

# DRYING AND OTHER RELATED PROPERTIES OF WESTERN HEMLOCK SINKER HEARTWOOD<sup>1</sup>

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## ABSTRACT

Sinker heartwood dries more slowly than normal heartwood of western hemlock. Extraction with ethanol or acetone and presteaming improved the rate of drying of sinker heartwood. Electron microscopy was used to examine bordered pits in normal and sinker heartwood and extracted specimens of sinker heartwood. Parallel capacitance was lowest in sapwood, higher in normal heartwood, and highest in sinker heartwood. The range in capacitance was related to total extractive content at similar moisture contents.

## INTRODUCTION

Western hemlock, *Tsuga heterophylla* (Raf.) Sarg., is a major saw timber and pulping species from southern Alaska to northern California and east into the Inland Empire region. In processing western hemlock lumber, a major problem is the large variation in moisture content after kiln drying. This problem has been discussed by Kozlik (1970) and is attributed directly to heartwood, which often includes zones of wood termed "wetwood," "sinker heartwood," or "heavywood" because of their very high moisture contents. Sinker heartwood in unseasoned material has a distinctive odor, is characteristically darker, and appears wetter than adjacent normal heartwood. Ring shake often is associated with sinker heartwood.

This study was initiated to examine drying characteristics, anatomy, and dielectric properties of sinker heartwood. Results reported in this paper include initial moisture contents, specific gravities, drying rates, anatomical observations, and dielectric properties.

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## MATERIAL AND METHODS

The timber was from the Snow Peak area, which is east of Albany, Oregon, in the Cascade Range. Two-inch dimension and shop lumber were collected from the green sorting chain and pieces 2 ft long were sawed from the end of each piece of lumber. Pieces with vertical or near-vertical grain were selected because wet zones would occur as well-defined strips along the grain and a rapid visual assessment of the area of sinker and normal heartwood in each piece could be made. A time-and-count system was used to select lumber so that each piece would come from a different log. No effort, however, was made to insure that each piece was from a different tree.

A total of 54 pieces of western hemlock heartwood were sampled. During sampling, storage, and handling, care was taken to insure against moisture loss.

Specific gravity and initial moisture content were measured on a specimen cut 2 inches from the freshly sawed end of each piece. For each specimen, the zones of sinker heartwood were separated from the

oratory, University of California, Berkeley, directed operation of the Cambridge Stereoscan Mark II Scanning Electron Microscope, and B. M. Collett, Forest Products Laboratory, Berkeley, assisted. The equipment was purchased under Grant No. GB-6428 from the National Science Foundation, and is operated under Grant No. GM15536 from the National Institute of Health.

normal heartwood zones, and the moisture content and specific gravity of each zone were determined.

Drying rates, dielectric properties, and anatomy were compared for several types of samples.

#### *Drying rates*

Matched specimens of sinker and normal heartwood were compared for drying rates.

Fifteen of the pieces sampled at the mill had a strip of sinker heartwood at least 2.5 inches wide immediately adjacent to a strip of normal heartwood at least 2.5 inches wide. One specimen each of sinker heartwood and normal heartwood was sawed from these adjacent zones in each piece and surfaced to  $\frac{3}{4}$  by 2 by 18 inches ( $\pm 0.003$  inch). These matched specimens were placed in a drying chamber controlled at 100 F and an equilibrium moisture content (EMC) of 3.8% for 190 hr, or until the specimens nearly reached equilibrium. Sixteen weighings were made during the drying time, followed by oven-drying for moisture content calculations.

Matched specimens of sinker heartwood unextracted (control), extracted with ethanol or acetone, and presteamed were compared.

Adjacent specimens of sinker heartwood were sawed across the thickness of the original sample and surfaced to  $\frac{1}{4}$  by 2 by 18 inches ( $\pm 0.003$  inch). Twenty-nine specimens were extracted with ethanol in a pressure vessel held at 125–130 F at atmospheric pressure for 140 hr. Thirty-seven specimens were extracted with acetone in a pressure vessel at 145 F at 4 to 5 psi for 168 hr. The solvent was changed after 80 hr in each extraction process. After each extraction, specimens were stickered and fan-dried for 10 days to remove excess solvent. Thirty-seven specimens were steamed in a small kiln held at 212 F and 99% relative humidity. A fourth specimen was cut as the control. Steamed and control specimens were fan-dried for 10 days with the extracted specimens to ensure similar treatments to all specimens. The moisture

content of the specimens, however, was not calculated after fan-drying. One would expect the moisture content of the extracted and steamed specimens to be lower than that of the control specimens, because drying would occur during extracting and steaming.

To restore the extracted and steamed specimens to their original unseasoned moisture content, all specimens including the controls were placed in water and treated at a vacuum of 29.9 inches of mercury for 3 hr, followed by 100 psi air pressure for 18 hr. The average green weight of all specimens before any extraction or steaming and after the water treatment tested not significantly different at the 5% level of probability.

For one comparison, the drying rates of 29 specimens that had been ethanol-extracted were compared with the drying rates of the unextracted (control) specimens. Drying conditions were 70 F at 11% EMC for 94 hr, followed by oven-drying. The specimens were weighed six times during the drying period.

For another comparison, the drying rates of 37 matched sets of three specimens were compared. The comparison was for specimens that had been acetone-extracted, steamed, and unextracted (control). Drying conditions were 70 F at 11.3% EMC for 64 hr, followed by oven-drying. Five weighings were made during the drying period.

Drying rates of matched specimens were compared by calculating moisture content, based on oven-dry weight, and expressing average drying rates as (1) moisture content over time; as (2)  $E$ -value over time, where  $E$  is the fraction of evaporable water remaining in the specimen at any given time  $\theta$  and is calculated from the equation  $E = (w - c_1)/(c_0 - c_1)$ , where  $w$  is the moisture content of the specimen at time  $\theta$ ,  $c_1$  is the equilibrium moisture content for the drying conditions used, and  $c_0$  is the original moisture content of the specimen; or as (3) a diffusion coefficient calculated from the straight-line segment of the plot of logarithm of  $E$  and time (Lin and Kozlik 1971).

### *Dielectric properties*

Capacitance (dielectric property) at 1 kHz was measured to examine the possibility of characterizing western hemlock sapwood, normal heartwood, and sinker heartwood. The effect of moisture content above the fiber saturation point on dielectric behavior also was studied.

Specimens were cut from unseasoned pieces sampled at the mill. From each piece, many specimens 3 inches (longitudinal) by 2 inches (radial) by  $\frac{1}{4}$  inch (tangential) were prepared and characterized visually as sapwood, normal heartwood, or sinker heartwood. Sapwood specimens were cut from those pieces that contained the cambium zone by having obvious wane. A total of 658 specimens were prepared for dielectric measurements.

The four-pin electrodes of a Delmhorst moisture detector were connected to the input terminals of a General Radio 1650 Impedance Bridge. Increase in R-C parallel capacitance ( $\Delta C$ ) was measured after electrodes had been inserted  $\frac{1}{4}$  inch into the green specimens.

Moisture contents of all specimens were determined by the oven-drying method. The extractive content also was determined for 17 sapwood, 17 normal heartwood, and 23 sinker heartwood specimens that had been selected randomly from those specimens with moisture contents between 90 and 110% and used for dielectric measurements. These specimens were ground in a Wiley mill to pass a 2-mm screen, extracted for 48 hr in cold water (23 C), for 48 hr in hot water (70 C), and finally for 48 hr in benzene-alcohol (1:2 mix) in a soxhlet extractor.

### *Anatomical studies*

Bordered pit membranes from normal heartwood and sinker heartwood were examined with the transmission electron microscope, by the direct carbon replica technique (Côté, et al. 1964) and with the scanning electron microscope by the technique outlined by Collett (1970). Specimens used in electron microscopy were cut

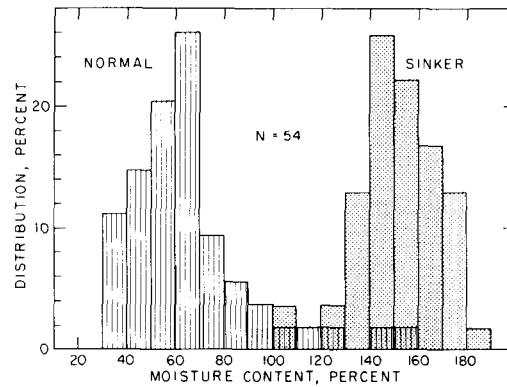


FIG. 1. Distribution of unseasoned moisture contents in normal and sinker heartwood.

from the oven-dried controls used for comparison of drying rates of acetone-extracted, steamed, and control specimens.

### RESULTS AND DISCUSSION

The initial moisture content averaged 66.1% (range, 32.9 to 152.4%) for normal heartwood and 153.0% (range, 108.4 to 185.7%) for sinker heartwood (Fig. 1). The high initial moisture content for some normal heartwood zones might be attributed to the presence of sapwood. Only five specimens of the normal heartwood out of 54 had moisture contents above 100%, however.

Average specific gravities, based on green volume and oven-dry weight, were 0.426 for

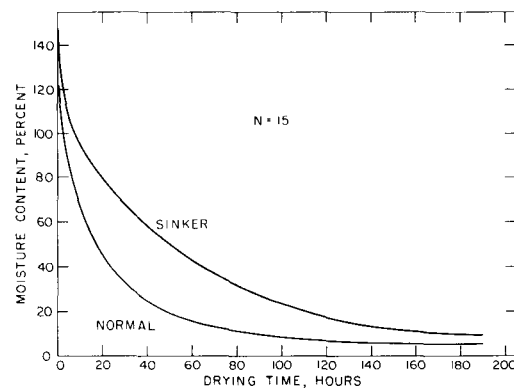


FIG. 2. Drying curves for normal and sinker heartwood.

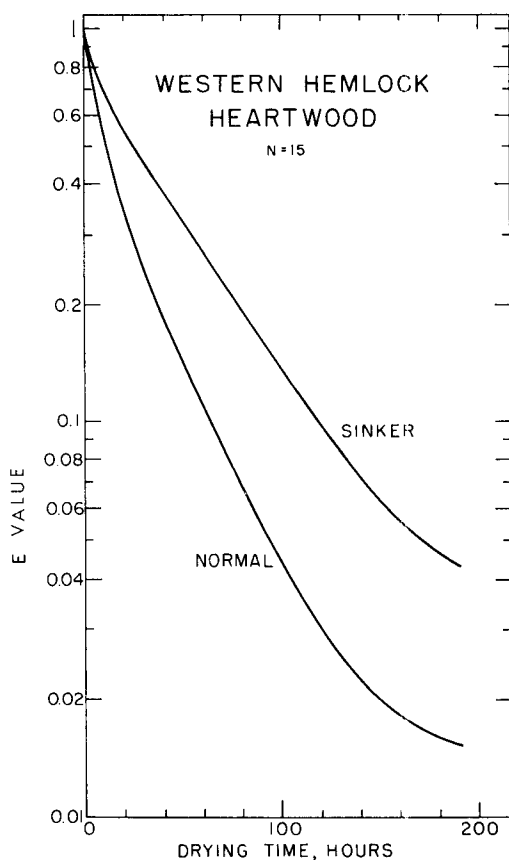


FIG. 3. Comparison of evaporable water in normal and sinker heartwood.

normal heartwood and 0.443 for sinker heartwood. This difference was significant at the 5% level of probability, and Schroeder and Kozlik (1972) concluded that the higher specific gravity for sinker heartwood could be accounted for fully by a higher content of extractives.

#### *Drying rates*

The relation of moisture content and time for sinker and normal heartwood is shown in Fig. 2. A large difference exists between the two curves because the normal heartwood reached a lower moisture content in the shortest time. There was a significant difference (at the 1% level of probability) between the average moisture contents for normal and sinker heartwood speci-

mens at each of the 16 times during the drying period when the specimens were weighed.

The  $E$ -time relation for sinker and normal heartwood is shown in Fig. 3. Normal heartwood again exhibits faster drying. Analysis of average  $E$ -values for sinker and normal heartwood at each of the 16 weighing periods throughout the drying cycles was significantly different at the 1% level of probability.

The average diffusion coefficient for normal heartwood was  $1.3891 \text{ cm}^2/\text{hr}$  with a standard deviation of  $0.20 \text{ cm}^2/\text{hr}$ , and for sinker heartwood  $1.1688 \text{ cm}^2/\text{hr}$  with a standard deviation of  $0.26 \text{ cm}^2/\text{hr}$ . Analysis of variance for difference between the average diffusion coefficients of normal heartwood specimens and sinker heartwood specimens was significant at the 1% level of probability. Therefore, the three methods of calculating moisture loss showed that sinker heartwood reaches a lower moisture content significantly more slowly than normal heartwood. Because the movement of water from wet pockets in hemlock during initial stages of drying possibly is dependent on liquid flow through openings in the cell wall, the lack of much drying of these wet pockets could indicate that deposits of extractives and other encrusting materials are blocking these openings. The primary openings between contiguous tracheids are bordered pit pairs, and deposits on the pit membranes have been shown to reduce the porosity and permeability of wood (Sebastian et al., 1965).

Bordered pit membranes were examined from both the air-dried sinker heartwood and the normal heartwood cells by the direct carbon technique for electron microscopy. Differences in the amount of materials deposited on the membranes from each zone were quite noticeable, as shown in Figs. 4, 5, and 6.

Pit membranes such as the one shown in Fig. 4 were observed in the heartwood zones. The incrustation of this pit membrane is typical for pits found in normal heartwood of western hemlock (Krahmer and Côté 1963). Openings are still evident

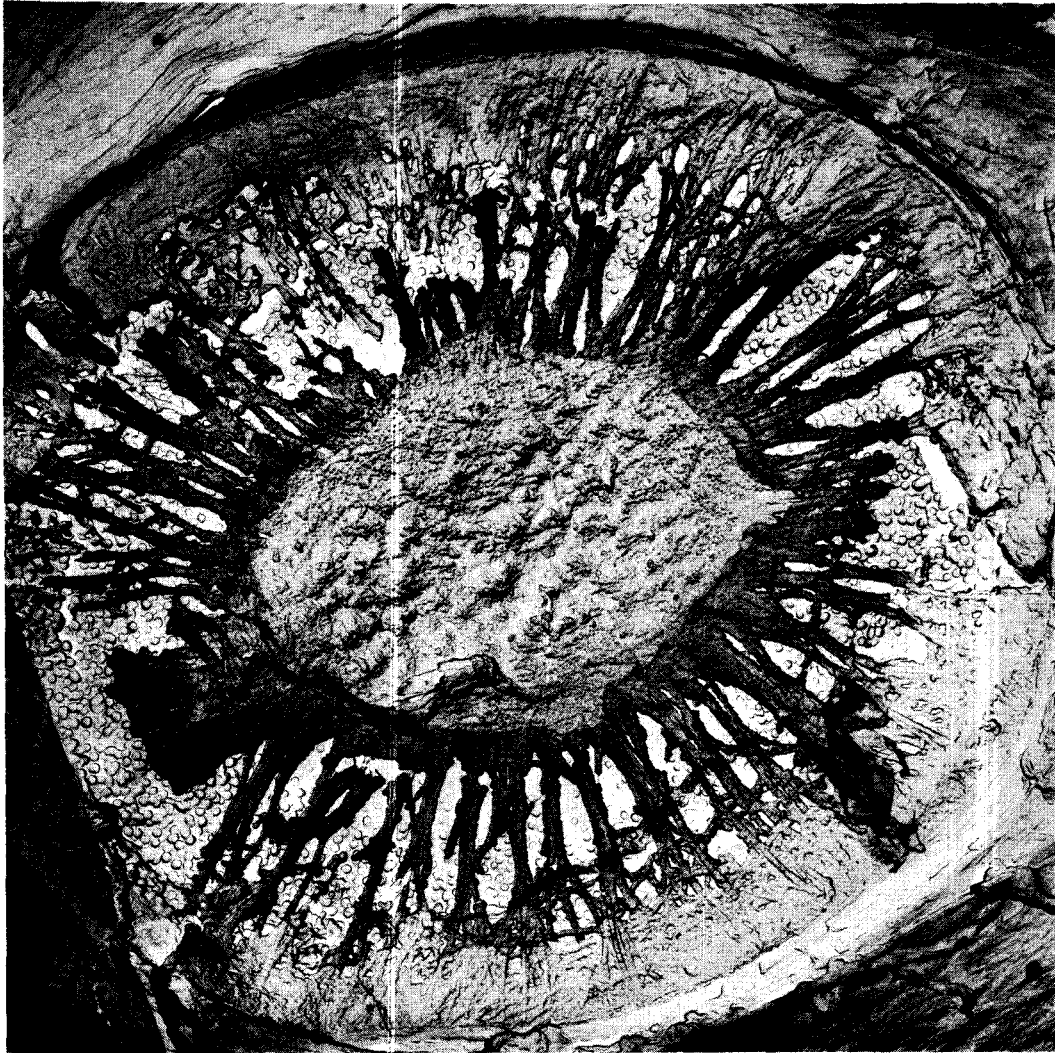


FIG. 4. Bordered pit membrane in normal heartwood of western hemlock. Incrustation of the pit membrane is typical of that observed in normal heartwood. 7500 $\times$ .

between the strands in the margo of the membrane. If pit aspiration has not completely sealed the aperture because of surface irregularities of the torus and surface irregularities of the pit border caused by a warty layer, fluid movement would be possible through this pit pair.

In the sinker heartwood, pit membranes such as those shown in Fig. 5 were observed. The membranes are coated completely with deposited materials and a minimal amount of liquid flow would be expected through

these pits. Diffusion through the cell wall could be reduced because of the heavy deposits also observed on the surface of the cell wall adjacent to the lumen (Fig. 6).

If the deposits were removed from pit areas and cell walls, an improved drying rate in sinker heartwood might be expected. This was examined by comparing the drying rates for unextracted, extracted, and steamed sinker heartwood.

A different drying schedule was used to compare the drying rates of ethanol-ex-



FIG. 5. Heavily encrusted bordered pit membrane observed in sinker heartwood of western hemlock. 7500 $\times$ .

tracted and unextracted specimens than was used for acetone-extracted and steamed specimens. Therefore, matched pairs of ethanol-extracted and unextracted specimens of sinker heartwood were prepared and a statistical analysis of drying rates was made for this comparison alone. Figure 7 shows the  $E$ -time relationship, and Fig. 8 shows the moisture content-time curve. These results show the extracted specimens reached EMC sooner than the unextracted or control specimens. Analysis of variance

for difference in average  $E$ -values and moisture contents at the six weighing periods was significant at some of the weighing times, as shown in Table 1.

Average diffusion coefficient for unextracted specimens was  $0.3981 \text{ cm}^2/\text{hr}$ , with a standard deviation of  $0.06 \text{ cm}^2/\text{hr}$ , and for extracted specimens  $0.3761 \text{ cm}^2/\text{hr}$ , with a standard deviation of  $0.08 \text{ cm}^2/\text{hr}$ . No significant difference (at the 5% level of probability) was found, however, between the average diffusion coefficients of un-

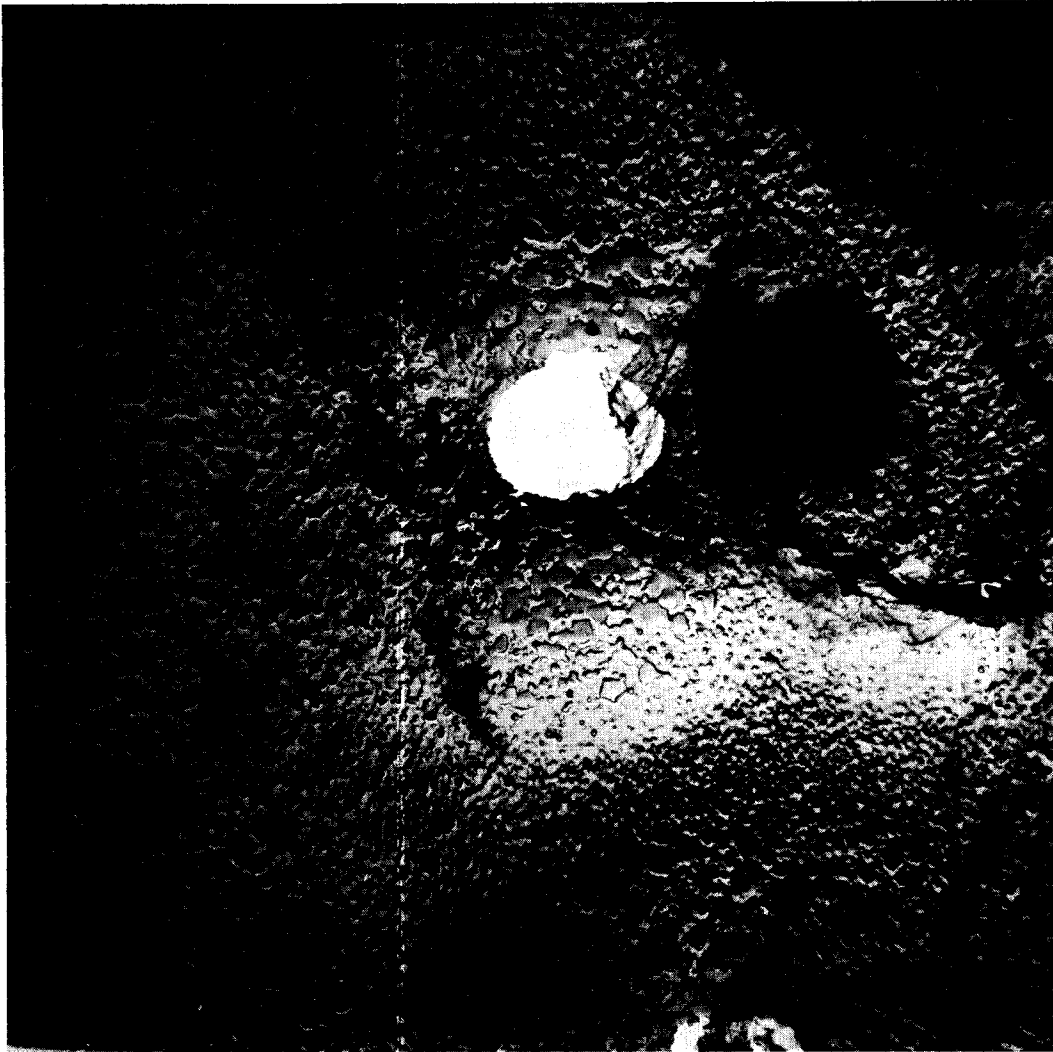


FIG. 6. Deposits of encrusting materials on the cell wall in sinker heartwood of western hemlock. 4000 $\times$ .

extracted and extracted specimens and, therefore, no difference in drying rates, at least during the drying time between about 20 and 45 hr, where the  $E$ -time curve is a straight line. Although there were significant differences in moisture contents and  $E$ -values during the 46- to 70-hr drying period, these actual differences are small and ethanol extraction of sinker heartwood appeared to have only a slight influence on the drying properties. The total amount of extractives re-

moved by ethanol was not calculated, but complete extraction of the individual specimens might not have been accomplished and longer extraction times possibly would change these results. In a study by Meyer and Barton (1971), the difficulty of totally extracting solid wood was illustrated for western red cedar.

A group of 37 matched sets of three specimens of sinker heartwood was used to compare drying rates of acetone-extracted, steamed, and control specimens. The  $E$ -

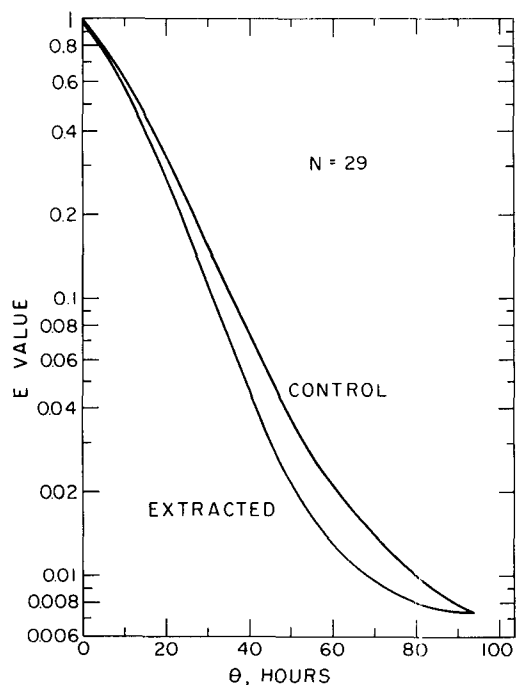


FIG. 7. Comparison of evaporable water in ethanol-extracted and control specimens of sinker heartwood.

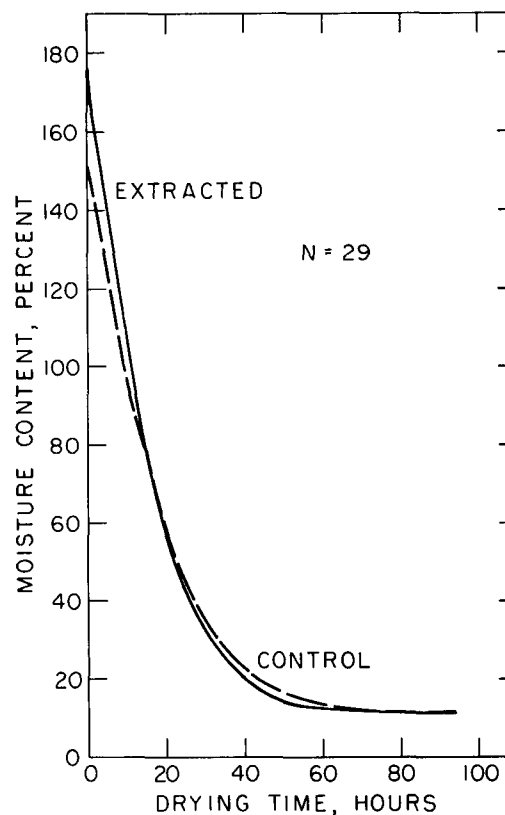


FIG. 8. Drying curves for ethanol-extracted and control specimens of sinker heartwood.

time relation is illustrated in Fig. 9 and the moisture content-time curve is shown in Fig. 10.

Drying time was reduced by acetone extraction and steaming of sinker heartwood. Past studies (Ellwood and Erickson 1962; Ellwood and Benvenuti 1964) have shown increased permeability in redwood and southern pine by presteaming. Removal of extractive materials with acetone and hot water from steaming perhaps caused significant removal of extractives from pit areas to provide freer movement of mois-

ture. Figures 11 and 12 are scanning electron micrographs of bordered pits in acetone-extracted specimens. Some removal of incrustations, along with actual rupturing of the membrane, would appear to make these pits more permeable. Analysis of variance of  $E$ -values and moisture contents at the five weighing periods had significant differences when comparing acetone-ex-

TABLE 1. Analysis of variance of  $E$ -values and moisture contents of ethanol-extracted and control specimens of sinker heartwood

Source	F-value at each weighing period <sup>1</sup>					
	24 hr	30 hr	46 hr	54 hr	70 hr	95 hr
Control and Extracted						
E-value	1.821	5.499*	12.14**	13.63**	22.61**	0.256
Moisture Content	0.245	2.682	8.570**	9.881**	14.88**	6.400*

<sup>1</sup> F-distribution with 1 and 56 degrees of freedom; \* indicates significance at the 0.05 level and \*\* at the 0.01 level of probability.



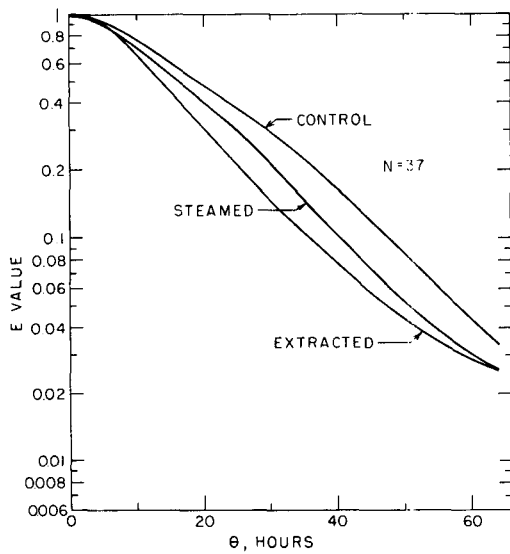


FIG. 9. Comparison of acetone-extracted, steamed, and control specimens of sinker heartwood.

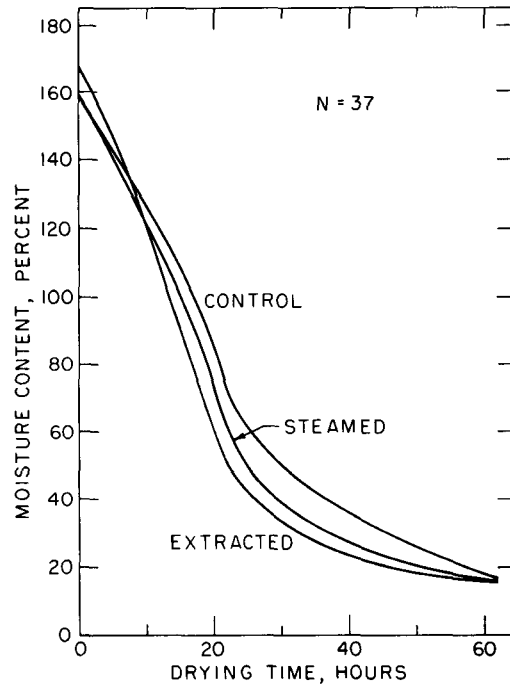


FIG. 10. Drying curves for acetone-extracted, steamed, and control specimens of sinker heartwood.

tracted, steamed, and unextracted (control) specimens, as shown in Table 2. After about 10 hr of drying, the extracted and steamed specimens averaged lower moisture contents than the control specimens, and the extracted specimens approached EMC sooner than did the steamed specimens.

Average diffusion coefficient for unextracted specimens was 0.4716 cm<sup>2</sup>/hr, with a standard deviation of 0.04 cm<sup>2</sup>/hr; for extracted specimens was 0.4949 cm<sup>2</sup>/hr,

with a standard deviation of 0.03 cm<sup>2</sup>/hr; and for steamed specimens was 0.5082 cm<sup>2</sup>/hr, with a standard deviation of 0.05 cm<sup>2</sup>/hr. Results of analysis of variance for differences in averages of diffusion coefficients were: control and acetone-extracted specimens were significantly different at the

TABLE 2. Analysis of variance of E-values and moisture content of acetone-extracted, steamed, and control specimens of sinker heartwood

Source	F-value at each weighing period <sup>1</sup>				
	7 hr	16 hr	24 hr	40 hr	64 hr
Control and Extracted					
E-value	8.740**	48.61**	34.80**	34.35**	18.83**
Moisture Content	0.269	19.50**	17.32**	21.73**	24.66**
Control and Steamed					
E-value	11.52**	8.470**	7.663**	12.98**	10.71**
Moisture Content	0.338	3.460	4.336*	9.988**	20.13**
Extracted and Steamed					
E-value	0.002	23.05**	9.934**	4.672*	0.276
Moisture Content	1.177	8.556**	4.713*	2.404	0.310

<sup>1</sup> F-distribution with 1 and 72 degrees of freedom; \* indicates significance at the 0.05 level and \*\* at the 0.01 level of probability.

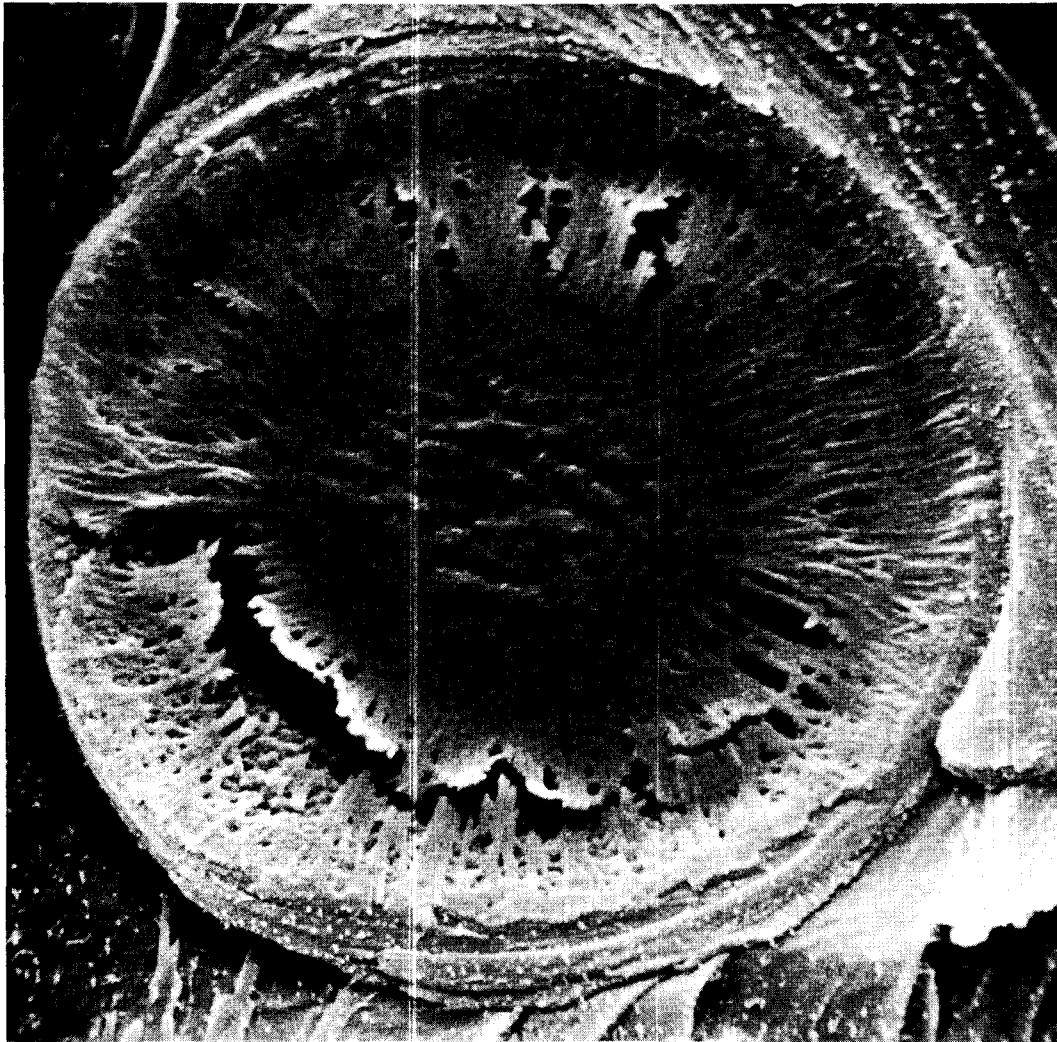


FIG. 11. Scanning electron micrograph of a bordered pit membrane in acetone-extracted sinker heartwood. Removal of some encrusting materials appeared to occur in this extracted wood. 8000 $\times$ .

5% level of probability; control and steamed specimens were significantly different at the 1% level of probability; and extracted and steamed specimens had no significant difference. The moisture content-time and  $E$ -time curves (Figs. 9 and 10) indicate that the extracted specimens dried faster than the steamed specimens. The average diffusion coefficient for steamed specimens, however, was greater than that for extracted specimens merely because of the position of the  $E$ -time curve where the diffusion

coefficient was measured. During the first 30 hr of drying, the extracted specimens have a steeper slope of line on the  $E$ -time curve, but after 30 hr the steamed specimens have a steeper slope, which indicates that in the initial part of drying the extracted specimens had a higher diffusion coefficient than the steamed specimens, but after approximately 30 hr of drying the steamed specimens had a higher diffusion coefficient than the extracted specimens.



FIG. 12. Scanning electron micrograph of ruptured bordered pit membrane in acetone-extracted sinker heartwood. 13,000 $\times$ .

#### *Dielectric properties*

The effect of moisture content of parallel capacitance was observed to determine the possibility of characterizing sapwood, normal heartwood, and sinker heartwood. Moisture contents of test specimens ranged from 25 to 204% and the parallel capacitance ( $\Delta C$ ) ranged from 190 to 3,560 picofarads (pF). The parallel capacitance was plotted against those moisture contents above the fiber-saturation point, where points on the graph were quite scattered

and were not significantly different at the 5% level of probability for sapwood, heartwood, and wetwood. Scattering of points was greater at higher moisture levels; however, parallel capacitance at a given moisture content was highest for wetwood and lowest for sapwood.

The parallel capacitance of wood is in part attributable to a complex mode of polarization, which includes ionic and dipolar polarization. These two modes of polarization are a function of the number

TABLE 3. *Extractive contents of western hemlock of different dielectric behavior at moisture range of 90-110 %*

Group	Capacitance range (pf)	Type	Number of specimens	Extraction <sup>1</sup>		
				Part	Range (%)	Average (%)
I	300-600 Avg 504	Sap Heart	15 3	Cold	0.148-1.396	0.550
				Hot	0.423-0.978	0.652
				B-a	0.408-0.706	0.530
				Total	1.170-2.914	1.732
II	600-900 Avg 794	Sap Heart Wet	2 12 1	Cold	0.731-2.980	1.660
				Hot	0.235-6.475	2.601
				B-a	0.392-2.385	1.077
				Total	1.809-9.847	5.348
III	900-1,300 Avg 1,135	Heart Wet	2 11	Cold	2.147-4.533	3.489
				Hot	1.150-7.173	3.715
				B-a	0.791-4.608	1.877
				Total	6.021-11.209	9.081
IV	1,300 and up Avg 2,750	Wet	11	Cold	1.687-4.974	3.166
				Hot	0.932-3.787	2.368
				B-a	0.905-2.906	1.776
				Total	4.862-10.938	7.310

<sup>1</sup> Percentage of extractives based on oven-dry weight. Cold—cold water (23 C) extraction for 48 hr. Hot—hot water (70 C) extraction for 48 hr. B-a—Benzene-alcohol (1:2 mix) for 48 hours. Total—total extracted contents.

of polar substances and their degree of freedom. Ionic polarization also depends on the concentration and types of ions present in the solution. Increase in conductivity of wood because of increase in the number of ions also would contribute to a higher parallel capacitance. This number of ions in wood might be associated with the amount of water-soluble extractives present. Table 3 shows the extractive contents of four groups of specimens arranged according to increasing parallel capacitance. These specimens were those with moisture contents between 90 and 110%. Group I, with a capacitance range from 300 to 600 pF, contains 15 sapwood and 3 heartwood specimens and has an average total extractives content of 1.73%. Group II contains 2 sapwood, 12 heartwood, and 1 wetwood specimen and has an average total extractives content of 5.34%. Groups III and IV contain mostly wetwood specimens and have the highest extractives content of 9.08 and 7.3%, respectively. The extractives content of individual specimens overlaps somewhat from group to group, although the increase in capacitance and extractives content

from sapwood to heartwood to wetwood is evident from the data. Classification of hemlock wood into sapwood, heartwood, and wetwood can be difficult and was done here by visual observation, which might account for some of the variation in the types of samples found in each group.

#### CONCLUSIONS

Sinker heartwood of western hemlock has a higher specific gravity and initial moisture content than normal heartwood.

Drying of sinker heartwood is much slower than normal heartwood. Extraction with ethanol or acetone and steaming of sinker heartwood before drying improved the rate of drying or equalization to a given condition of EMC over matched control or untreated specimens of sinker heartwood.

Parallel capacitance of green western hemlock was highest in wetwood, somewhat lower in normal heartwood, and lowest in sapwood at a given moisture content. The range in capacitance was related closely to the total extractives content of specimens from the different zones that had similar moisture contents.

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