MACROSCOPIC AND MICROSCOPIC ANALYSES OF COLOR CHANGES OF WOOD PRESSURE STEAM-DRIED ABOVE ATMOSPHERIC PRESSURE

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ABSTRACT

Yellow poplar, silver maple, red oak, and southern pine heartwood samples were evaluated for color changes occurring as a result of pressure steam-drying above atmospheric pressure. Luminance significantly decreased, purity increased, and dominant wavelength shifted toward the red zone of the spectrum for yellow poplar and silver maple. Luminance significantly decreased, while other color parameters did not significantly change, for red oak wood. Southern pine wood decreased in luminance and increased in purity. Extractives of all woods appeared to have "softened" upon pressure steam-drying and became migratory, moving from the ray tissue system to the surface through the vessel system for the hardwood species. Pressure steam-dried white oak exhibited a drastically modified tyloses system; tyloses were missing from numerous heartwood vessels and those remaining appeared torn or ruptured.

Key words: Drying, steam, yellow poplar, silver maple, red oak, white oak, southern pine, extractives.

INTRODUCTION

Pressure steam-drying is a new method for rapidly seasoning lumber at pressures above 1 atmosphere in steam generated from the lumber itself (Rosen 1981; Rosen et al. 1982). Several species have been dried to 4 to 9% moisture content in 34 h or less at temperatures to 140 C and pressures to 2 atmospheres. Although the wood remained structurally sound, all woods studied so far have darkened throughout the wood to varying degrees after pressure steam-drying.

Darkening of lumber occurs normally on a thin layer on the surface, but sometimes throughout the wood, during high-temperature drying in a conventional kiln (McMillen and Wengert 1978). Schneider (1973) evaluated the discoloration of beechwood and pine sapwood, dried in a kiln from 110 to 180 C, by means of a spectrophotometer. He found that an increase in drying temperature as well as drying time caused a decrease in reflectance (increase in darkening) of the wood samples. Keith and Chang (1978) examined hygroscopic properties of hardwoods heat-treated and darkened at temperatures from 180 to 220 C and found that equilibrium moisture contents were lower and shrinkage was less with change in content than similar undarkened material.

Research was undertaken to characterize the color change in heartwood samples for yellow poplar, silver maple, red oak, and southern pine species that have been

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successfully pressure steam-dried. Subsequently, white oak, which developed excessive degrade when pressure steam-dried, was microscopically examined to determine possible causes for this degrade.

EXPERIMENTAL

Five wood species, yellow poplar (*Liriodendron tulipifera*), silver maple (*Acer saccharinum*), red oak (*Quercus rubra*), southern yellow pine (*Pinus* sp.), and white oak (*Quercus alba*) were obtained from trees found locally in southern Illinois. Bolts were sawn into 2.5-cm-thick boards and dried in a prototype pressure steam-dryer (Rosen 1981) to approximately 8% moisture content at 127 C temperature and 1.3 atmospheres pressure. Dried boards were planed, and heartwood samples 5 cm square by 2.5 cm thick were sanded for color analyses. Matched controls, consisting of conventional kiln-dried material for yellow poplar and silver maple and air-dried material for the other species, were prepared similarly. Air-dried rather than kiln-dried material was used for three of the species because kiln-dried samples from local trees could not be made available. All samples were then sent to the wood science laboratory of the School of Forestry, Fisheries, and Wildlife, University of Missouri, Columbia, for subsequent studies enumerated below.

Color analysis

The wood squares of yellow poplar, silver maple, red oak, and southern pine were analyzed using a Hitachi Perkin-Elmer Model 139 UV-VIS Spectrophotometer equipped with a diffuse reflectance apparatus to measure reflectance readings at 10-nanometer (nm) intervals over the visible wavelength range of 400–700 nm. Ten replicates per sample condition were measured for reflectance data. Barium sulfate was the color standard employed, and all reflectance readings were done at ambient temperature and relative humidity. The spectrophotometer was connected in-line to a digital voltmeter, which was connected to a data tape punch. Resultant reflectance data were used to compute luminance, dominant wavelength, and purity values for both pressure steam-dried samples and the controls. Statistical evaluation was then undertaken utilizing methods set forth in recent color studies of walnut (Phelps and McGinnes 1980). The white oak samples were not evaluated for color parameters.

Hot water extractives

To separate evaluations of the extractive contents of the samples were done: (1) the amount of water extractives was determined for steam-dried vs. control samples, and (2) the filtrates plus resultant residues were qualitatively compared for color luminance). Sample blocks used for the preceding color analysis (plus similar 5-cm^2 by 2.5-cm-thick white oak samples) were also used for extractive content studies. Samples obtained from these blocks were ground in a Wiley Mill, and the 40-mesh fraction was used for determination of hot-water extractives content. All determinations were done in triplicate. Samples were placed in extraction thimbles and reflexed in a Soxhlet apparatus for 72 h. Temperature in the extraction chamber averaged 85 C. Residues of pressure steam-dried vs. controls were visually compared for luminance on a qualitative basis only.

Microscopic analyses

All five species were microscopically examined to compare influences of the drying method on selected anatomical characteristics.

Radial and tangential sections were cut from portions of the same blocks used for color analyses. These sections, averaging 18 microns in thickness, were divided into two groups. The first group was stained with an $FeCl_3$ solution to enhance cell inclusions, while the second was left unstained for evaluation of cell-wall darkening due to pressure steam-drying. All sections were mounted in permount.

Extractives are usually concentrated in parenchymatic cells and vessel segments of hardwood species, whereas fibers in native hardwoods seldom contain noticeable inclusions in their lumens (Panshin and de Zeeuw 1980). Extractives in cell lumens of native coniferous species are usually most noticeable in parenchymatic cells of rays or resin canals. For any cell type selected, the following classification system was employed. The cell lumen was empty of inclusions (0%); the lumen was between empty and 50% filled with extractives; the lumen was greater than 50% but less than completely filled; or the cell lumen was completely (100%) filled with extractives. A counting grid with 100 squares was used to randomly select cells for evaluation. These cells were then classified into one of the four categories of lumen inclusion contents (0%, $<50\%$, $>50\%$ or 100%). The $FeCl_3$ stain was quite helpful in providing contrast between lumen inclusions and cell walls. Distributions were obtained from both radial and tangential sections for the hardwood species. Only complete cells with no torn wall structure were included in the assessment of cell lumen inclusion distributions. This work was done at magnifications of 35× to 250×, and the number of cells examined ranged from approximately 1,000 for yellow poplar to 3,000 for red oak. Distribution of the amounts of inclusions found in lumens of vessel segments and ray parenchyma cells were statistically evaluated based on chi-square tests of independence.

Preliminary studies of tangential and radial sections of the southern pine sample indicated that the low volume of ray tissue of this wood, particularly as shown on the radial face, prohibited comparison of inclusion distributions with the

TABLE 1. *Luminance, purity, and dominant wavelength values for pressure steam-dried heartwood of yellow poplar, silver maple, red oak and southern pine compared to samples which had been either kiln-dried or air-dried.*

Species	Drying method	Luminance (percent)	Dominant wavelength (nm)	Purity (percent)
Yellow poplar	Control[1]	56.23**	575.3*	19.40**
	Steam-dried	36.36**	578.3*	34.43**
Silver maple	Control[1]	65.25**	580.2*	17.42*
	Steam-dried	50.46**	582.8*	23.23*
Red oak	Control[1]	39.63**	584.7 n.s.	26.45 n.s.
	Steam-dried	29.15**	583.3 n.s.	24.93 n.s.
Southern pine	Control[1]	58.78**	580.0 n.s.	29.74*
	Steam-dried	49.19**	580.2 n.s.	32.82*

[1] Yellow poplar and silver maple were conventionally kiln-dried; red oak and southern pine were air-dried.
* Difference between drying methods for a given species significant at the 5% level.
** Difference between drying methods for a given species significant at the 1% level.
n.s. No significant difference between treatments (drying methods).

TABLE 2. *Percent hot water extractives content for pressure steam-dried heartwood of five commercial wood species compared to samples that had been either air-dried or kiln-dried.*

		Percent extractives
Silver maple	Pressure steam-dried	2.8 n.s.
	Control[1]	1.6 n.s.
Southern pine	Pressure steam-dried	4.1 n.s.
	Control[1]	2.9 n.s.
Yellow poplar	Pressure steam-dried	6.4 n.s.
	Control[1]	5.3 n.s.
Red oak	Pressure steam-dried	11.1*
	Control[1]	6.0*
White oak	Pressure steam-dried	11.7*
	Control[1]	16.4*

[1] Silver maple and yellow poplar were conventionally kiln-dried; other species were air-dried.
* Difference between drying methods significant at the 5% level.
n.s. No significant difference between drying methods for a particular species.

hardwood species; for this reason, quantitative assessment of cell inclusions for this sample was not undertaken.

The set of unstained slides were used to ascertain if darkening of pressure steam-dried wood was due not only to cell inclusions but perhaps also attributable to darkening of the cell wall. Radial and tangential sections were observed utilizing a Leitz MPV microspectophotometer system devised by McGinnes and Melcarek (1976) and later utilized by Phelps and McGinnes (1980). The technique consists of measuring transmittance of light through the cell wall (usually the S_2 layer) and expressing this as a percent of the light transmittance through an adjacent selected void or lumen region of the optical field. A variable measuring diaphragm allows transmittance through a selected portion of the optical field in comparison with an "empty" lumen. This work was done at a magnification of 950× using a 95× Leitz oil immersion objective, and at least 50 measurements per sample type were obtained and statistically evaluated by means of a *t*-test.

RESULTS AND DISCUSSION

Color analysis

Luminance, dominant wavelength, and percent purity data for yellow poplar, silver maple, red oak, and southern pine woods are presented in Table 1. Since these parameters describe color wood for a normal observer, brief definitions of each color characteristic are presented, as well as the magnitude of differences required between samples being compared for the eye to detect a color change. Luminance refers to the brightness of an object and is expressed as a percentage of a scale where 0% is black and 100% is white. Dominant wavelength is the principal hue (color) of an object and is expressed over the visible range from 400–700 nanometers. Purity, related to hue, is the percent of this principal hue in the total color of an object. These three terms have been in wide use for many years and serve to characterize the color of an object both quantitatively and objectively in a physical sense. Nelson et al. (1969) suggested that differences of 3.5% in luminance, 4 nanometers in dominant wavelength, and 5% in purity would be visually detectable to a normal observer evaluating black walnut. Using

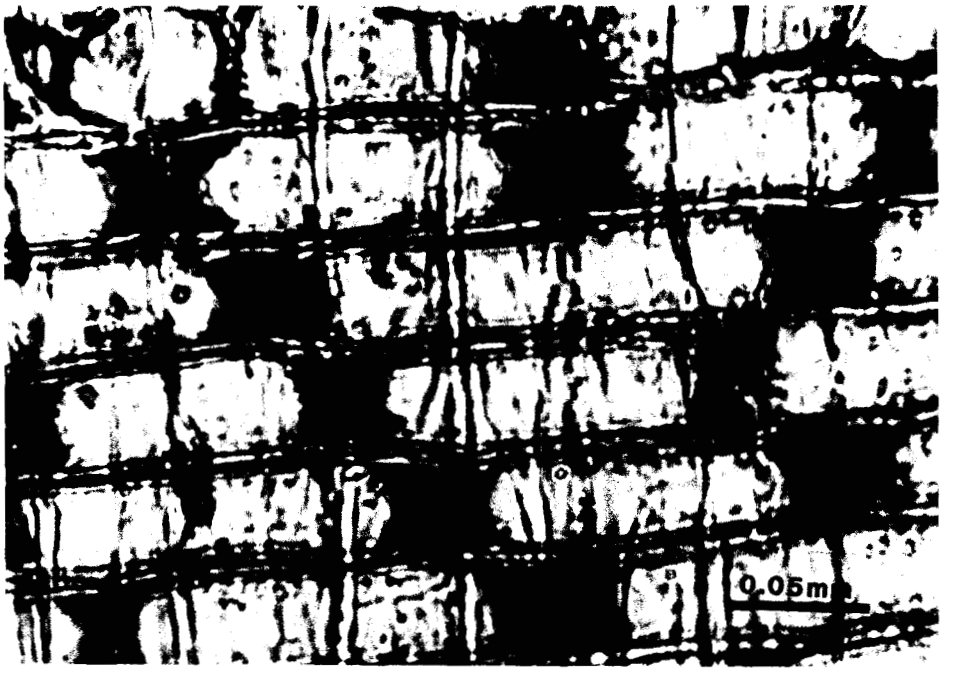

FIG. 1. Extractives in ray parenchyma of pressure steam-dried red oak. Note concentration of these inclusions at end-walls of ray cells and lack of globular masses of extractives throughout cell lumens. Compare to Fig. 2.

these criteria, it is obvious that all woods characterized in Table 1 darken noticeably upon pressure steam-drying as compared to the control samples. Dominant wavelength shifts toward the red end of the visible spectrum for all woods except red oak; however, these differences are not detectable to the unaided eye. Percent purity, like luminance, increases significantly for all woods except red oak, where a decrease in purity is noted. This increase in percent purity is visually detectable for yellow poplar and silver maple. These woods thus become more yellow and darker after steaming, whereas the others become noticeably darker without additional detectable changes in hue.

Hot water extractives

All species except white oak contained a higher water extractive content when pressure steam-dried; however, this increase is significant only for red oak (Table 2). White oak showed a significantly lower water extractive content when pressure steam-dried. Since this trend for white oak departed from that of the other four species, the extraction was repeated for both 72 and 92 h. In each instance the original results were verified; i.e., the control sample of white oak contained a significantly higher water extractive content than the pressure steam-dried white oak sample.

For all five species, the hot water extract from the pressure steam-dried samples appeared considerably darker to the unaided eye when compared to the extract from the control samples.

From the data shown in Table 2, plus the visual observation of a dark extractive

FIG. 2. Extractives in ray parenchyma of air-dried red oak. Compare location and form of these inclusions with sample of pressure steam-dried red oak in Fig. 1.

residue obtained from each wood pressure steam-dried, it is apparent that extractives are significantly influenced by pressure steam-drying and that this influence is species-dependent. Microscopic examination confirmed this evaluation.

Microscopic analysis

Microscopic examination of radial and tangential sections of woods that had been pressure steam-dried resulted in three significant findings when compared to sections of wood that had been either kiln-dried or air-dried. These three general observations were (1) the appearance or shape of the inclusions in cell lumina were modified; (2) the distribution of the amount of inclusions within cell lumina was changed; and (3) the transmittance of light through ray parenchyma and vessel segment cell walls was modified for hardwood species analyzed. In addition, the tyloses system of white oak heartwood was altered as a result of the pressure steam-drying treatment.

Figures 1 and 2 compare the appearance of shape of inclusions in ray parenchyma cell lumina for red oak that had been either pressure steam-dried or air-dried. Note that in the pressure steam-dried sample (Fig. 1) inclusions no longer appear as distinct globules rather randomly dispersed but rather as one unified mass concentrated at cell end-walls. This is in marked contrast to the more "normal" extractive appearance for the air-dried red oak ray tissue shown in Fig. 2. Figures 1 and 2 are typical portrayals of inclusion form and location for all the hardwood species studied as well as, to a lesser degree, the southern pine samples.

Table 3 presents the distribution based on four arbitrary classes of the amounts of inclusions found in the lumens of vessel segments or ray parenchyma cells of

TABLE 3. *The distribution of inclusions found in the lumens of vessel segments and ray parenchyma cells of pressure steam-dried heartwood of four species compared to samples that had been either kiln-dried or air-dried.*

Species	Drying method	Cell type: Vessel segment (percent of lumen filled)[1]				Ray parenchyma—in contact with vessels (percent of lumen filled)[1]				Ray parenchyma—no vessel contact (percent of lumen filled)[3]			
		0	<50	>50	100	0	<50	>50	100	0	<50	>50	100
Silver maple	Control[2]	6	88	6	0	3	38	47	12	0	48	51	1
	Pressure steam-dried	72	28	0	0	8	57	34	1	1	76	21	2
Yellow poplar	Control[2]	16	68	16	0	26	50	22	2	0	78	22	0
	Pressure steam-dried	73	27	0	0	43	26	22	9	0	91	9	0
Red oak	Control[2]	7	60	33	0	0	42	58	0	0	64	26	10
	Pressure steam-dried	93	7	0	0	4	35	59	2	0	63	36	1
White oak	Control[2]	—	—	—	—	91	3	3	3	76	10	4	10
	Pressure steam-dried	—	—	—	—	14	37	44	5	2	28	54	16

[1] Four arbitrary classes of inclusion distributions in cell lumens; values expressed on a percentage basis because of varying sample size (≈1,000 for yellow poplar up to ≈3,000 for red oak).

[2] Silver maple and yellow poplar were kiln-dried; oaks were air-dried.

[3] No significant difference between methods for distribution of inclusions within ray parenchyma cells not in vessel contact for yellow poplar; all other distributions in the table are significant at the 5% level. Significance based on a Chi-square test of independence.

the heartwood of woods pressure steam-dried as compared to those either kiln-dried or air-dried. These distributions are based on measurements made from radial sections only, since tangential sections of ray cells showed cell lumens to be either empty or 100% filled with inclusions for those samples that had been pressure steam-dried. This condition is predictable because of the nature of cell lumen inclusion distributions shown in Figs. 1 and 2. With one exception, ray parenchyma cells not in contact with vessels for yellow poplar samples, pressure steam-drying significantly modified the distribution of the amount of cell lumens filled with inclusions as compared to controls. This distribution shifts most noticeably for vessel segment contents where the "empty" lumen class increases drastically for pressure steam-dried samples of the three hardwoods examined. It was impractical to include a similar analysis for white oak because only a few intact white oak vessel segments were found in the pressure steam-dried sample. As indicated earlier, the tyloses system was ruptured upon pressure steam-drying and numerous vessel perforation plates were also destroyed, so that few complete vessel segments remained throughout the wood.

The percent transmittance data in Table 4 indicate a significant "darkening" of ray cell walls and vessel segment walls attributable to pressure steam-drying for red oak. Comparative results for yellow poplar and silver maple show no significance. Since the extractive content (Table 2) of pressure steam-dried red oak is considerably higher than that of either yellow poplar or silver maple—and the red oak is darkest (Table 1)—one might expect a higher and darker concentration of extractives to occur in red oak cell walls as compared to the other species after pressure steam-drying. Hillis (1971) has shown that a significant amount of extractives are located in cell walls as well as in cell lumens.

On the basis of microscopic evidence shown in Figs. 1 and 2, coupled with statistical data shown in Tables 3 and 4, it may be concluded that pressure steam-drying not only softens extractives but also is responsible for a migration of

TABLE 4. *Percent transmittance of white light through cell wall of indicated cell type for samples of yellow poplar, silver maple, and red oak heartwood that had been pressure steam-dried compared to samples either kiln-dried or air-dried.*

			Transmittance (percent)
Yellow poplar	Control[1]	Ray parenchyma	93 n.s.
		Vessel segment	93 n.s.
	Pressure steam-dried	Ray parenchyma	96 n.s.
		Vessel segment	96 n.s.
Silver maple	Control[1]	Ray parenchyma	95 n.s.
		Vessel segment	95 n.s.
	Pressure steam-dried	Ray parenchyma	97 n.s.
		Vessel segment	97 n.s.
Red oak	Control[1]	Ray parenchyma	96**
		Vessel segment	91*
	Pressure steam-dried	Ray parenchyma	80**
		Vessel segment	85*

[1] Yellow poplar and silver maple were kiln-dried; red oak was air-dried.
* Difference between drying methods for a given cell type within a species significant at the 5% level.
** Difference between drying methods for a given cell type within a species significant at the 1% level.
n.s. No significant difference between similar cell types within a species for drying method used.

extractives from parenchyma systems through vessel systems and out into the drying chamber from the wood surfaces for the woods evaluated. Certainly the extractive contents of the lumens of the vessel systems in the woods examined is reduced (Table 3). It is also apparent that species respond differently since significant cell-wall darkening occurs only for red oak, while both silver maple and yellow poplar exhibit "lighter" cell walls upon pressure steam-drying although this effect is not significant for these two species. Whether other species with extensive tyloses development in their heartwood would respond the same as white oak upon pressure steam-drying is not currently known.

CONCLUSIONS

From this initial study of color characteristics of several woods that have been pressure steam-dried, several observations may be made.

1. All wood species examined became significantly darker when pressure steam-dried as compared to those air- or kiln-dried.
2. Extractive contents of pressure steam-dried wood were greater for all species except white oak. This could be due to degradation of lower molecular weight hemicelluloses as well as increased solubility of the "extractive" component of the woods themselves.
3. Microscopic examination of residual extractives in various ray cells of pressure steam-dried woods indicated modification in form or texture of these extractives plus a preferential concentration at the ray cell end walls attributable to the drying process.
4. White oak samples that were pressure steam-dried had altered tyloses within the vessel system. Many tyloses were ruptured and several vessels were devoid of tyloses. Although the influence of the sectioning process for microscopic examination upon tyloses alteration is not currently known, con-

ditions of pressure steam-drying such as temperature and rate of moisture removal definitely influenced the tyloses conditions observed.

5. Since extractives were "softened" and migratory under conditions of pressure steam-drying, species with high extractive contents and certain anatomical features (tyloses, obstructed pit openings, etc.) should be studied more extensively for development of modified drying methods.

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