EFFECT OF STEAMING ON SOME PHYSICAL AND CHEMICAL PROPERTIES OF BLACK WALNUT HEARTWOOD[1]

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ABSTRACT

The influence of steaming time and temperature on some physical and chemical properties of black walnut heartwood was studied. One-inch cube sample blocks were steamed at two different temperatures and four different times, and the pH, surface tension, and color of the wood fluids, as well as the extractives and area of cell lumina, were determined.

The pH and surface tension were not affected enough by steaming to be related to color changes of wood, swelling the wood beyond that normally expected in water at room temperature, or reducing drying defects. Prolonged and high temperature (above 100 C) steaming increased alcohol-benzene extractives of the steamed wood. Prolonged and high temperature steaming caused cell walls to swell beyond that in water at room temperature, especially in earlywood. Steaming temperature and time were highly effective in changing the color of wood fluids.

Keywords: Hygroscopicity, permeability, transmittance, mechanical strength properties, dimensional stability, hydrolytic processes, *Juglans nigra*.

INTRODUCTION

Steaming walnut lumber, squares, and gunstock blanks before kiln-drying has been an important commercial process for decades. Black walnut has been steamed to darken sapwood color or to improve permeability, drying rate, and quality of the seasoned wood. Brauner and Conway (1964), in steaming walnut for color, found that higher temperature, greater moisture content, and longer steaming times darkened sapwood faster and more uniformly throughout. Chen (1975), in steaming walnut for permeability improvement, found that higher temperature and longer steaming times increased longitudinal permeability in heartwood but failed to alter the already extremely permeable sapwood. Torgeson and Smith (1942), in steaming walnut gunstock blanks for kiln-drying, found that steaming in 212 F for 3 days increased the initial drying rate of the steamed stock considerably; but as drying progressed and higher temperatures were used, the advantages became less. Steaming also generally improves the color of the heartwood. For decades, buyers have demanded steamed walnut lumber because the

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lessened contrast between heartwood and sapwood in the steamed material facilitates a more uniform finishing (USDA Forest Products Laboratory 1961).

No information is available concerning the effect of steaming time and temperature on color of wood fluids, cell-wall thickness, pH, surface tension, and amount of extractives in black walnut heartwood. These parameters were believed to influence the mechanical strength properties, drying characteristics, and dimensional stability of black walnut wood. The pH of wood was reported to be a decisive factor in changing wood color and, therefore, its aesthetic value (Kai 1975), as well as controlling the degree of wood swelling and, therefore, its mechanical strength properties (Stamm 1964). Troughton and Rozon (1974) showed that saturated steam heating was most detrimental to the mechanical strength properties of two softwoods when compared to hot-press, oven, and oil bath heating. High surface tension of free water in green wood was believed to contribute to drying defects, such as collapse (Cech 1968; Ellwood et al. 1960). The extractives in black walnut wood were reported by Cooper (1974) to be more hygroscopic than cell walls. The water bound by the extractives was adsorbed-compressed more than that bound by the cell walls, resulting in a higher moisture content and less swelling at a given relative vapor pressure in black walnut with extractives than in the same wood without extractives.

The purpose of this study was to determine qualitatively and quantitatively the influence of steaming time and temperature on some physical and chemical properties of walnut heartwood, which in turn might influence the final wood color, mechanical strength properties, alteration in drying characteristics, and dimensional stability of walnut heartwood.

MATERIALS AND METHODS

All the sample blocks were prepared from a single black walnut tree. The bolts were broken down into 1¼-inch-thick flitches by a bolter saw. Approximately 4-inch-wide heartwood boards were cut from each flitch. After the boards were planed to 1-inch thickness, they were ripped and crosscut into 1-inch cubes, sealed in plastic bags, and stored in a cooler at 40 F until time of use. Only heartwood was employed in this study. Sapwood has already been reported to be extremely permeable (Chen 1975) and very easy to dry; thus there is no need to steam it for drying improvement. As for steaming sapwood for color, Brauner and Conway (1964) reported it comprehensively.

For testing purposes, 324 cubes were randomly assigned to 9 treatments with 36 cubes per treatment. One of the treatments was a green control, while the other eight treatments were subjected to different steaming conditions. Two different temperatures, 100 C and 120 C, and four steaming times, 2, 4, 8, and 16 h, were employed in this study.

Steamings were carried out in a vertical sterilizer that could be adjusted to produce steam at temperatures between 100 C and 130 C.

Twenty of the steamed cubes were squeezed at high pressure to extract the fluids from wood. Kubinsky and Ifju (1973a) reported that using press-extracted fluids from wood samples of high MC was a simpler, faster, and more accurate method to determine pH of wood than conventional methods. Each cube was squeezed at a pressure of 10 tons per square inch for 0.6 min. Then the press-extracted wood fluids were filtered through a fiberglass filter and set aside to cool

to room temperature. Approximately 40 ml of wood fluids were extracted from 20 cubes of the sample material. These fluids were then tested for pH, surface tension, and color; pH was determined with an Instrumentation Laboratory Model 165 pH meter[2], surface tension with a Cenco-Dunouy Tensiometer[2], and color with a Bausch & Lomb Spectronic 20 Spectrophotometer[2]. For the color measurement, the fluids were diluted to a 10% solution and were tested for degree of transmittance with red light at 650 mμ wave length. Effect of prolonged heating of fluids press-extracted from green heartwood of black walnut was also studied by repeated boiling and condensing of the fluids and measuring their degree of transmittance with red light.

Cell lumina changes after steaming were measured by means of an incident light microscope and a micrometer. Immediately after steaming, four cubes from each treatment were randomly picked for splitting and microtoming. Each cube was split radially to the growth rings and parallel with the grain. A microtome was then used to cut a smooth surface across the end grain of the splinters ($^3/_{16}$ inch by $^3/_{16}$ inch). The splinters were mounted on a microscope slide with the sliced end grain-up for viewing. The splinters were viewed under a Zeiss photomicroscope II[2] at a magnification of 640:1. A point sampling method utilizing a 100-cross grid micrometer was used for determining the percent of cell lumina (Barrett and Philbrook 1970). From the prepared splinters, eight growth rings were randomly chosen for sampling. Each growth ring was further divided into two areas—earlywood and latewood—under the microscope. One count was taken from each area giving a total of eight counts to determine the area percent of cell lumina, per unit area (earlywood or latewood), per treatment.

For determining moisture content (MC) of the cubes after each treatment, a standard method was used. Three cubes were randomly picked from the steamed sample, weighed, oven-dried at 103 ± 2 C for 24 h, and reweighed again to determine the weight of the water in the wood.

The remaining nine cubes were used for hot water and alcohol-benzene (A-B) extractions. The cubes were placed in an environmental chamber to condition down to 6% MC. After drying, these cubes were ground in a Wiley mill[2] and sifted for a 40/60 mesh sawdust sample. Hot water and A-B extractions were carried out according to TAPPI Standards No. T1m-59 and No. T6m-69.

RESULTS AND DISCUSSION

An attempt was made to understand the influence of steaming on wood color by studying the influence of steaming on the color of press-extracted wood fluids, the extractives in the wood fluids being the main pigments of wood color. Steaming sample blocks up to 2 hours at both 100 C and 120 C significantly reduced the percent transmittance of red light (650 mμ) through press-extracted wood fluids, but as steaming times were increased so was the percent transmittance compared to the percent transmittance of red light through fluids press-extracted from non-steamed black walnut heartwood (Fig. 1).

Kubinsky and Ifju (1973b) observed a similar phenomena in steaming red oak.

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[2] The use of trade or corporation names in this publication does not constitute an official endorsement by the U.S. Department of Agriculture.

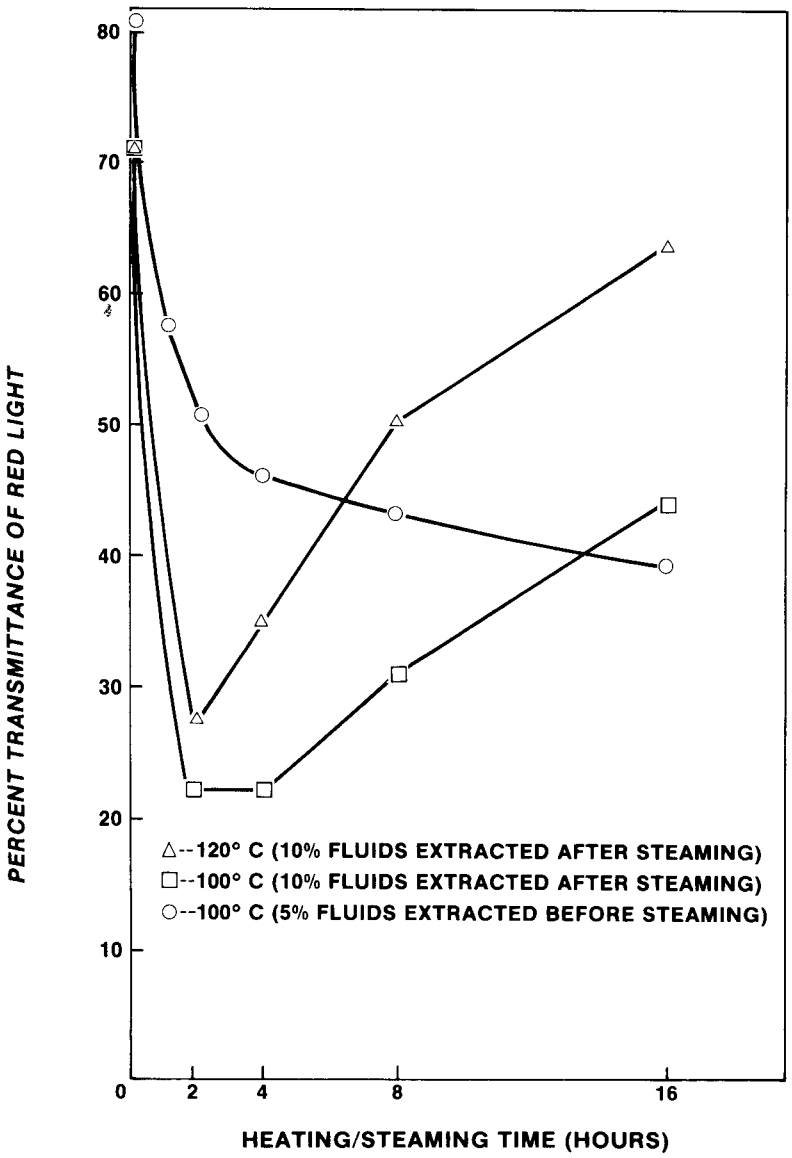

FIG. 1. Effect of steaming or heating on color of wood fluids.

They found after 1½ hours of steaming that there was a slight darkening of the orange-brown fluid, which became a lighter orange color after 3 h of steaming. Further steaming of wood to 24 and 48 h resulted in a colorless liquid extract with a very small amount of light brown precipitate.

When the press-extracted fluids from green wood were heated by boiling and condensing these fluids, longer heating time produced darker fluids (Fig. 1). The pattern of changes in color of black walnut heartwood due to steam treatments was observed to be more similar to prolonged heating of press-extracted fluids

TABLE 1. *Effect of steaming on earlywood lumen.*

A. Area of earlywood lumen (%).[a]

Green controls	100 C				120 C				F
	2 h	4 h	8 h	16 h	2 h	4 h	8 h	16 h	
39.13	34.75	32.88	32.38	31.88	30.13	36.63	28.00	31.63	4.00**

[a] Each number is an average of eight determinations.
** Significant at 1% level.

B. Newman-Keul's test.

28.00	30.13	31.63	31.88	32.38	32.88	34.75	36.63	39.13

C. Analysis of variance.

Source of variation	D.F.	F
Total	63	—
Temperature	1	1.35NS
Time	3	2.57NS
Temperature × time	3	2.81*
Error	56	—

** Significant at 1% level.
* Significant at 5% level.
NS—Nonsignificant.

from green black walnut wood; i.e., longer steaming time and higher steaming temperature produced a darker wood color. Similarly, Brauner and Conway (1964) found that steam treatments changed walnut heartwood from a purple cast to a chocolate brown color.

The discrepancy in color change between press-extracted fluids from steamed black walnut wood (longer steaming produced lighter color) and steamed black walnut wood itself (longer steaming produced darker color) was most probably due to the fact that steam-induced precipitate (Kubinsky and Ifju 1973b) became harder to squeeze out of the cell lumina.

The one-way F-test among the nine treatments showed that steaming significantly reduced (at the 1% level) the area of cell lumina of earlywood—that is, increased cell-wall thickness of earlywood (Table 1-A). Newman-Keul's test further revealed that 2, 8, and 16 h of steaming at 120 C, and 16 h of steaming at 100 C significantly reduced the area of cell lumina of earlywood (Table 1-B).

The analysis of variance excluding controls revealed that the steaming temperature-time combination affected the area of earlywood cell lumina (Table 1-C). Therefore, in studying the effect of steaming on earlywood cell lumina of black walnut heartwood, one should consider the interaction of steaming temperature and time, not only their individual effect alone.

The effect of steaming on cell walls of latewood was different from that of earlywood. Steaming failed to alter the area of cell lumina or cell-wall thickness of latewood (Table 2-A). Although no difference among the four steaming times was found, a significant difference was found between the two temperatures (Table 2-B). Earlywood is easier to alter by steam treatments than latewood, since

TABLE 2. *Effect of steaming on latewood lumen*

A. Area of latewood lumen (%).[a]

Green controls	100 C				120 C				F
	2 h	4 h	8 h	16 h	2 h	4 h	8 h	16 h	
39.25	38.00	38.75	38.88	39.38	34.63	35.63	37.00	32.13	2.00NS

[a] Each number is an average of eight determinations.
NS—Nonsignificant.

B. Analysis of variance.

Source of variation	D.F.	F
Total	63	—
Temperature	1	9.87**
Time	3	<1NS
Temperature × time	3	<1NS
Error	56	—

** Significant at 1% level.
* Significant at 5% level.
NS—Nonsignificant.

the thicker cell walls of latewood provided a greater resistance to changes from steaming.

Permanent reduction in the area of cell lumina or swelling in cell walls reduces the mechanical strength properties of walnut heartwood subjected to higher temperature and longer steaming, as reported by Troughton and Rozon (1974) in steaming two softwoods with saturated steam.

The one-way F-test among the nine treatments showed that steaming changed pH of black walnut heartwood (Table 3-A). Newman-Keul's test further revealed, however, that only the 16-h steaming at 120 C significantly reduced pH of black walnut heartwood compared to that of the nonsteamed controls (Table 3-B).

The analysis of variance (excluding controls) showed that the temperature-time combination affected pH value of steamed walnut heartwood (Table 3-C). The rate of decrease in pH value due to steaming was found to be greater at 120 C than at 100 C. Similarly, Kubinsky and Ifju (1973b) found, in steaming red oak, that pH of press-extracted wood fluids was reduced from 3.6 for nontreated red oak to 2.9 for samples that received 96 h of steaming at 100 C.

Considering the magnitude and direction of change in pH of black walnut caused by steaming, it is reasonable to state that pH altered by steaming in this study did not contribute to cell-wall swelling and darkening of the wood. Stamm (1964) reported that the dilute acidic solutions in the pH range of 2 to 6 showed little or no swelling action on wood beyond the swelling in water. Dilute alkaline solutions, on the other hand, caused swelling beyond the swelling in water, which became appreciable above a pH of 8. Kai (1975) reported that the reddish heartwood of Sugi (*Cryptomeria japonica*) could be darkened by soaking the wood in an alkaline solution of pH 12 for 24 h. However, the darkened wood would return to its original reddish color if neutralized with an acidic solution and rinsed with plenty of water. This was attributed to the fact that phenols, the most important pigment substances in wood, were stable in a neutral and acidic solution but

TABLE 3. *Effect of steaming on pH of wood fluids.*

A. pH values.[a]

Green controls	100 C				120 C				F
	2 h	4 h	8 h	16 h	2 h	4 h	8 h	16 h	
4.25	4.43	4.34	4.40	4.30	4.35	4.24	4.08	3.90	7.87**

[a] Each number is an average of two replications.
** Significant at 1% level.

B. Newman-Keul's test.

3.90	4.08	4.24	4.25	4.30	4.34	4.35	4.40	4.43

C. Analysis of variance.

Source of variation	D.F.	F
Total	15	—
Temperature	1	49.0**
Time	3	14.0**
Measure	1	4.7NS
Temperature × time	3	7.0*
Error	7	—

** Significant at 1% level.
* Significant at 5% level.
NS—Nonsignificant.

ionized to phenoxide anions in an alkaline solution. The ionized phenols, in turn, changed their spectra of absorption and gave the darkening effect to wood.

The one-way F-test among the nine treatments showed that steaming did not alter the surface tension of wood fluids (Table 4-A).

The analysis of variance (excluding controls) also showed that there were no differences between two steaming temperatures and among the four steaming times in altering the surface tension of black walnut heartwood fluids. The significant difference in the measures (at 1% level) was most probably due to the higher moisture content of replication 1 (65.9%) compared to replication 2 (57.6%), since pure water has a higher surface tension (73 dynes/cm at 20 C) than that of press-extracted black walnut heartwood fluids (Table 4-B).

In order to have less collapse, surface tension of wood fluids must be lowered before drying. Cech (1968) showed that extreme checking and collapse in red oak and yellow birch were eliminated when the water in the specimens was replaced by a 50 to 70% alcohol solution having a lower surface tension (surface tension of pure alcohol is 22.8 dynes/cm at 20 C). Ellwood et al. (1960) also reported that by replacing the moisture in collapse-susceptible hardwood blocks by low surface tension liquids such as methanol and ethanol, they were able to dry some of the blocks defect-free under severe drying conditions, whereas untreated green blocks collapsed severely and were entirely ruined.

In this study steaming did not lower the surface tension of black walnut wood fluids; therefore, no reduction in drying defects could be attributed to changes in surface tension of walnut wood fluids due to steaming.

TABLE 4. *Effect of steaming on surface tension of wood fluids.*

A. Surface tension (Dynes/cm).[a]

Green controls	100 C				120 C				F
	2 h	4 h	8 h	16 h	2 h	4 h	8 h	16 h	
51.1	52.5	53.4	53.5	53.1	53.1	53.0	53.7	55.8	0.22NS

[a] Each number is an average of two replications.
NS—Nonsignificant.

B. Analysis of variance.

Source of variation	D.F.	F
Total	15	—
Temperature	1	1.53NS
Time	3	1.39NS
Measure	1	74.21**
Temperature × time	3	1.25NS
Error	7	—

** Significant at 1% level.
NS—Nonsignificant.

The one-way F-test among the nine treatments showed that steaming sample blocks affected their availability of hot water extractives (Table 5-A). Newman-Keul's test further revealed that only the 8-h steaming at 120 C significantly reduced the hot water extractives of steamed black walnut heartwood compared to that of the nonsteamed controls (Table 5-B).

TABLE 5. *Effect of steaming on hot water extractives.*

A. Hot water extractive contents (%).[a]

Green controls	100 C				120 C				F
	2 h	4 h	8 h	16 h	2 h	4 h	8 h	16 h	
9.32	8.80	8.58	8.85	8.94	8.72	9.26	8.47	9.35	3.83**

[a] Each number is an average of three determinations.
** Significant at 1% level.

B. Newman-Keul's test.

8.47	8.58	8.73	8.80	8.85	8.94	9.26	9.32	9.35

C. Analysis of variance.

Source of variation	D.F.	F
Total	23	—
Temperature	1	1.63NS
Time	3	3.03NS
Temperature × time	3	3.83*
Error	16	—

* Significant at 5% level.
NS—Nonsignificant.

TABLE 6. *Effect of steaming on alcohol-benzene extractives.*

A. Alcohol-benzene extractives (%).[a]

Green controls	100 C				120 C				F
	2 h	4 h	8 h	16 h	2 h	4 h	8 h	16 h	
9.20	9.76	9.54	9.21	9.94	9.49	9.96	9.52	10.39	15.08**

[a] Each number is an average of three determinations.
** Significant at 1% level.

B. Newman-Keul's test.

9.20	9.21	9.49	9.52	9.54	9.76	9.94	9.96	10.39

C. Analysis of variance.

Source of variation	D.F.	F
Total	23	—
Temperature	1	11.80**
Time	3	25.48**
Temperature × time	3	6.55**
Error	16	—

** Significant at 1% level.

The analysis of variance (excluding controls) revealed that temperature-time combination affected the availability of hot water extractives of steamed walnut heartwood (Table 5-C).

Some water-soluble extractives were leached out during steam treatments because the clear distilled water used to provide steam in the sterilizer turned a dark brown color at the end of each steam treatment. Thus, some reduction in hot water extractives was evident among these samples that received steam treatments. The fact that the more severe steaming conditions at 120 C on the average caused less reduction in hot water extractives was probably because the steaming also improved the permeability of black walnut (Chen 1975). The increased accessibility of the hot water to more areas resulted in more extraction than the lower temperature steaming treatments. Kubinsky and Ifju (1973b) proposed that the extractives removed during steam treatments might be replenished partly by compounds resulting from hydrolytic processes associated with steaming, and enhanced by acidity generated simultaneously.

The one-way F-test among the nine treatments showed that steaming sample blocks of black walnut heartwood affected their availability of alcohol-benzene (A-B) extractives (Table 6-A). Newman-Keul's test further showed that steaming for 2 and 16 h at 100 C and steaming for 4 and 16 h at 120 C all significantly increased the amount of A-B extractives compared to that of the nonsteamed controls (Table 6-B). As explained in the previous section, steaming increased wood permeability which, in turn, increased the accessibility of A-B solvent to more areas to remove extractives. The hydrolytic processes associated with steaming, enhanced by higher acidity generated simultaneously, also added to the A-B extractive contents.

The analysis of variance (excluding controls) showed that the steaming temperature-time combination significantly affected the availability of alcohol-benzene (A-B) extractives of steamed walnut heartwood (Table 6-C). The rate of increase in A-B extractives due to steaming was also found to be greater at 120 C than at 100 C.

In view of the small reduction in hot water extractives and slight increase in A-B extractives due to steaming, it was felt that steaming treatment did not alter the hygroscopicity of walnut heartwood significantly.

CONCLUSIONS

The pH and surface tension of wood fluids were not affected enough by steaming to contribute to the changes in wood color, cell-wall swelling beyond swelling in water at room temperature, and reduction of drying defects.

It is more accurate to determine the effect of steaming on wood color by determining the effect of steaming on the color of fluids extracted from green wood rather than on the color of fluids extracted from wood after steaming treatments.

Short-time and low-temperature steaming reduced hot water extractives, but prolonged and high temperature (above 100 C) steaming increased alcohol-benzene extractives of the steamed walnut heartwood.

Prolonged and high temperature (above 100 C) steaming caused cell walls to swell beyond that in water at room temperature, but more so in earlywood than in latewood.

The study revealed that steaming temperature and time interacted. Therefore, a single factor experiment (steaming temperature or time alone) would possibly lead to erroneous conclusions.

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