account for anywhere from 1 to 20% of the oven-dry weight of wood, they can have marked effects on wood density values. In order to establish a more consistent baseline for comparing wood density values among trees or for examining relationships of other stem traits or tree and site conditions to wood density, the extractives are commonly removed with solvents prior to

† Member SWST.

measuring the density of many tree species (especially in the case of resinous conifers).

There are several methods of extraction, all with the same basic goal of removing these materials that are not part of the cell wall. In the past a widely used method of extraction involved boiling with alcohol-benzene (Voorhies 1969; Zobel and van Buijtenen 1989; Zobel and Jett 1995; TAPPI Standard T204 1987), but most labs no longer use this process because of the hazards of benzene. Other solutions for extraction used more recently include 70% cyclohexane-30% ethanol (Varem-Sanders and Campbell 1996), 1:2 alcohol-cyclohexene (Jozsa 1998), and acetone (Evans, per. comm. 2002). Generally, the samples are immersed in these solutions and boiled for as long as 24 h, sometimes followed or preceded by boiling in water.

Past studies of several softwood species have shown that the presence of extractives generally results in higher overall wood density (Taras and Saucier 1967; Clark and Taras 1969). Older conifer trees tend to have a higher extractive content, likely due to a higher percentage of heartwood (contains larger quantities of resinous material), compared to young trees (Harris 1981; Megraw 1985). Comparing sapwood densities of extracted and unextracted samples, differences of 2–3% were found for *Pinus taeda* (Megraw 1985). Phenolic components were found to be much higher in the inner heartwood compared to the outer sapwood in a 150-year-old western hemlock tree (Barton 1968). Resin contents of 2% for sapwood and 12% for heartwood were found in *Pinus radiata* (Harris 1981).

There are several issues to consider when evaluating whether to extract samples for density analysis. If the goal is to compare density of wood from location to location within a tree, or between trees or between sites, then it needs to be determined whether extractives also vary with these factors, and thus likely to obscure the true differences that are being measured. Zobel and van Buijtenen (1989, p. 11) suggest that "a good estimation of the extracted values can be obtained from unextracted samples. For operational studies on young trees, extractive removal is usually not done; unextracted values will be inflated by about the same amount and useful comparative values are obtained with unextracted wood."

On the other hand, if the amount of structural material per volume is of interest, then the use of extracted values is more appropriate, particularly if there is significant variation in the amount of extractives among the samples one is comparing. To compare wood density to strength among species, it may be more appropriate to use extracted values. Likewise, to infer the relative strength of wood from two sites if extractive content is site-dependent, one can probably make a more accurate comparison using the extracted values.

Although data on extractive contents are available for some softwood species, data for western hemlock (*Tsuga heterophylla* (Raf.) Sarg.) are limited, and there are inconsistencies among laboratories as to whether extraction is routinely done for the species. In this study we used X-ray densitometry to determine mean ring density, earlywood density, and latewood density of western hemlock samples before and after extraction. The objectives were to examine 1) overall differences in densities due to extraction, 2) how such differences in density vary from pith to bark, and 3) how well extracted densities can be predicted from unextracted densities.

METHODS

Study area and samples

The samples for this analysis come from trees felled for a larger research project on industrial forest land in the northern Coast Range of Oregon. The project area extended from Tillamook to the Columbia River and reached inland to include the eastern foothills of the Coast Range mountains. (approximately 46°0′N latitude, 123°45′W longitude). The land base ranges in elevation from approximately 30 to 300 m, annual average precipitation varies from 200 to 250 cm, and the average July maximum and January minimum

TABLE 1. *Attributes of the ten sample tress (crown ratio is the proportion of total tree height that contains live crown).*

	Mean	St. dev.	Min	Max
Dbh (cm)	45.1	20.0	15.6	79.6
Total height (m)	28.8	9.0	14.9	40.1
Crown ratio	0.49	0.13	0.36	0.74
Tree age (yrs)	45.8	15.0	20.0	71.0

temperatures are 21 to 26.5°C and 0 to 4.5°C, respectively. Trees were selected from a list of trees within plots established in pure western hemlock stands that represented a matrix of three stand age, site, and stand density classes. The age classes of stands in the matrix were 1) under 30 years, 2) 30–45 years, and 3) 45–60 years; and both site and stand density were classified as low, medium, and high. For this study, a total of nineteen radial samples were used, selected from 10 trees representing a range of sizes and ages (Table 1). Eighteen of the samples were long and short radii from nine trees, and one sample was a short radius from a tenth tree.

One-inch-thick cross-sectional disks were collected from sample trees at various heights within trees, but only breast height (1.3-m) disks were used in this study. The disks were transported from the field to OSU Forest Research Lab within 4 days after cutting and stored at 5°C to curtail drying and fungal growth. After all the samples were collected and stored, a small cut was made from bark to pith on each disk to prevent radial checking when dried. The disks were then placed on pallets and air-dried using fans to prevent mold.

Two radial samples were sawn from each disk for X-ray density analysis. These were obtained from a bark-to-bark sample through the pith such that it included a short and a long radius, the two of which summed to the average sample diameter. A table saw and band saw were used to cut each disk into strips 7 mm wide (cross-sectional) by 1.6 mm thick, the dimensions needed for X-ray analysis.

X-ray and extraction procedures

The samples were placed in the room where X-raying was performed so they could reach an equilibrium moisture content. The samples were then run through a direct-scanning X-ray wood densitometer (Gartner et al. 2002) to determine density variation within and between growth rings. Data were collected every 200 μm along the scan, then deconvoluted using standard methods (Liu et al. 1988) to give estimates of density back-calculated to dry mass per green volume (g/cm^3). The mass attenuation coefficient needed to convert manipulated values to basic densities was calculated for a subset of eight radial samples before and after extraction.

After the initial X-raying was complete, the samples were extracted and X-rayed again. The extraction procedure was done as follows: The samples were boiled in an 800-ml beaker in 95% ethyl alcohol-toluene solutions. The samples were loaded vertically into the beaker and boiled first with a 2:1 ratio of 95% ethyl alcohol and toluene. Every 1.5 h, the solution was changed and the ratio increased (4:1, 6:1, 10:1, and 20:1) until the last solution was 100% ethyl alcohol. Samples were then spread on blotter paper, weighted to prevent out-of-plane warping, and allowed to air-dry. They were then equilibrated to the moisture content of the X-ray room, then X-rayed and analyzed again as described above.

Data analysis

Paired *t*-tests were performed on samples comparing ring density, earlywood density, and latewood density before and after extraction. The *t*-tests were performed on the means of 5-year ring intervals (rings 1–5, 5–10, 10–15, etc.). The 51–55 year mean ring interval was eliminated because only two observations existed. Differences were considered significant at $\alpha = 0.005$, after applying Bonferroni's simultaneous inference method (Miller 1981) for simultaneous paired *t*-tests. In addition, radial density profiles were plotted in order to

TABLE 2. *Paired* t-*test results of 5-year ring intervals for total ring, earlywood, and latewood density, comparing extracted to unextracted samples.*

		Ring density		Earlywood density		Latewood density	
N	Rings	T	p-value	T	p-value	T	p-value
18	1–5	13.59	<0.0001	12.07	<0.0001	8.31	<0.0001
19	6–10	17.74	<0.0001	16.43	<0.0001	12.49	<0.0001
19	11–15	30.20	<0.0001	16.66	<0.0001	13.91	<0.0001
19	16–20	18.25	<0.0001	17.04	<0.0001	15.42	<0.0001
17	21–25	26.83	<0.0001	14.27	<0.0001	13.85	<0.0001
17	26–30	9.65	<0.0001	13.72	<0.0001	10.49	<0.0001
15	31–35	24.65	<0.0001	13.33	<0.0001	12.60	<0.0001
12	36–40	15.21	<0.0001	10.01	<0.0001	7.07	<0.0001
10	41–45	14.39	<0.0001	15.84	<0.0001	8.57	<0.0001
8	46–50	14.33	<0.0001	9.24	<0.0001	5.87	0.0006
19	all rings	43.26	<0.0001	35.26	<0.0001	23.36	<0.0001

examine the magnitude of the differences from pith to bark.

A simple linear regression equation was developed to determine how well extracted ring density can be predicted from unextracted density. The model was based on the overall means (across all rings) for each sample. This equation was then applied to the means of rings 1–10 to determine how well a model across all rings can predict specific parts of the radial profile.

RESULTS

Extracted samples were significantly different from unextracted samples for all 5-year interval groups for ring density, earlywood density, and latewood density (Table 2). Mean density across all rings was 0.45 before extraction and 0.41 after extraction (Table 3a).

Ring density was on average 0.04 to 0.06 (9% to 13%) lower for extracted samples than for unextracted samples, depending on interval compared (Table 3a). Earlywood density values averaged 0.03 to 0.05 (9% to 13%) lower for extracted vs. unextracted samples (Table 3b), and latewood density averaged 0.05 to 0.07 (9% to 14%) lower for extracted samples than for unextracted samples (Table 3c).

The differences in extracted and unextracted densities were relatively constant across radial gradients for ring density (although slightly higher for rings 1–10), as shown from the small range of percentage differences across

TABLE 3a. *Total ring density (g/cm^3) of unextracted vs. extracted western hemlock samples by growth ring intervals from the pith (mean, standard error, and differences between unextracted and extracted).*

	No extract		Extract			
Rings	Mean	St Err	Mean	St Err	Actual difference	Percent difference
1–5	0.509	0.007	0.451	0.006	0.06	12.7%
6–10	0.453	0.006	0.404	0.005	0.05	12.2%
11–15	0.434	0.005	0.394	0.005	0.04	10.3%
16–20	0.431	0.004	0.391	0.004	0.04	10.1%
21–25	0.446	0.006	0.401	0.006	0.05	11.2%
26–30	0.447	0.006	0.408	0.006	0.04	9.5%
31–35	0.454	0.005	0.410	0.005	0.04	10.8%
36–40	0.454	0.005	0.415	0.005	0.04	9.2%
41–45	0.477	0.007	0.430	0.007	0.05	10.9%
46–50	0.460	0.009	0.414	0.009	0.05	11.2%
all rings	0.455	0.002	0.410	0.002	0.04	10.9%

TABLE 3b. *Earlywood density (g/cm³) of unextracted vs. extracted western hemlock samples by growth ring intervals from the pith (mean, standard error, and differences between unextracted and extracted).*

Rings	No extract		Extract		Actual difference	Percent difference
	Mean	St Err	Mean	St Err		
1–5	0.465	0.007	0.411	0.005	0.05	13.0%
6–10	0.408	0.005	0.365	0.005	0.04	11.8%
11–15	0.385	0.004	0.349	0.004	0.04	10.4%
16–20	0.382	0.004	0.348	0.003	0.03	9.7%
21–25	0.394	0.004	0.355	0.004	0.04	10.9%
26–30	0.395	0.005	0.361	0.005	0.03	9.7%
31–35	0.392	0.005	0.354	0.004	0.04	10.7%
36–40	0.386	0.005	0.354	0.004	0.03	8.8%
41–45	0.395	0.006	0.354	0.005	0.04	11.6%
46–50	0.397	0.007	0.353	0.008	0.04	12.6%
all rings	0.401	0.002	0.361	0.002	0.04	10.9%

ring intervals (Table 3a). Further support of the consistency of extractive material was found when examining pith to bark profiles for each variable (Fig. 1a). Earlywood and latewood density showed a similar pattern as ring density with the exception of ring interval 46–50, which had greater mean differences between extracted and unextracted densities. However, a relatively high amount of variability was associated with these ring interval means, possibly influencing these differences (Tables 3b–c, Figs. 1b–c).

A large proportion of the variation of the extracted ring densities was explained by unextracted ring densities ($R^2 = 0.984$) (Fig. 2). The model performed well in the prediction of extraction values for the means of rings 1–10 (Fig. 3). The absolute value of the residuals ranged from 0.0018 g/cm³ to 0.026 g/cm³. For predicting ring groups 10–20, 20–30, 30–40, and 40–50 from this model, the mean of the absolute values of the residuals for each of group was less than for the prediction of rings 1–10, suggesting that this model was sufficient for predicting all of the ring groups.

DISCUSSION AND CONCLUSIONS

The effect extraction can have on wood density analysis depends on the factors being evaluated. A difference of 0.02 g/cm³ in wood density can be rather important; it can change the modulus of rupture by about 1000 lbs/in² (70.4 kg/cm²) (Mitchell 1963) and the yield of

TABLE 3c. *Latewood density (g/cm³) of unextracted vs. extracted western hemlock samples by growth ring intervals from the pith (mean, standard error, and differences between unextracted and extracted).*

Rings	No extract		Extract		Actual difference	Percent difference
	Mean	St Err	Mean	St Err		
1–5	0.614	0.007	0.555	0.004	0.06	10.5%
6–10	0.590	0.005	0.535	0.005	0.06	10.4%
11–15	0.588	0.006	0.535	0.005	0.05	9.9%
16–20	0.568	0.005	0.519	0.005	0.05	9.6%
21–25	0.595	0.007	0.533	0.006	0.06	11.6%
26–30	0.594	0.007	0.539	0.007	0.06	10.3%
31–35	0.591	0.006	0.535	0.006	0.06	10.5%
36–40	0.604	0.008	0.552	0.007	0.05	9.4%
41–45	0.603	0.009	0.551	0.006	0.05	9.4%
46–50	0.599	0.017	0.527	0.015	0.07	13.7%
all rings	0.593	0.002	0.537	0.002	0.06	10.4%

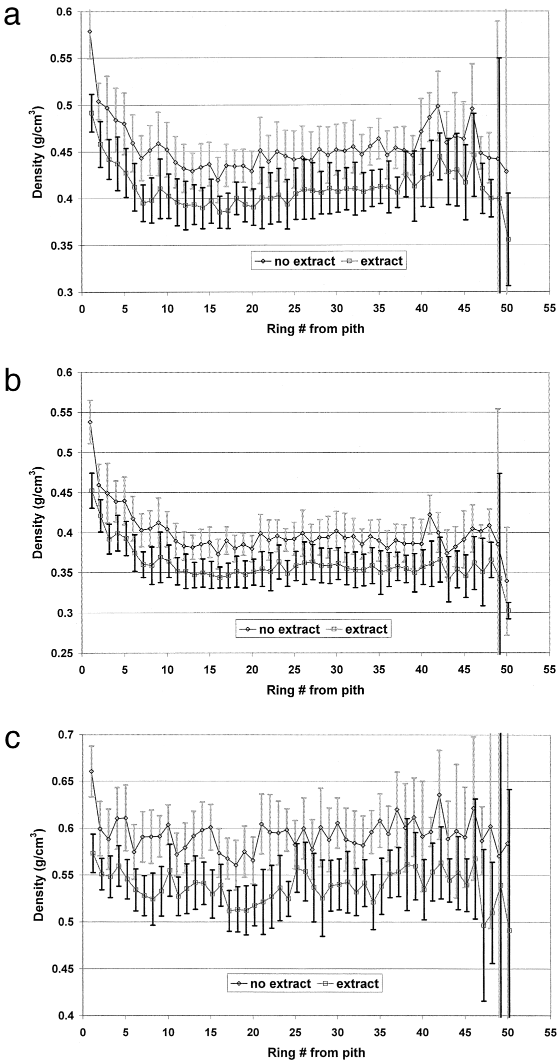

Fig. 1(a–c). Density profiles (means with standard error bars) of extracted and unextracted samples for total (a) ring density, (b) earlywood density, and (c) latewood density.

pulp from a cord of pulpwood by 50 lbs (22.7 kg) (Zobel and van Buijtenen 1989). Thus, in genetic evaluations or timber assessments, we may wish to identify genotypes or geographic sources that differ by 0.02 g/cm^3 from the norm. Similarly, in basic studies of factors that influence density, we may want to detect any trends in density with growth rate, site conditions, stand spacing, or with other attributes that lead to a change of 0.02 g/cm^3 or more.

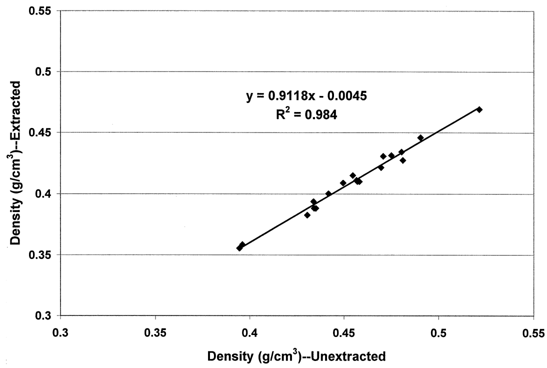

Fig. 2. Simple linear regression with plotted overall mean density values for extracted vs. unextracted samples.

Data from the current study indicate that variation around any adjustment of nonextracted values is not great enough to mask detection of differences of 0.02 g/cm^3 in structural wood density (average residual = 0.0094 g/cm^3). However, applying a model outside a study's scope of inference would pose a significant risk of loss of accuracy, particularly when considering the effect of a small difference in density. Thus, if valid extracted values are desired, at least a subset of samples should be extracted in order to build an applicable model.

It is evident from the analyses that significant amounts of extractives are present in young western hemlock. The differences between extracted and unextracted samples varied between 9% and 14% for total ring density, earlywood density, and latewood density.

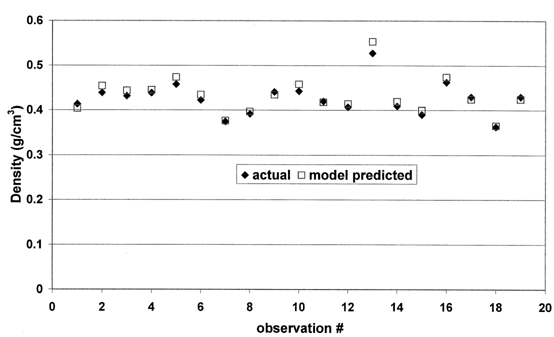

Fig. 3. Actual extracted mean density values and model estimated extracted density values for rings 1–10.

Rings 1–10 tended to have slightly more extractives than other ring intervals, although in general the amount of extractives tended to remain consistent across radial gradients in these samples. Differences between extracted and unextracted wood densities near the pith appeared to be the same as wood in the middle and outer rings. Some of this may be due to having samples of relatively young trees; thus the presence of heartwood may have been minimal. If greater amounts of heartwood were present in these trees, we might have observed much higher overall differences before and after extraction in the inner rings.

Density difference caused by extractions was similar across the radius and among samples, suggesting it is appropriate to compare unextracted samples to look at density variation. In contrast, where accurate estimations of extracted density values are appropriate (such as when strength properties are related to density), study-specific models estimating extracted density from unextracted density are needed.

ACKNOWLEDGMENTS

This research was supported by the Wood Compatibility Initiative from the PNW Research Station, Olympia, Washington. We thank David Marshall for his support and guidance throughout the entire study. The authors would like to acknowledge Willamette Industries for providing the land base for this project and, in particular, the support of Greg Johnson. We also thank Bob Krahmer and Sean Canavan for helpful review comments.

REFERENCES

BARTON, G. M. 1968. Significance of western hemlock phenolic extractives in pulping and lumber. Forest Prod. J. 18(5):76–80.

CLARK, A., AND M. A. TARAS. 1969. Wood density surveys of the minor species of yellow pine in the eastern United States, II—Sand pine (*P. clausa*). U.S. For. Serv. Res. Pap. SE-51. 14 pp.

EVANS, R. Personal communication. Feb. 24, 2002.

GARTNER, B. L., E. M. NORTH, G. R. JOHNSON, AND R. SINGLETON. 2002. Wood density in relation to live crown position in 34-year-old trees of Douglas-fir (*Pseudotsuga menziesii*). Can. J. For. Res. 32:439–447.

HARRIS, J. M. 1981. Wood quality of radiata pine. NZ For. Ser. Reprint 1474. Rotorua, New Zealand. pp. 211–215.

JOZSA, L. A. 1998. Basic wood properties of second-growth western hemlock. Forintek Canada Corp. Special Publication No. Sp-38. 51 pp.

LUI, C. J., OLSEN, J. R., TIAN, Y., AND Q. SHEN. 1988. Theoretical wood densitometry: I. Mass attenuation equations and wood density models. Wood Fiber Sci. 20:22–34.

MEGRAW, R. A. 1985. Wood quality factors in loblolly pine. TAPPI Press, Atlanta, GA. 89 pp.

MILLER, R. G., JR. 1981. Simultaneous statistical inference. Springer-Verlag, New York, NY. 299 pp.

MITCHELL, H. L. 1963. Specific gravity variation in North American conifers. IUFRO Sect 41 Committee on Fiber Characteristics. Melbourne, Australia, Vol. 2, 15 pp.

TARAS, M. A., AND J. R. SAUCIER. 1967. Influence of extractives on specific gravity in southern pines. Forest Prod. J. 17:97–99.

TECHNICAL ASSOCIATION OF PULP AND PAPER INDUSTRY (TAPPI). 1987. Solvent extractives of wood and pulp. Approved by the Chemical Properties Committee of the Process and Product Quality Division, TAPPI, Atlanta, Ga. Tech. Inf. Sheet T 204 1–88.

VAREM-SANDERS, T. M. L., AND I. D. CAMPBELL. 1996. Dendroscan: A tree-ring width and density measurement system. Nat. Resour. Can., Can. For. Serv., North. For. Cent., Edmonton, Alberta. Spec. Rep. 10.

VOORHIES, G. 1969. Specific gravity studies of young-growth southwestern ponderosa pine. Forest Prod. J. 19(6):45–46.

ZOBEL, B. J., AND J. B. JETT. 1995. Genetics of wood production. Springer-Verlag, New York, NY. 337 pp.

———, AND J. P. VAN BUIJTENEN. 1989. Wood variation: Its causes and control. Springer-Verlag, New York, NY. 363 pp.