

DETERMINING THE EFFECT OF EXTRACTIVES ON MOISTURE MOVEMENT USING A “CONTINUOUS” MEASURING SYSTEM¹

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ABSTRACT

Red oak (*Quercus* sp.) heartwood samples were treated by (1) steaming at atmospheric pressure for 6 h; (2) hot-water soaking at 70 C for 24 h; and (3) ethyl alcohol extraction in a Soxhlet extractor for 24 h after hot-water soaking. All samples were soaked in water and treated by vacuum-atmosphere method. Each sample was then coated with waterproof resin to ensure unidirectional radial movement of moisture. An environmental box equipped with an electric fan was designed and built for the purpose of achieving “continuous” measuring. Potassium chloride salt was used to control the relative humidity to 82% at 40 C. The sample was hung on a digital balance, which was connected to a printer, and the weight loss was recorded in 5-min intervals by a computer-controlled data acquisition system. Experimental results indicated that all treated samples, as compared with the untreated samples, increased the diffusion coefficients and therefore the drying rate significantly. The differences among treatments were not statistically significant. The major effect of the extractives came from the water-soluble components. The removal of extractives increased the drying rate in the falling rate period.

Keywords: Extractives, red oak, diffusion, moisture movement, falling rate period, drying rate, steaming, hot-water soaking.

INTRODUCTION

Wood extractives are natural products extraneous to a lignocellulose cell wall. They are a mixture of different chemical components with a wide range in molecular weight and water-solubility (Rowe and Conner 1979). The literature (Nearn 1955; Spalt 1979; Choong and Achmadi 1991) reported that wood extractives have significant effects on some wood properties such as equilibrium moisture con-

tent and dimensional stability. Theoretically, wood extractives also affect transport properties (Choong and Achmadi 1989), since they may block not only the macrocapillary passageways such as cell lumens and pits, but also the microcapillary passageways within the cell wall. However, no published data are available to describe how and to what extent wood extractives affect moisture transport during drying. The reasons may be due to several technical problems. First, the amount of wood extractives and their composition vary greatly among different wood species and also within different parts of the same tree (Rowe and Conner 1979). Therefore, the true effect of wood extractives on moisture transport might be

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masked by the great variation of extractive content and composition among samples. Second, the traditional manual-weighing procedure introduces an experimental error because of an indeterminate change of temperature and moisture when a sample is taken out of an environmental chamber and weighed on a balance. Thus, these errors must be minimized in order to reveal the true effect of extractives during drying.

This study was undertaken to determine the effect of extractives on moisture movement. The diffusion coefficient, as an index of drying rate, was calculated from the solution of Fick's second law of diffusion. To overcome the aforementioned difficulties, (1) a randomized complete block design with four replications was employed to control the variation among samples; and (2) an instrumentation method was applied to collect drying data at 5-min intervals by a computer-controlled data acquisition system in order to eliminate errors of a manual-weighing procedure. In addition, we examined the variation of drying rates with drying time and moisture content (MC).

EXPERIMENTAL PROCEDURE

A flatsawn board of red oak (*Quercus* sp.) heartwood was obtained from the green chain of a local mill. Four experimental block locations were determined along the longitudinal direction. From each location, a 60-cm strip was cut, with a cross section 2 cm in the tangential direction and 1.5 cm in the radial direction. The wood strips were planed to 1.5 cm in the tangential direction and 0.8 cm in the radial direction. Four samples 1.5 cm long were obtained from each strip successively, and they were marked to distinguish them from the samples of other strips. Within each block, the four samples were assigned randomly to four treatments as described below. Altogether, there were 16 samples.

The four treatments were: (1) untreated control; (2) steaming at atmospheric pressure for 6 h; (3) hot-water soaking at 70 ± 2 C for 24 h; and (4) ethyl alcohol extraction in a Soxhlet extractor for 24 h after hot-water soaking. The

hot-water soaking was accomplished in a beaker that was wrapped with an insulation material. A stirring hot plate was used to heat the water inside the beaker. The water was stirred continuously to keep the water temperature evenly distributed. A thermostat was employed to control the water temperature to 70 ± 2 C. The sample was suspended in the water by a copper wire attached to a plastic plate, which served as the cover of the beaker. Steaming treatment was accomplished in a flask. An electric stove was used to boil the water inside the flask. A sample was introduced into the flask through the neck and hung on the rubber plug. There were two small holes on the rubber plug, so the inside of the flask was always kept at atmospheric pressure. For the ethyl alcohol extraction, the samples were placed inside a Soxhlet extractor immediately after they had been soaked for 6 h in hot water at 70 ± 2 C. After treatment, the treated and control samples were soaked in water inside a vacuum desiccator for 7 days; twice each day a vacuum (559 mm Hg) was applied for about 15 min. At the end of this process, each sample was coated three times with a waterproof polymer substance (Dow's Saran F-120 resin) on the cross-sectional and radial surfaces to ensure that moisture loss was limited to the radial direction.

An environmental box ($18 \times 20 \times 22$ cm 3) equipped with an electric fan was designed and built for the purpose of achieving "continuous" measurement. The schematic diagram of the measuring system is shown in Fig. 1. The temperature in the box could be adjusted from room temperature to about 100 C. The air speed inside the box was about 1.3 meters per second, which was measured near the bottom of the sample with a Hastings air meter. Potassium chloride (KCl) salt was used to control the relative humidity (RH) to about $82 \pm 2\%$ at 40 ± 0.5 C. A Weathertronics RH and temperature indicator in conjunction with a RH and temperature probe was used to monitor the variation of RH and temperature continuously. After the RH and temperature in the environmental box became stabilized, a single

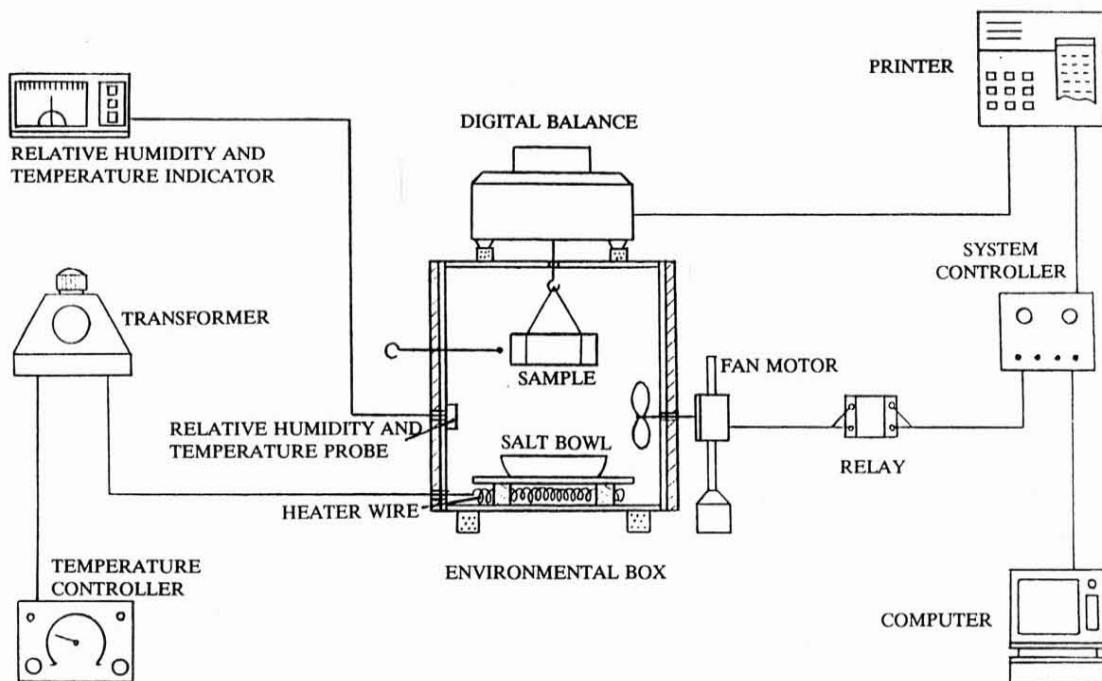


FIG. 1. Schematic diagram of measuring system.

sample was blotted with a paper towel to eliminate the surface water before it was introduced into the box. The sample was hung on a Sartorius digital balance, which was connected to a printer. Its weight loss was recorded periodically at 5-min intervals (the air flow was stopped for 10 sec before the sample was weighed). The data acquisition and control of the fan were regulated by a computer. After equilibrium was attained, the samples were oven-dried at 103 ± 2 C, and the moisture content (MC) at each data point was calculated.

The average diffusion coefficient D (cm^2/sec) was calculated according to the equation derived by Boltzmann (Stamm 1964):

$$D = \frac{\pi a^2 (1 - \bar{E})^2}{4t} \quad (1)$$

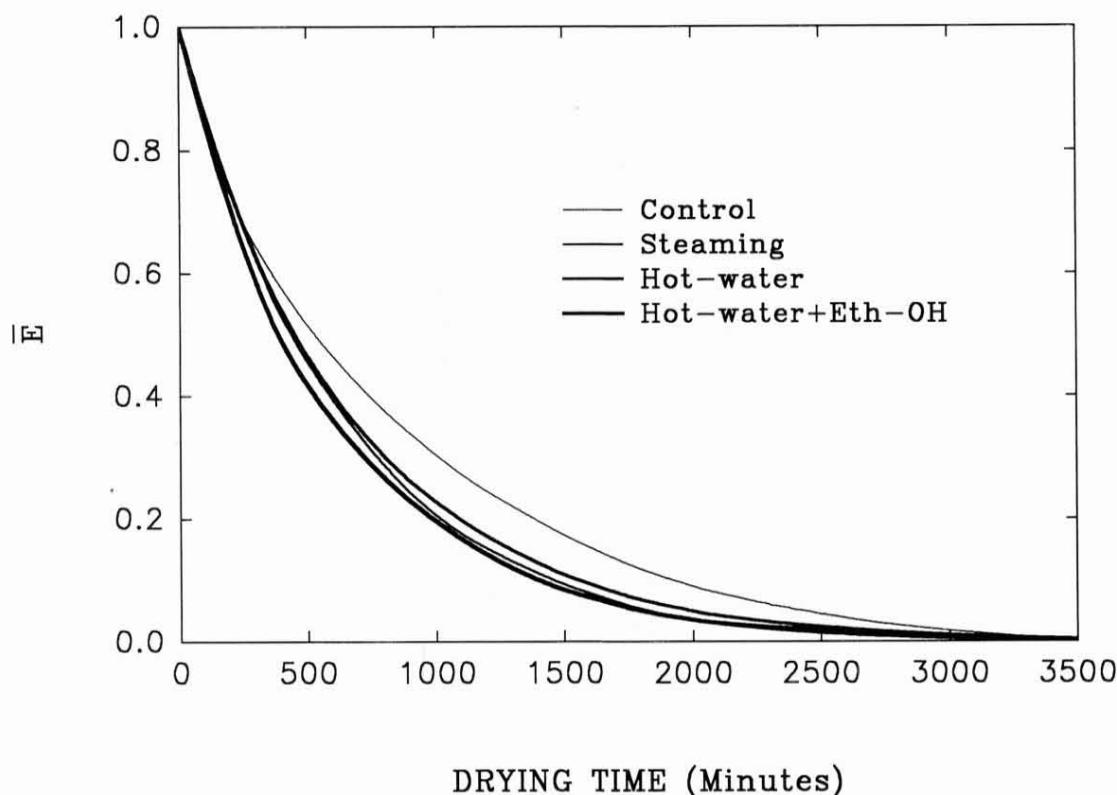
where a is the half-thickness of the sample (cm); \bar{E} , the fraction of evaporable water remaining in the wood, is defined as $(M - M_e)/(M_i - M_e)$, where M is the average MC in wood at

any drying time t ; M_e is the equilibrium moisture content (EMC); and M_i is the initial MC. In this study, the average diffusion coefficient was calculated at the half-drying time, that is, when \bar{E} is equal to 0.5, as suggested by Crank (1975) and used by Choong and Skaar (1979). In this study, the diffusion coefficient was considered a working parameter over the entire MC range in accordance with Stamm's (1964) hypothesis that the moisture movement is diffusion-controlled even in capillary movement above the fiber saturation point.

The drying rate F ($\text{g}/\text{cm}^2 \cdot \text{sec}$) at the drying time t was derived according to the central difference equation (Borse 1991) given as follows:

$$F = \frac{W_{i+1} - W_{i-1}}{2A\Delta t} \quad (2)$$

where W_{i-1} and W_{i+1} are the sample weights (g) at the previous time point t_{i-1} and next time point t_{i+1} , respectively; A is the exposed surface area (green basis) of the drying sample

FIG. 2. \bar{E} values as a function of drying time.

(cm^2); and the Δt is the time interval $t_{i+1} - t_{i-1}$, which was 300 seconds (5 min) in this study.

RESULTS AND DISCUSSION

The \bar{E} values as a function of drying time, shown in Fig. 2, indicate that they decreased faster in the treated samples than in the untreated samples when dried from about 100 to

about 15% MC. The average diffusion coefficients at $\bar{E} = 0.5$ are given in Table 1. An analysis of variance for the diffusion coefficients (Table 2) shows that treatments had a significant effect ($P < 0.05$) on moisture movement during drying. The large block effect ($F = 12.53$) suggested that the wood in the same board at different locations was not homogeneous. The relative efficiency (RE) of the block design relative to the completely random design is 3.2, based on Fisher's procedure (Steel and Torrie 1980); therefore, it was necessary to apply the block technique to screen out the

TABLE 1. Average diffusion coefficients of red oak among four treatments.

Block	Control	Steaming	Hot-water	Hot-water + Eth-OH
$\times 10^{-6} \text{ cm}^2/\text{sec}$				
I	1.85	4.09	4.01	5.76
II	0.97	1.40	1.41	1.68
III	1.55	2.27	2.19	2.56
IV	1.51	2.32	2.33	2.63
Mean*	1.47 B	2.52 A	2.48 A	3.15 A

* Means within columns having common letters are not significantly different at the 0.05 significance level by the Duncan test procedure.

TABLE 2. Analysis of variance of diffusion coefficients of red oak.

Source	df	MS	F	P
Treatment	3	1.947	5.21	0.023
Block	3	4.682	12.53	0.002
Error	9	0.374	—	—

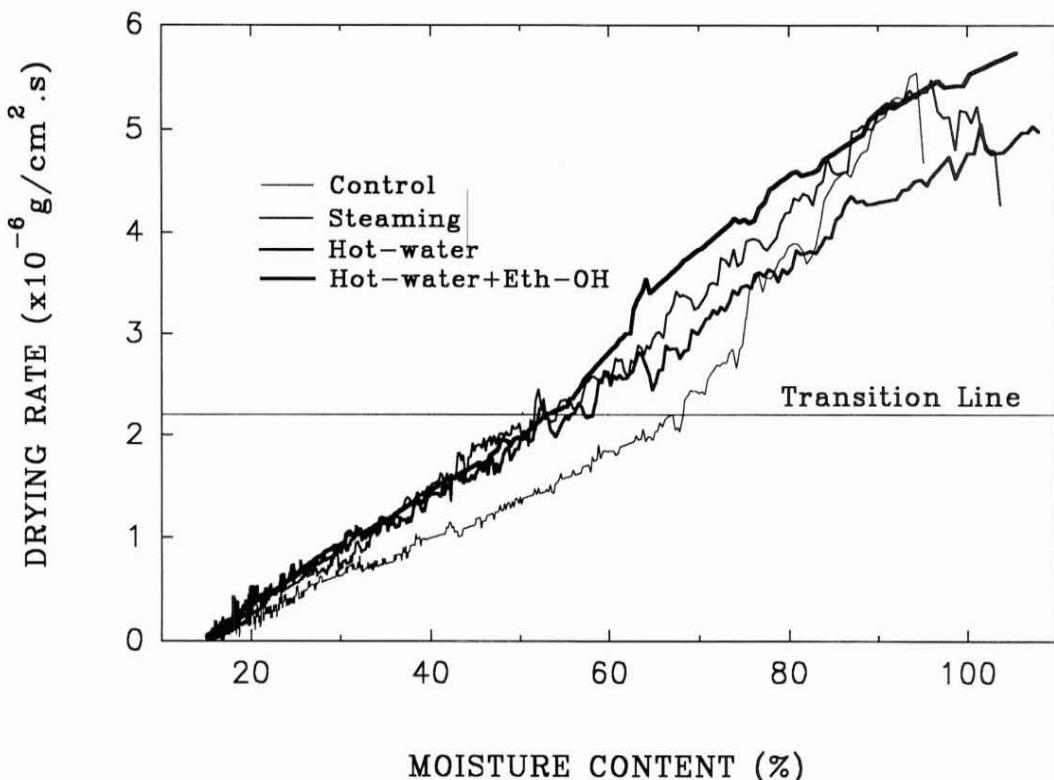


FIG. 3. Drying rate as a function of moisture content.

variations due to location. Further analysis based on the Duncan test indicated that all treatments to remove extractives increased the diffusion coefficients, but there was no statistical difference among the treatments at the 0.05 significance level (Table 1). The results indicate that the main effect came from the water-soluble extractive components, since further treatment with ethyl alcohol could not significantly increase the diffusion coefficient.

The drying of wood theoretically is separated into three stages: (1) constant rate period, (2) first falling rate period, and (3) second (exponential) falling rate period (Kollmann and Côté 1968; Hildebrand 1970). The recorded curves from above the fiber saturation point (FSP) to below the FSP did not show any sharp transition over the entire drying period. The constant rate period occurs when the rate of moisture moving from the inside to the surface of wood is higher than the rate of water evaporating at the surface. The second falling rate

period is formed when the surface MC is already below the FSP. The first falling rate period is a transitional period, when various drying processes are partially superimposed (Hildebrand 1970). The drying rates for various treatments as a function of MC (Fig. 3) show no constant drying rate period, but only a falling rate period (which is the combined first and second falling rate periods). This phenomenon is expected for transverse movement of moisture in hardwoods because the cell-wall passageways are practically impermeable (Palin and Petty 1981). Siau (1984) stated that the openings on the pit membranes of hardwoods are approximately one order of magnitude smaller than the values for softwoods. As a result, the resistance to moisture movement is large. The surface MC could drop below the FSP at the beginning of drying; therefore a constant rate period, if any, could exist for only a short period of time.

The treatments to remove extractives led to

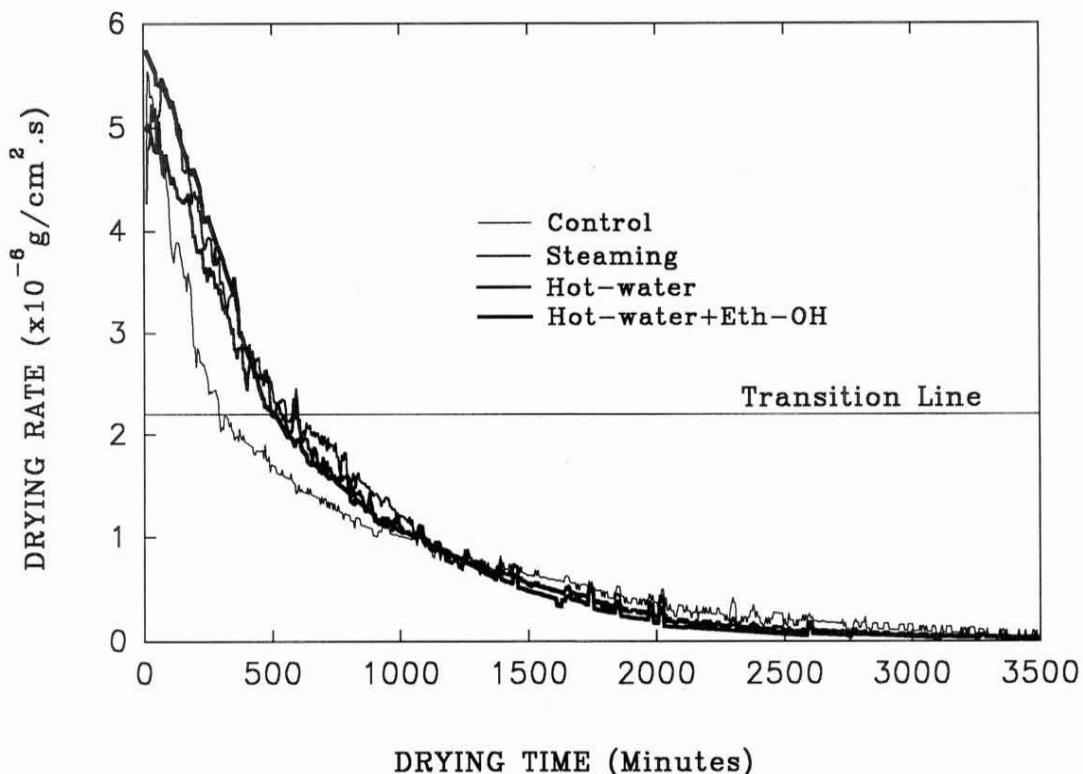


FIG. 4. Drying rate as a function of drying time.

changes in drying rates in the falling rate period. There was a sharp transition in the drying rate at about $2.2 \times 10^{-6} \text{ g/cm}^2 \cdot \text{sec}$. For the untreated (control) samples, the transition point is 300 min; and for the treated samples, the transition points range from 500 to 600 min (Fig. 4). The drying rates, when expressed as a function of MC (Fig. 3), also show the same transition. For the untreated samples, the transition point is 67% MC, and for the treated samples the transition points range from 51 to 56% MC. The shapes of these curves are not the same. Above the transition line, the slope of drying rate in the untreated samples is much steeper than the slopes of drying rates in the treated samples. Below this line, the slopes in the treated samples are higher than the slope in the untreated samples, suggesting that the resistance to moisture movement in the untreated samples was relatively large and that treatments to remove and rearrange the extractives reduced this resistance and increased

the drying rates over the entire falling rate period.

There were some limitations in this study. Only small samples were used, which may not be representative of a large piece of lumber in commercial drying. In addition, only one sample could be introduced into the measuring system each time; therefore, the drying conditions in the environmental box must be controlled to obtain a reliable comparison among samples and to accurately determine treatment effects.

CONCLUSIONS

Wood extractives in red oak heartwood affected the moisture movement during drying, and removing and/or rearranging the extractives by steaming and hot-water soaking increased the diffusion coefficient as well as the drying rate. The effect of wood extractives was due mainly to the water-soluble extractive components. Randomized complete block de-

sign was effective and necessary to minimize the confounding effect of variable extractive distribution in various locations within a board. The constant drying rate period was not observed, but the falling rate was noticeable. The drying rate for the untreated controls decreased with a decrease in MC at a faster rate at high MC range than at low MC range; but the drying rates for the treated samples are approximately proportional to MC.

The "continuous" measuring system was reliable, accurate, and labor-saving. The drying rate could be readily determined from the measured data. However, only one sample could be introduced into the system, and therefore the drying conditions of the measuring system needed to be carefully controlled.

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