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Application of Optimum Solutions of a Diffusion Model to Evaluate the Effects of Extractives in Wood Drying.

Yong Chen

Louisiana State University and Agricultural & Mechanical College

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**Application of optimum solutions of a diffusion model to
evaluate the effects of extractives in wood drying**

Chen, Yong, Ph.D.

The Louisiana State University and Agricultural and Mechanical Col., 1994

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Ann Arbor, MI 48106

APPLICATION OF OPTIMUM SOLUTIONS OF A DIFFUSION MODEL
TO EVALUATE THE EFFECTS OF EXTRACTIVES
IN WOOD DRYING

A Dissertation

Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
in partial fulfillment of the
requirements for the degree of
Doctor of Philosophy

in

The School of Forestry, Wildlife, and Fisheries

by
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ABSTRACT

This study evaluates the influence of extractives on moisture diffusivity, and thereby the drying behavior of wood. Extractives are minor chemical components of wood, and their effect on moisture diffusion is sensitive to experimental and calculation errors. A series of studies designed to accurately determine the proper solutions of a diffusion model was done; then studies to determine the effect of extractives in a wood drying system were undertaken. The drying curves derived from the optimum diffusion coefficients gave better prediction of experimental drying data than those derived from traditional approximate methods; therefore, optimum methods were applied in the study on natural extractives as well as artificial extractives. Depending on treatment times, both hot-water soaking and steaming treatments increased the diffusion coefficients. This phenomenon suggested that both the amount of extractives and their locations in wood influenced the moisture movement during drying. When green wood samples were impregnated with polyethylene glycol or tannin, the drying rate decreased significantly. The contributing effect of extractives during drying is due to blockage of the moisture passages in the void spaces.

INTRODUCTION

Increasing the drying rate and improving the drying quality of wood require understanding all the factors affecting moisture movement. The amount of extractives is one factor, the effect of which on moisture diffusivity in wood has not been well documented. The hindrance comes from the fact that, as minor components relative to the cell wall substances, the effect of extractives on moisture diffusion is sensitive to experimental and calculation errors; therefore, the errors must be reduced in order to examine the effect of extractives on the diffusion coefficients accurately.

The main objectives of this research were to :

- 1) Study the effect of extractives on moisture movement using an instrumentation technique to minimize experimental errors,
- 2) Determine the best method of calculating the diffusion coefficient, by using optimum solutions of a diffusion model to enhance the accuracy and predictability of drying data,
- 3) Examine the effect of predrying treatments to remove or rearrange extractives in wood on moisture movement during drying by using the optimum solutions, and
- 4) Study the contribution of extractives in the cell walls or void spaces to drying rate.

Extractives, natural products extraneous to the lignocellulose cell wall, have considerable effects on many

wood physical properties, including shrinkage (Nearn 1955, Choong and Achmadi 1991), equilibrium moisture content (EMC) (Spalt 1958, Choong and Achmadi 1991), density, strength (Imamura 1989), and permeability (Choong and Achmadi 1989). Extractives in wood have also been reported to affect drying behavior. Resch and Ecklund (1964) found that among 12 variables, the amount of extractives was an influential factor in the reduction of the drying rate in redwood. The steaming of wood has been used as an aid in drying because it decreases the amount of extractives (Chen and Workman 1980) and redistributes it in wood (Nicholas and Thomas 1968, Alexion et al. 1990). Steaming has been reported by several researchers to decrease the drying time (Ellwood and Erickson 1962, Kininmonth 1971), and to increase the drying rate (Sharma and Bali 1969, Simpson 1975) and the diffusivity (Mackay 1971) in wood.

The moisture movement in wood during drying is controlled by the diffusion process; therefore, the diffusion coefficient in wood, which determines the rate of internal movement in relation to the existing moisture gradient, best describes the dryability of wood. In the past, approximate methods of calculating diffusion coefficients have been used by many researchers to describe general drying trends, but they do not give an accurate prediction of the drying rate. Besides experimental errors, there are several other sources of errors with approximate methods, including (1) truncation errors when

the theoretical solutions of the diffusion model are simplified, (2) subjective error from data selection, and (3) error due to inappropriate assumptions of the drying parameters and boundary conditions. A great effort has been made in this study to find better methods to accurately determine the effect of extractives during drying.

In Chapter I, a preliminary study was undertaken to ascertain whether the effect of extractives on dryability could be determined accurately by minimizing experimental errors. This was done using a "continuous" system of measuring drying data. The results indicated that the moisture movement in red oak heartwood during drying was affected by wood extractives. The conclusion, however, may not be applicable over the entire drying period since the accuracy of the diffusion coefficient was affected by inherent errors from using the approximate method in the Boltzmann equation.

Three methods for calculating the representative diffusion coefficient over the entire drying period are described in Chapter II through Chapter IV. These methods are based on the least squares principle and optimization techniques. Chapter II describes a method which allows the diffusion coefficient calculated from the theoretical Fourier series solution of a diffusion equation by assuming the surface moisture content drops immediately to equilibrium with the surroundings at the beginning of drying. It has been

demonstrated that this method can give a better description to the overall drying process than other existing approximate methods. Chapter III suggests a method to calculate the diffusion coefficient and the surface emission coefficient simultaneously from the corresponding Fourier series solution of the diffusion equation. Compared with the method in Chapter II, this method is more general since the moisture content at the surface of the wood actually drops to equilibrium gradually, rather than immediately. Chapter IV describes a method to evaluate the diffusivity of wood by using a numerical method. Application of this method makes it possible to calculate the diffusion coefficient as a function of moisture content, rather than by assuming it is constant. In addition, this method is more flexible than those from the theoretical approach as described in Chapters II and III when various boundary conditions related to the surface moisture are assumed.

With the calculation methods described above, Chapters V and VI examine the effect of extractives on dryability of wood over the entire drying period, but different aspects have been emphasized in each chapter. Chapter V studies the effects of steaming and hot-water soaking on the diffusivities of southern hardwoods. As a means of predrying treatments, steaming and hot-water soaking could not only decrease the amount of extractives but also to relocate them in wood. The experimental results indicated that these predrying treatments

improved the dryability, although their effects were not as large as those due to species and structural directions of wood. Chapter VI examines the effects of bulking agents (including natural extractives and bulking chemicals as artificial extractives) on the dryability of wood. The experimental results indicated that hot-water soaking slightly increased the diffusion coefficients of redwood and red oak heartwood, while impregnating southern pine and yellow poplar sapwood with bulking chemicals (polyethylene glycol or tannin) considerably decreased their diffusion coefficients. In addition, the effects of the bulking agents on shrinkage and permeability of the woods were also examined. The data supported the hypothesis that the change in drying rate is due to the blockage of the macro-passage in the cell void spaces.

It is hoped that the knowledge gained from analyzing the diffusion model and the effect of extractives on moisture movement will enhance the predictability of the wood drying process. This may eventually lead to the development of a better drying process that will dry wood faster and improve its quality.

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CHAPTER I

DETERMINING THE EFFECT OF EXTRACTIVES ON MOISTURE MOVEMENT USING A "CONTINUOUS" MEASURING SYSTEM

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) has reported that wood extractives have significant effects on some wood properties such as equilibrium moisture content 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 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-minute intervals by a computer-controlled data acquisition system in order to eliminate errors of a manual-weighing procedure. In addition, the variation of drying rates with drying time and moisture content (MC) was examined.

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 hours; (3) hot-water soaking at $70\pm 2^{\circ}\text{C}$ for 24 hours; and (4) ethyl alcohol extraction in a Soxhlet extractor for 24 hours after hot-water soaking. The hot-water soaking was accomplished in a beaker which 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 $70\pm 2^{\circ}\text{C}$. 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 in 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 hours in hot water at $70\pm 2^{\circ}\text{C}$. 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 minutes. 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 (18cm × 20cm × 22cm) 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 Figure 1.1. 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±2 percent 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 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-minute intervals (the air flow was stopped for 10 seconds 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.

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

$$D = \frac{\pi a^2 (1 - \bar{E})^2}{4t} \quad (1.1)$$

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. 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 (FSP).

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

$$F = \frac{W_{i+1} - W_{i-1}}{2A \Delta t} \quad (1.2)$$

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

(cm²), and the Δt is the time interval of sampling, which was 300 seconds (5 minutes) in this study.

RESULTS AND DISCUSSION

The \bar{E} values as a function of drying time, shown in Figure 1.2, indicate that they decreased faster in the treated samples than in the untreated samples when dried from about 100 to about 15 percent MC. The average diffusion coefficients at $\bar{E}=0.5$ are given in Table 1.1. An analysis of variance for the diffusion coefficients (Table 1.2) shows 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 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 1.1). 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.

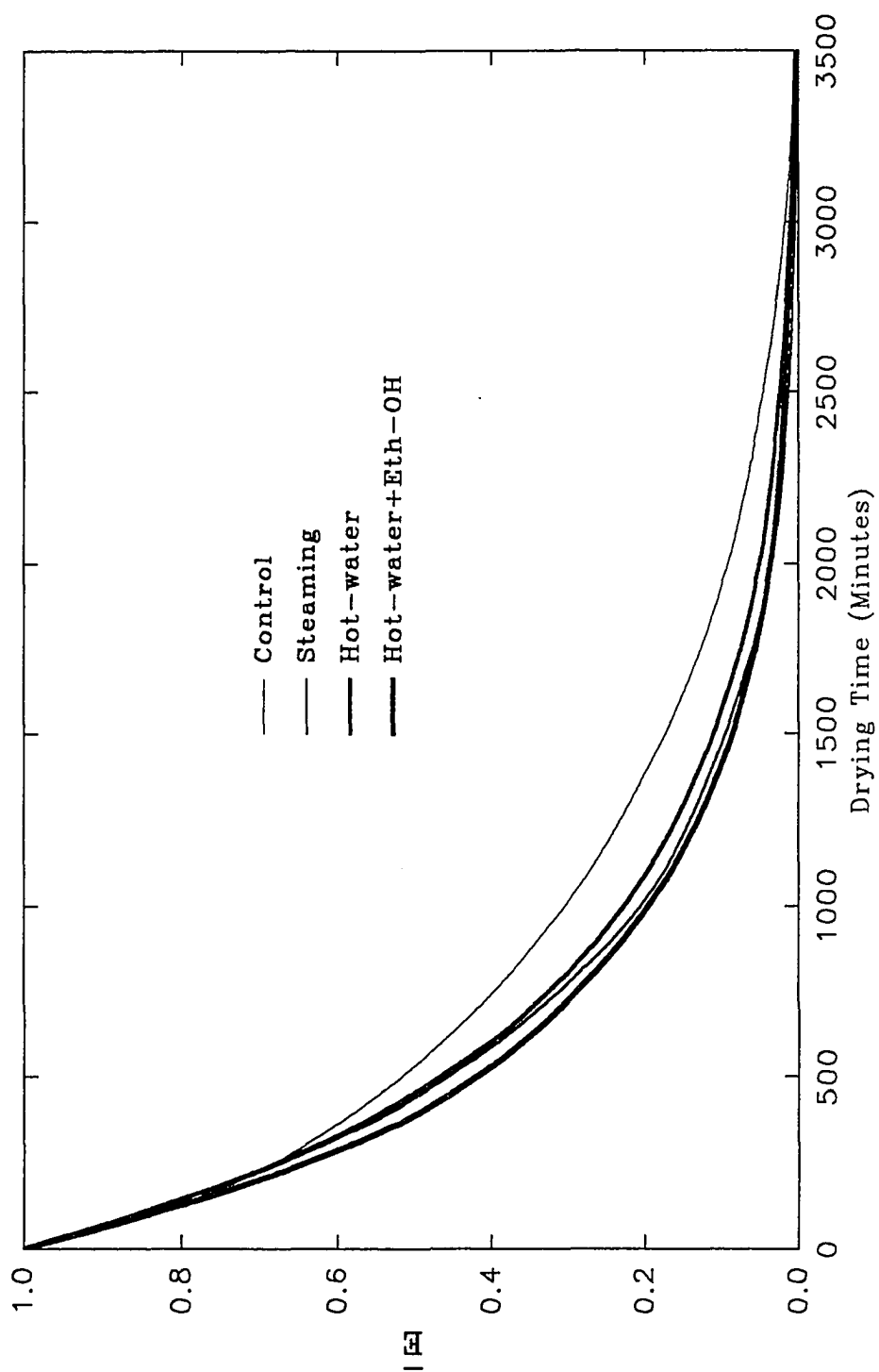


Figure 1.2 \bar{E} values as a function of drying time

Table 1.1. Average diffusion coefficients of red oak among four treatments

Block	Control	Steaming	Hot water	ethanol
... $\times 10^{-6} \text{cm}^2/\text{s}$...				
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 ^a	1.47 B	2.52 A	2.48 A	3.15 A

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

Table 1.2. 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	--	--

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 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 one, when various drying processes are partially superimposed (Hildebrand 1970). The drying rates for various treatments as a function of MC (Figure 1.3) 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) has 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 great. 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 time.

The treatments to remove extractives led to changes in drying rates in the falling rate period. There was a sharp transition in the drying rate at about 2.2×10^{-6} g/cm².s. For the untreated (control) samples, the transition point is 300 minutes, and for the treated samples the transition points range from 500 to 600 minutes (Figure 1.4). The drying rates, when expressed as a function of MC (Figure 1.3), also show the same transition. For the untreated samples, the transition point is 67 percent MC, and for the treated samples the

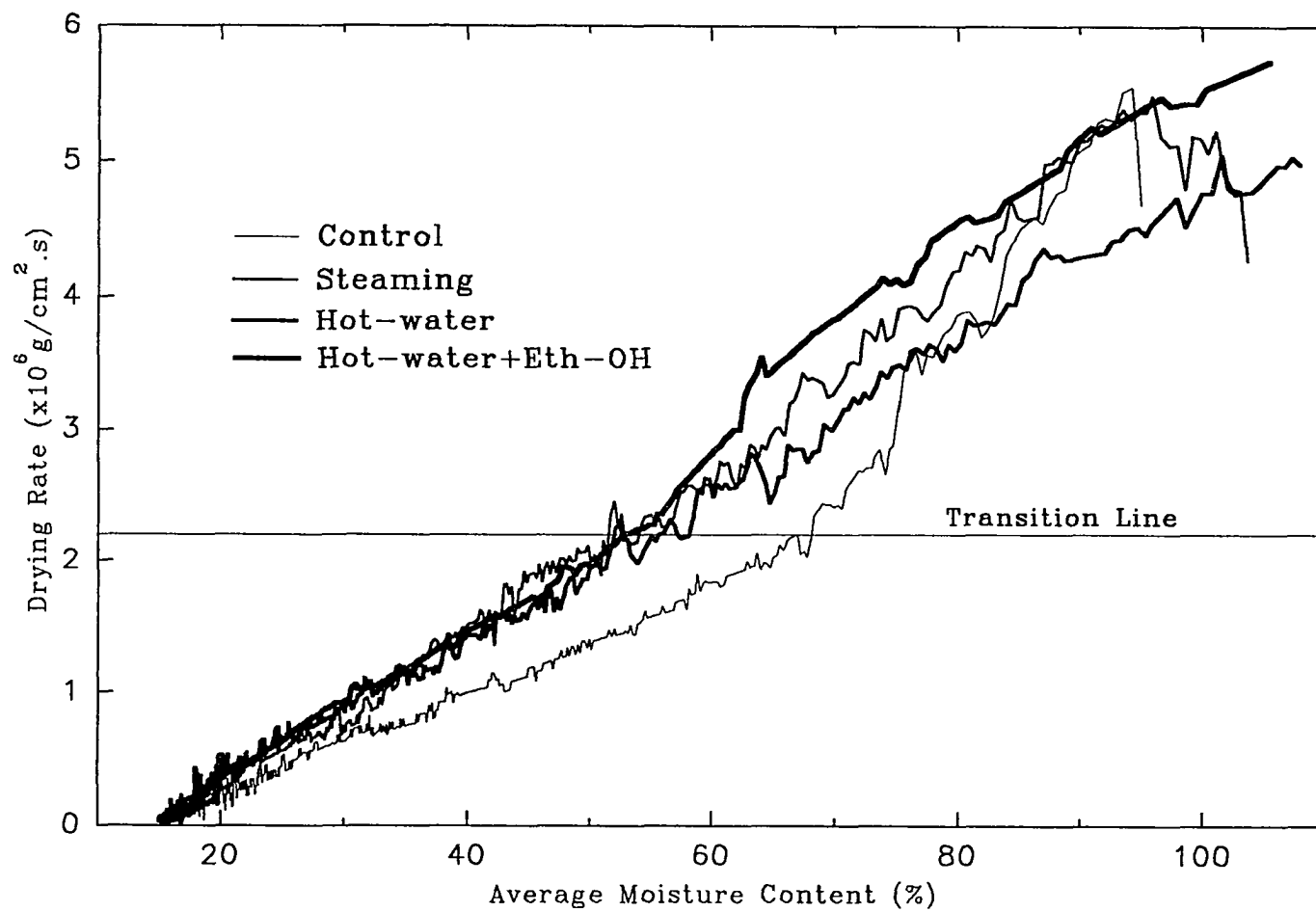


Figure 1.3. Drying rate as a function of moisture content

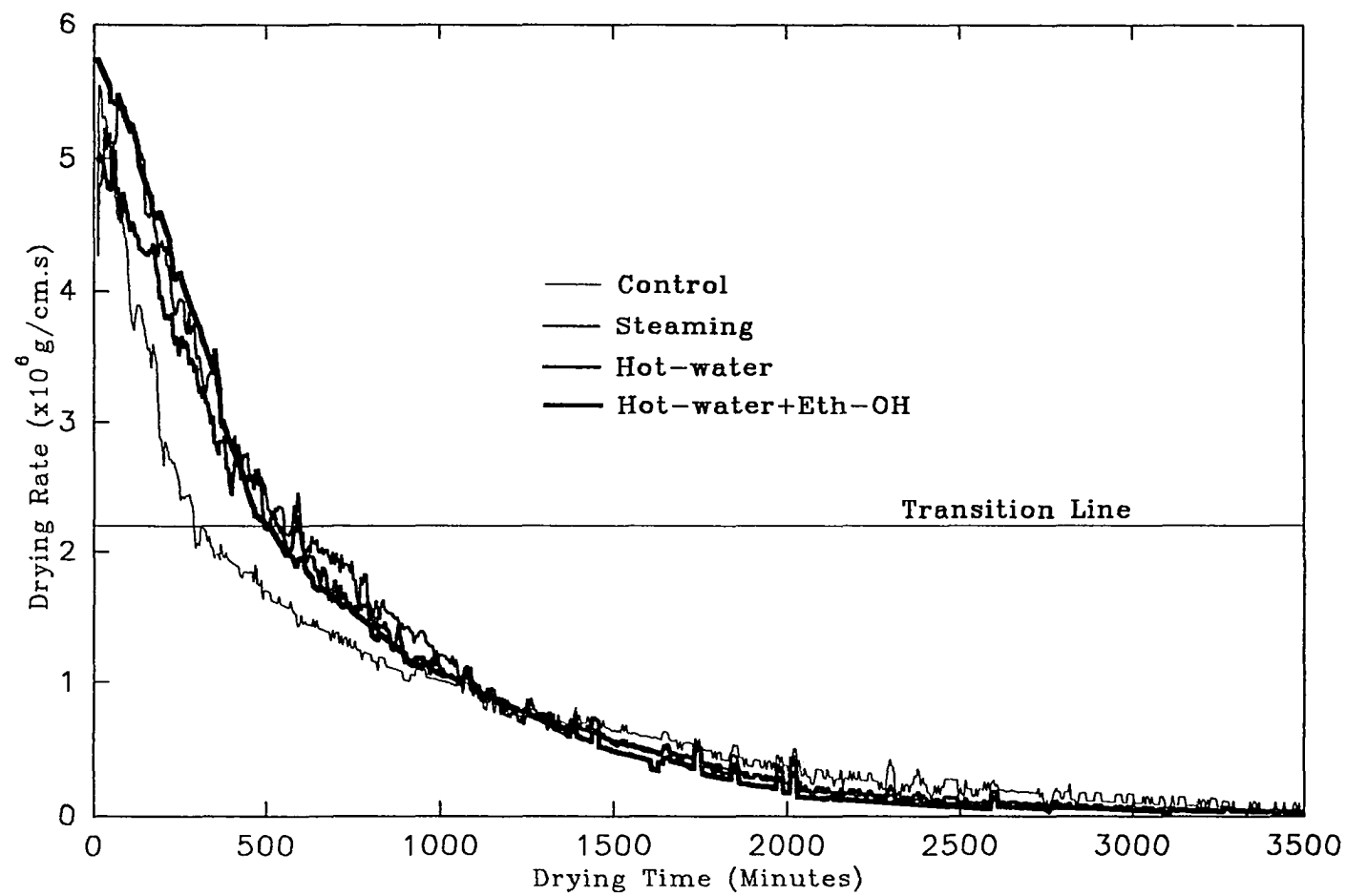


Figure 1.4. Drying rate as a function of drying time

transition points range from 51 to 56 percent 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 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 determine accurately the effects of treatments. In addition, the conclusion may be not applied over the entire drying period since the accuracy of the diffusion coefficient was affected by inherent errors from using the approximate method in the Boltzmann equation.

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 mainly due to the water-soluble extractive components. A randomized complete block design 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. In addition, an appropriate method to calculate the diffusion coefficient should be studied in order to reduce the inherent errors due to the approximate nature of the Boltzmann equation.

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CHAPTER II

OPTIMUM AVERAGE DIFFUSION COEFFICIENT: AN OBJECTIVE INDEX IN DESCRIPTION OF WOOD DRYING DATA

INTRODUCTION

The moisture diffusion coefficient is an important index in determining the drying rate of wood. Although the surface emission coefficient may also prove a predominant factor under certain circumstances, such as in thin wood boards drying at low air velocity, its effect is usually not important in common kiln drying conditions with high air velocity. As a result, most research has concentrated on determining the properties of the moisture diffusion coefficient. With the diffusion coefficient assumed constant, the mathematics become relatively simple due to the availability of the theoretical solution of the partial differential equation expressed by Fick's second law. However, past research results (Stamm 1964, Choong 1965, Moschler and Martin 1968, Rosen 1976) indicate that the moisture diffusion coefficient is not a constant, but rather a function of moisture content and diffusion direction. Therefore, only average values of diffusion coefficients can be derived from the unsteady-state drying curve.

In practice, three schemes (i.e., square-root, half- \bar{E} , and logarithmic methods) have been suggested for determining the average diffusion coefficient.

The square-root scheme, based on the equation derived by Boltzmann (Stamm 1964), is valid for short times:

$$D = \frac{\pi a^2 (1 - \bar{E})^2}{4t} \quad (2.1)$$

In Eq. 2.1, \bar{E} , a , D , and t represent the fraction of evaporable moisture present in wood, half-thickness of a wood sample, diffusion coefficient, and drying time, respectively. The average diffusion coefficient derives from the slope of the curve when $(1-\bar{E})$ is plotted versus \sqrt{t} (Siau 1984).

The half- \bar{E} scheme is based on Crank's (1975) suggestion that the average diffusion coefficient can be estimated by the value of D in Eq. 2.1 when $\bar{E}=0.5$.

The logarithmic scheme, valid for long times (Crank 1975), is:

$$D = -\frac{4a^2}{\pi^2} \frac{d(\ln \bar{E})}{dt} \quad (2.2)$$

This equation is derived from the theoretical solution of Fick's second law under the equilibrium boundary condition (Skaar 1954):

$$\bar{E} = (8/\pi^2) \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} e^{-\left(\frac{2n-1}{2a}\right)^2 \pi^2 D t} \quad (2.3)$$

by truncating all terms except the first, taking the natural logarithm on both sides, and then differentiating the simplified equation. The average diffusion coefficient can be calculated from the slope of $\ln(\bar{E})$ versus t .

Generally, these schemes have two major common characteristics. First, because they were approximate equations, only the first term in the infinite series was used in each equation. While the truncation procedure is

appropriate when having a truly constant diffusion coefficient, few discussions in the literature (Crank 1975) justify it when the diffusion coefficient is not a constant, or give the percent error due to such a procedure. Second, they are all subjective methods. For example, when using the square-root scheme, different people may obtain different slopes. This variation arises because the curve is usually not strictly straight, and, therefore, the portion taken as the straight line depends on personal preference. The decision making may prove more difficult if relatively large experimental errors occur. In the half- \bar{E} scheme, $\bar{E}=0.5$ is obviously an arbitrary choice. Unless we know the exact form of a diffusion coefficient as a function of moisture content, there is no guarantee that $\bar{E}=0.5$ is the best representative point for the calculation of the average diffusion coefficient. Additionally, the diffusion coefficients calculated from these three schemes are generally expected to differ. In the square-root scheme, the diffusion coefficient is calculated from drying data when the moisture content is still high. In the logarithmic scheme, the diffusion coefficient is calculated when the moisture content is low.

To overcome the aforementioned limitations, a new scheme to calculate the average diffusion coefficient is suggested. Unlike other schemes, this method takes all data points into account in the calculation, so that the calculated results represent average values of the diffusion coefficient. As an

objective method, a given set of drying data will yield a unique answer. In addition, the error due to truncation in the other schemes will be greatly diminished. The average diffusion coefficient is an optimum one in the sense of the least squares principle. Once the optimum average diffusion coefficient is found, the theoretical \bar{E} value curve can accordingly be derived. Therefore, the effect of the deviation of the diffusion coefficient from a constant value can be examined by comparing the experimental data with the calculated curves.

The objective of this study is to determine the optimum average diffusion coefficient. The hypothesis is that the experimental drying data over the entire range of drying period can be best predicted, in the sense of the least squares principle, by using the optimum average diffusion coefficient.

OPTIMIZATION PROCEDURE

The optimization procedure is based on Eq. 2.3, the theoretical Fourier series solution. If an arbitrary average diffusion coefficient is selected, a theoretical \bar{E} for any time value can be calculated. When the sum of squares of the differences of each experimental and theoretical \bar{E} pair is studied, it can be observed in Figure 2.1, there exists a unique average diffusion coefficient at which the sum of squares of the \bar{E} differences $\Sigma(\Delta\bar{E})^2$ is minimized. This point

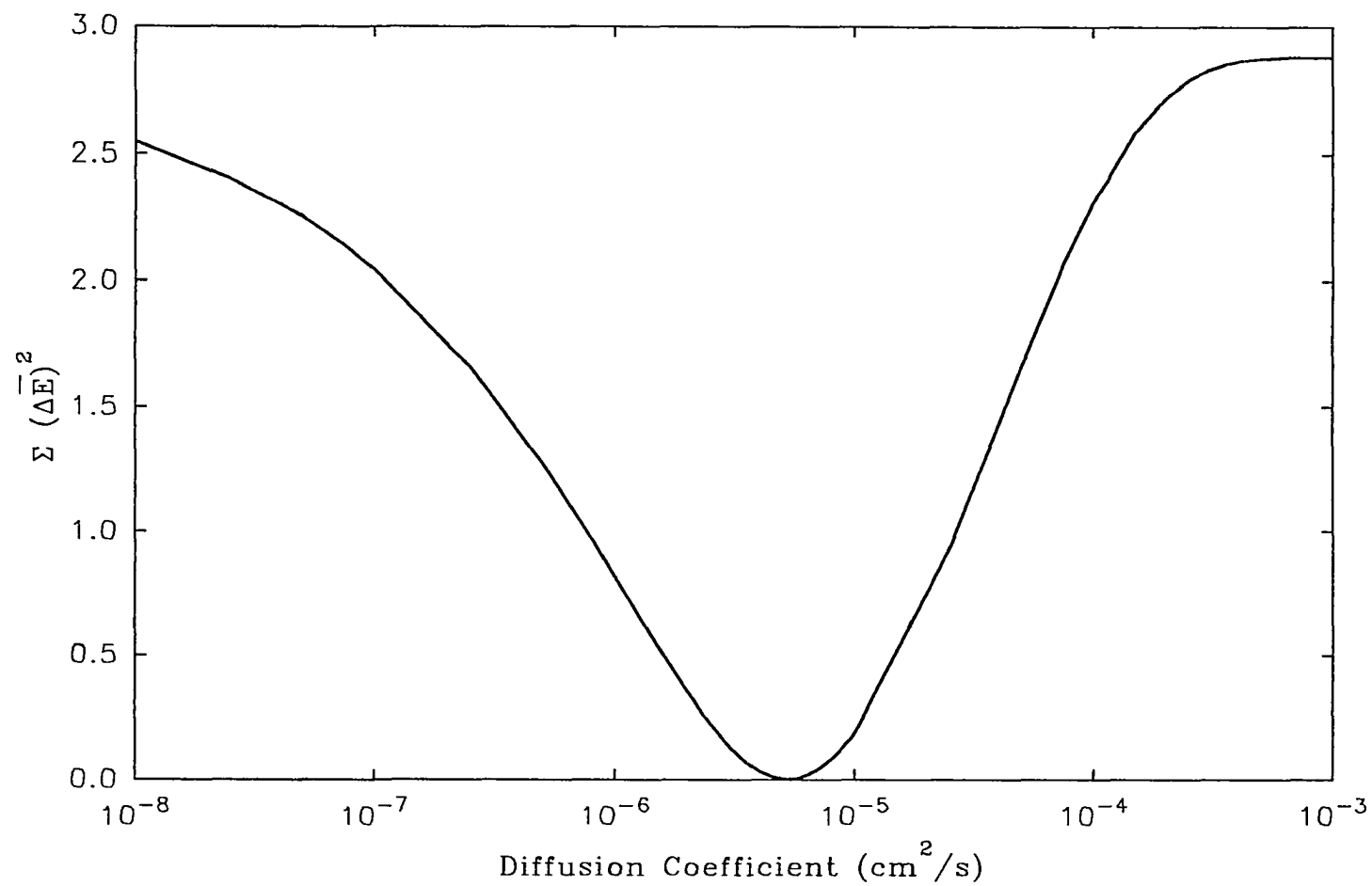


Figure 2.1. Variation of sum of squared difference of predicted and experimental \bar{E} pair as a function of average diffusion coefficient.

is defined as the optimum average diffusion coefficient. To find this point, the expected lower bound and upper bound values of the diffusion coefficient were first set. Then, the optimum average diffusion coefficient based on the *golden section search* principle (Fletcher 1980) was located. This task would be tedious if attempted by hand; therefore, a computer algorithm was developed, and the FORTRAN program (Appendix A) was written. In this program, all terms in the PARAMETER declaration can be adjusted according to each situation. The units of half-thickness are in centimeters. The ITEM value should be adjusted to the number of data points for each experiment. As reflected in the real variable declaration in the main program, the maximum number of data points is 30; however, this can be adjusted according to individual situations. The lower bound, upper bound, and optimum values of diffusion coefficient are output as the final results. The program can be executed in most 32-bit machines.

NUMERICAL EXAMPLE AND DISCUSSION

Mamit (1983) dried samples of six hardwood species with a nominal half-thickness of 1.27 cm in either the longitudinal or the tangential direction from the fiber saturation point (FSP) to an equilibrium moisture content of 12 percent. The environmental chamber, controlled at temperature 43 °C and 45 percent relative humidity (RH), maintained an air speed of

about 1.5 meters per second. To demonstrate the effect of the proposed scheme, Mamit's data of red oak and sweetgum heartwood were reanalyzed.

Examples of graphs used to determine the average diffusion coefficients based on the square-root scheme and the logarithmic scheme are given in Figures 2.2 and 2.3, respectively. The experimental values of \bar{E} and the calculated curves based on the optimum average diffusion coefficients are shown in Figure 2.4. The average diffusion coefficients based on the four schemes are summarized in Table 2.1. When the calculated values of the present optimum scheme are taken as a basis, the percent deviation of the Half- \bar{E} scheme from the present one is relatively small, with an average value of 5.9 percent. The percent deviations for the square-root scheme and the logarithmic scheme are relatively large, with average values of 25.4 percent for the square-root scheme and 27.8 percent for the logarithmic scheme, respectively.

When compared with those for other schemes, as shown in Table 2.2, the calculated values of the sum of squares of the differences of the experimental and theoretical \bar{E} pair for the present optimum scheme prove minimal. As a result, a close prediction to the experimental data of drying can be achieved over the entire drying period (Figure 2.5). The data in Table 2.2 also support Crank's suggestion that calculated diffusion coefficients based on the half- \bar{E} scheme give reasonable average values, though not optimum ones. The average values

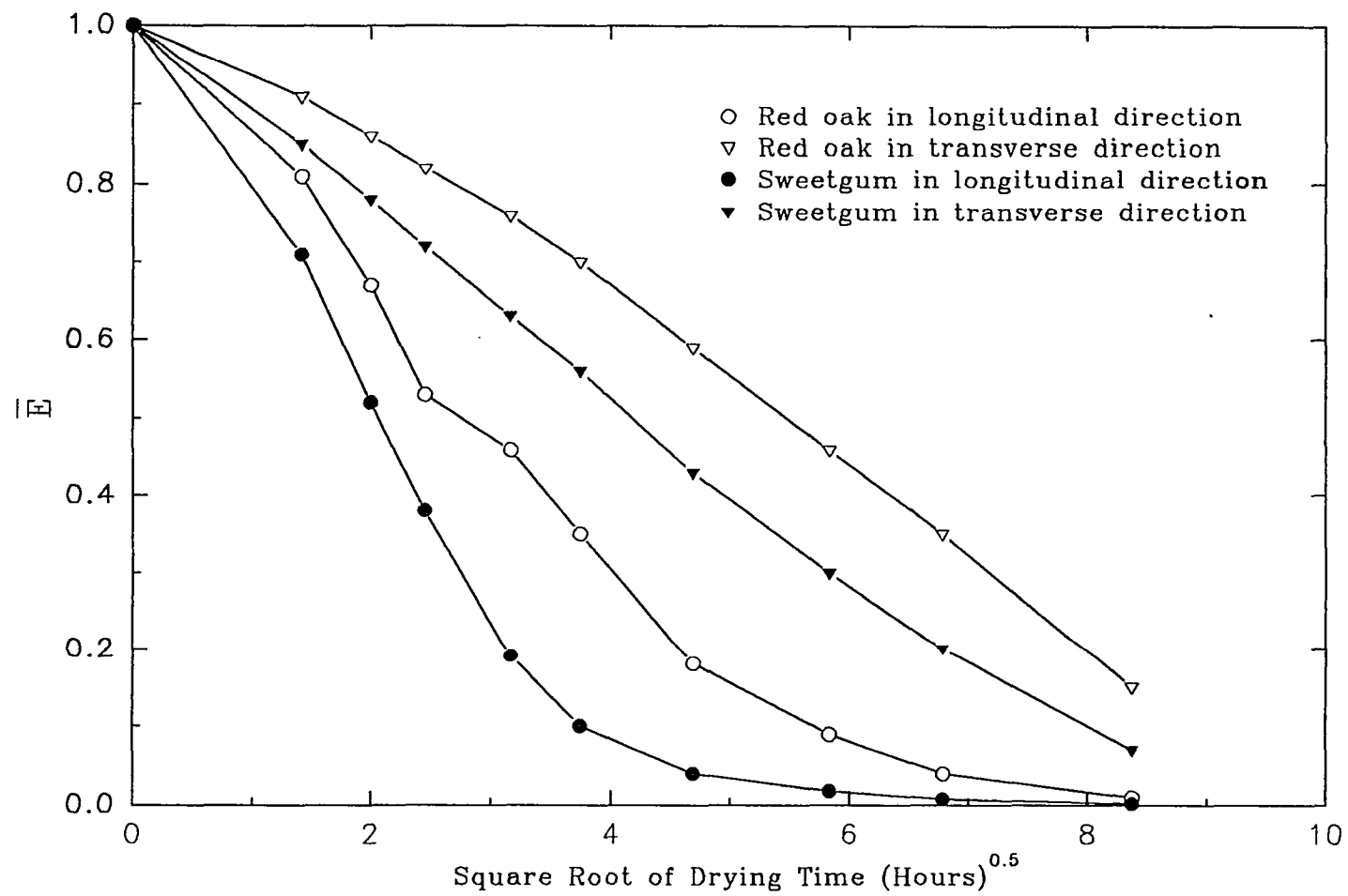


Figure 2.2. Relationship between \bar{E} and square root of time for red oak heartwood and sweetgum heartwood.

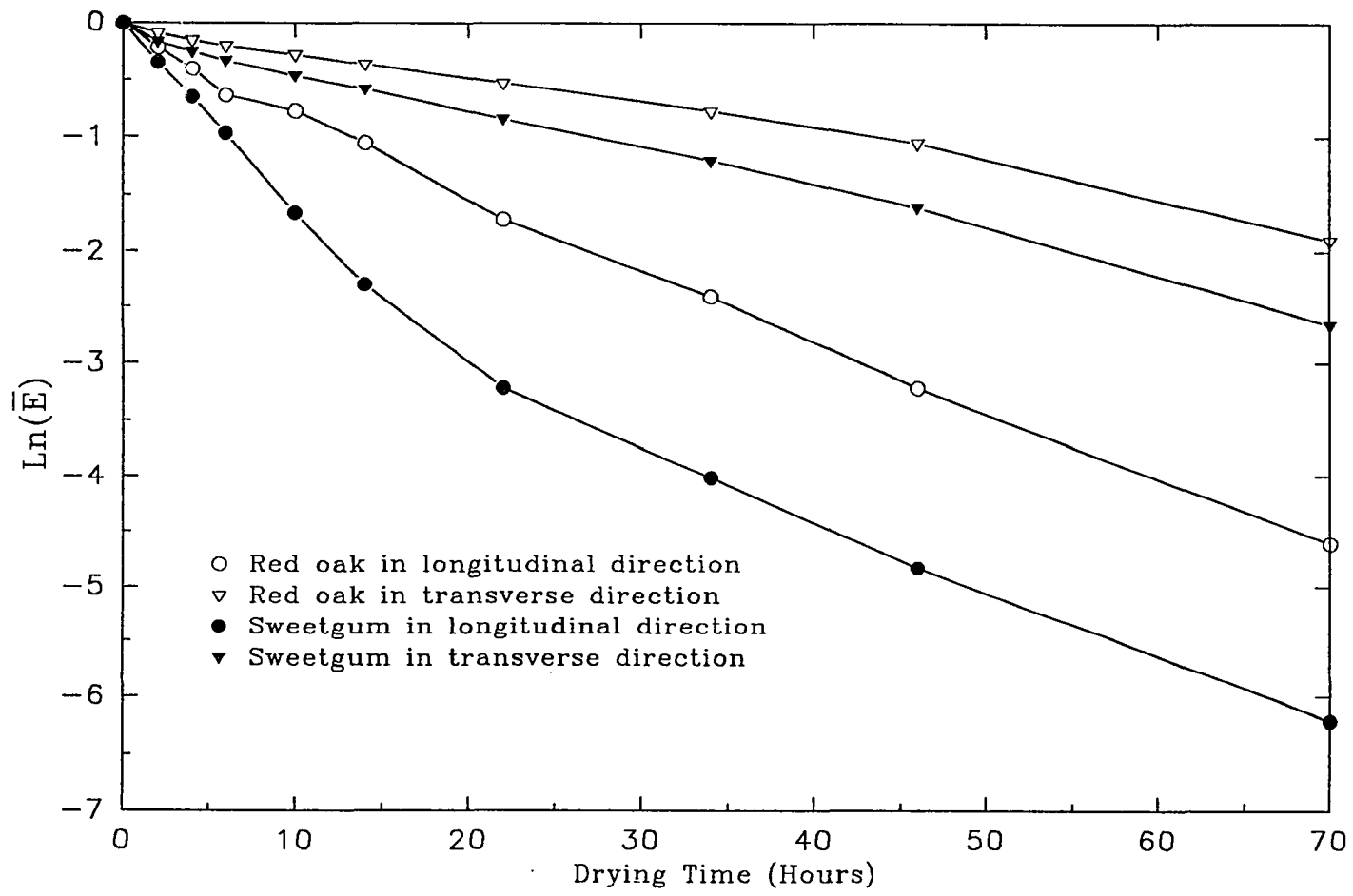


Figure 2.3. Relationship between natural logarithmic \bar{E} and time for red oak heartwood and sweetgum heartwood.

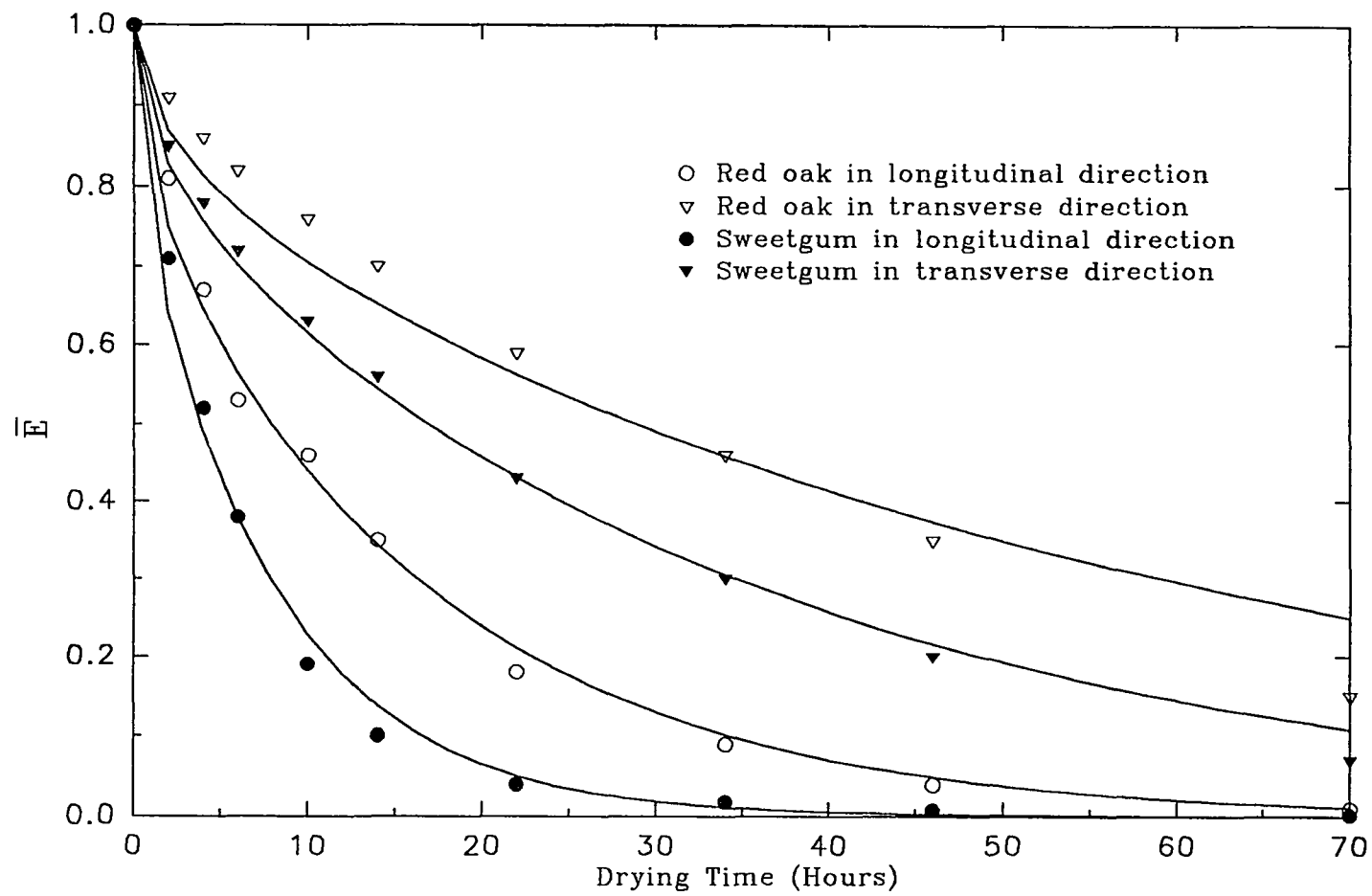


Figure 2.4. Comparison of predicted \bar{E} curves with experimental \bar{E} data as a function of time for red oak heartwood and sweetgum heartwood.

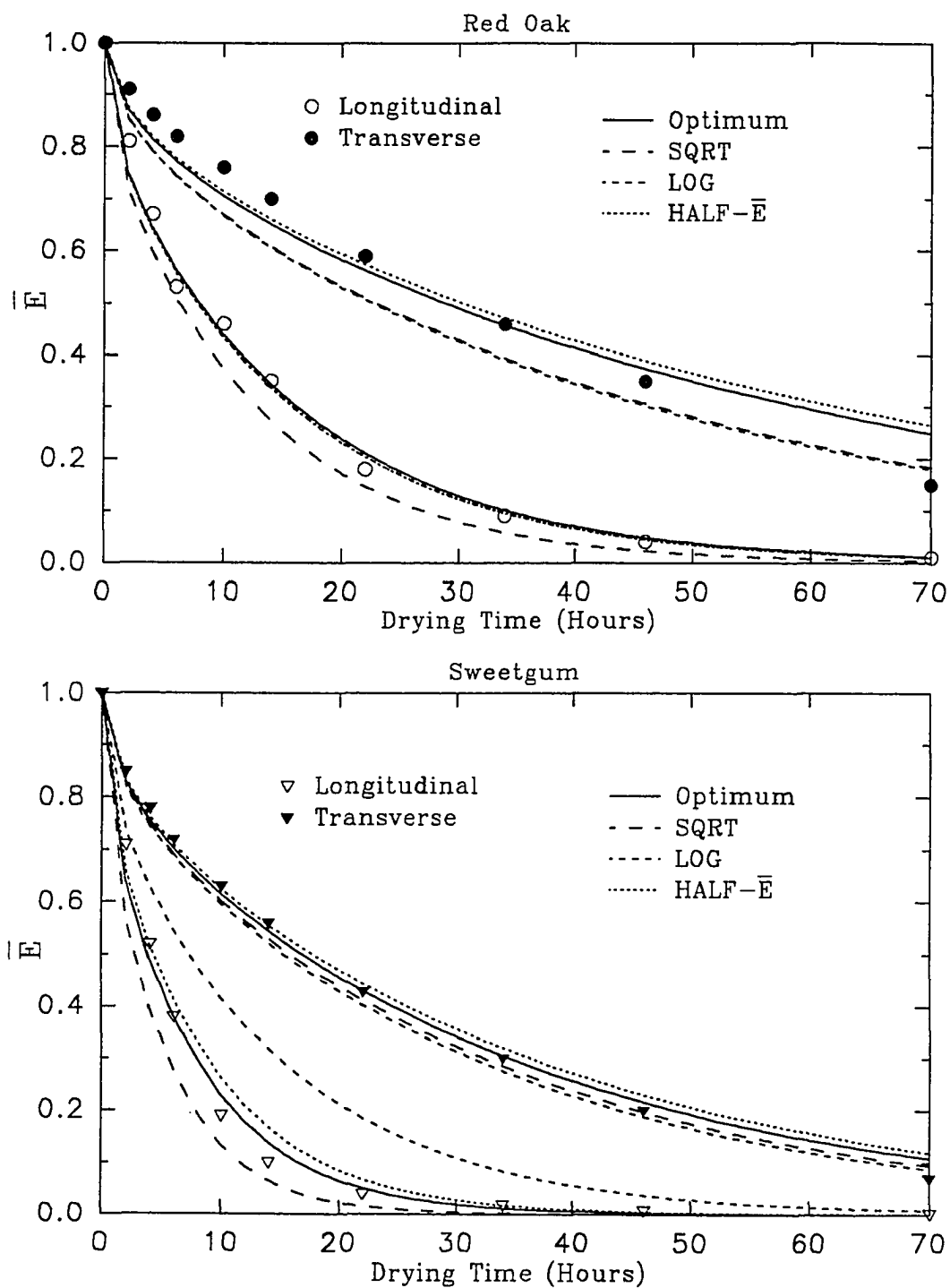


Figure 2.5. Comparison of predicted curves by four methods for red oak heartwood and sweetgum heartwood.

Table 2.1. Values^a of the average diffusion coefficients based on the four schemes

Species	Direction	SQRT ^b	LOG	HALF- \bar{E}	OPTIMUM
... cm ² /s ...					
Red oak	Longitudinal	1.41N5	1.14N5	1.14N5	1.11N5
	Transverse	3.85N6	3.90N6	2.90N6	3.06N6
.....					
Sweetgum	Longitudinal	3.28N5	1.22N5	2.05N5	2.30N5
	Transverse	5.58N6	5.77N6	4.97N6	5.23N6

^a The notation xNy means $x \times 10^y$.^b Results from Mamit (1983).Table 2.2. Values^a of $\Sigma(\Delta\bar{E})^2$ based on the four schemes

Species	Direction	SQRT	LOG	HALF- \bar{E}	OPTIMUM
Red oak	Longitudinal	2.97N2	7.42N3	7.45N3	7.21N3
	Transverse	4.44N2	4.70N2	2.39N2	2.28N2
.....					
Sweetgum	Longitudinal	5.03N2	1.63N1	1.40N2	9.14N3
	Transverse	5.49N3	7.93N3	4.75N3	3.61N3

^a The notation xNy means $x \times 10^y$.

for the square-root, the logarithmic, and the half- \bar{E} schemes are 3.3, 5.8, and 1.2 times larger than that for the present optimum one, respectively. They are 2.5, 1.8, and 1.1 times larger when the values of sweetgum in longitudinal direction with a relatively large deviation from the optimum one are excluded.

Two advantages of the present optimum scheme have been demonstrated in the example. First, the subjective error in the calculation has been eliminated. For example, in order to determine the average diffusion coefficients from the curves in Figure 2.2 based on the square-root scheme, one must decide which "straight" portion of a curve should be taken for the calculation of average diffusion coefficients. There is no such a decision-making step in the present scheme, since the optimum average diffusion coefficients are calculated objectively based on the least squares principle by a computer. Second, the present optimum scheme is most efficient when the diffusion coefficient is not a constant. For instance, since the optimum drying curve of red oak in the longitudinal direction (Figure 2.5) fits the experimental data satisfactorily, the diffusion coefficient should be close to a constant. Under the circumstances, the drying curves derived from the logarithmic scheme give the prediction of the experimental data as well as that from the present optimum one. The predicted curves of red oak in the transverse direction, on the other hand, deviate obviously from the

experimental data, suggesting the diffusion coefficient is not a constant. In this case, the value of $\Sigma(\Delta\bar{E})^2$ of the logarithmic scheme is 2.1 times larger than that of the present optimum one. Therefore, an improved prediction to the experimental data by the optimum scheme results.

Caution must be taken when the optimum scheme is applied. The assumption that the value of \bar{E} at the surface immediately drops to zero when drying begins should hold. If not, an error due to the neglect of surface resistance will result. In the example of Mamit's data, the air velocity is less than the critical value (3 m/s), as suggested by Rosen (1978); therefore, we expect that some errors due to surface resistance may already be confounded in the calculation. Several approaches have been suggested to estimate the magnitude of surface resistance (Choong and Skaar 1969 and 1972, Liu 1989), and a more complex computer algorithm should be developed to calculate the moisture diffusion coefficients and surface emission coefficients objectively.

CONCLUSIONS

- 1) The optimum values of diffusion coefficient can be determined from a single drying curve from the present optimum scheme.
- 2) The drying curves calculated from the present scheme gave a more accurate prediction to the experimental data than

those from the square-root, the logarithmic, and the half- \bar{E} schemes.

- 3) The present optimum scheme is most efficient when the diffusion coefficient is not a constant.

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CHAPTER III

EVALUATION OF DIFFUSION COEFFICIENT AND SURFACE EMISSION COEFFICIENT BY AN OPTIMIZATION TECHNIQUE

INTRODUCTION

The diffusion coefficient in wood determines the rate of internal moisture movement, while the surface emission coefficient describes the rate at which moisture is emitted from the surface of drying wood. Methods to evaluate these two coefficients in wood drying practice have been studied extensively since Skaar (1954) introduced Newman's general solution (Newman 1931) of the unsteady-state diffusion equation into the field of wood drying. Based on a graphical solution of Newman's equation, Choong and Skaar (1969) suggested an approximate method to evaluate the diffusion coefficient, D , and the surface emission coefficient, S . According to this method, D and S can be calculated if, for two samples with different thicknesses, the drying times are known for reduction of the fractional moisture content (MC) \bar{E} to 0.5. Later, Choong and Skaar (1972) developed a more general form to enable D and S to be calculated from simultaneous drying experiments on samples of several different thicknesses. Using the same analytical procedure, Rosen (1978) proved that air velocity had a significant influence on the surface emission coefficient, which was related to the external resistance in the sorption process. He found that the relative importance of external resistance to moisture movement in wood increased as specimen thickness and/or air velocity decreased. Liu (1989) described a modified method based on Choong and Skaar's (1969) approach to

estimate D and S from a single lumber-drying curve. His method requires knowing both the drying time and the curve's slope of the fractional moisture content-drying time curve at $\bar{E}=0.5$.

There are two common characteristics of these approximate methods. First, the calculations are carried out at one point, $\bar{E}=0.5$. Second, the resulting diffusion coefficient and surface emission coefficient are assumed to be constant, independent of \bar{E} . If the final aim is to predict accurately the experimental data over the entire drying period, the pair of coefficients evaluated at $\bar{E}=0.5$ may not be the only ideal ones, since the diffusion coefficient is generally not constant but a function of wood MC (Comstock 1963, Stamm 1964, Choong 1965). There were no published data available to describe how closely the pair evaluated at $\bar{E}=0.5$ corresponded to those evaluated at other values of \bar{E} .

The objectives of the this study were: 1) to search the best pair of diffusion coefficients and surface emission coefficients by using an optimization technique, based on the least squares principle and Newman's general theoretical solution, and 2) to evaluate qualitatively how the variation of diffusion coefficients with MC affects the predictability of the drying curve. The hypothesis is that a better prediction of the drying curve can be achieved when the optimum pair of coefficients is applied.

BASIC EQUATIONS AND OPTIMIZATION PROCEDURE

The general partial differential equation for unsteady-state moisture diffusion in the direction perpendicular to the surface can be expressed by Fick's second law:

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial X} \left(D \frac{\partial C}{\partial X} \right) \quad (3.1)$$

where C is moisture concentration (or MC); t time; X distance from the centerline; and D diffusion coefficient, which is a function of moisture concentration.

Initial and boundary conditions for moisture evaporation at the surface are given by

$$C = C_o, \quad -a < X < a, \quad t=0 \quad (3.2)$$

and

$$\pm D \frac{\partial C}{\partial X} = S(C_e - C_a), \quad X = \pm a, \quad t \geq 0 \quad (3.3)$$

where C_e is the moisture concentration in equilibrium with the water vapor pressure in the surrounding air, C_a is the actual moisture concentration in the surface at any time, a is the half-thickness of the specimen, and S is the surface emission coefficient.

If D is a constant, a theoretical solution for Eq. 3.1, as given by Newman (1931), is:

$$\bar{E} = \frac{\bar{C} - C_e}{C_o - C_e} = 2L^2 \sum_{n=1}^{\infty} \frac{\exp(-\beta_n^2 \tau)}{\beta_n^2 (\beta_n^2 + L^2 + L)} \quad (3.4)$$

where \bar{C} is the average value of C across the specimen thickness, β_n are the positive roots of

$$\beta_n \tan \beta_n = L \quad (3.5)$$

where

$$L = Sa/D \quad (3.6)$$

and

$$\tau = Dt/a^2. \quad (3.7)$$

Since the diffusion coefficient in wood during drying is generally a function of moisture concentration, the D calculated from Eq. 3.4 represents some average value over the concentration range.

In the present work the alternate approach of searching for an optimum pair of constants, D and S , was used. This approach minimized the sum of squares of the differences between experimental data and Eq. 3.4. To fulfill this task, an ascent optimization algorithm was applied. The search can begin from any reasonable pair of D and S values. After each iteration, a new D and S pair with a smaller sum of squares is obtained. Finally, the search stops after the reduction in the sum of squares from one iteration to the next is less than the given closure criterion. A FORTRAN program was written (Appendix B) to accommodate the calculation¹. In this

¹A part of the FORTRAN codes was from class notes in the computer course "Advanced Numerical Methods" offered at Louisiana State University.

program, a bisection technique is employed to guarantee the finding of all positive roots accurately in Eq. 3.5 for any L . A conjugate gradient technique is used to increase the climbing speed toward the minimum absolute value of *Negative Mean Sum of Squares* (NMSS), which is defined as $-\Sigma(\Delta\bar{E})^2/N$, where $\Delta\bar{E}$ is the difference in the fractional moisture content between the fitted curve and the experimental data, N is the number of data points. When the optimum pair of D and S has been found, the corresponding drying curve can be computed from Eq. 3.4. It is also easy to develop the response surface of the NMSS as a function of D and S , or D and L , so as to examine the sensitivity of the NMSS near the vicinity of their optimum values.

NUMERICAL EXAMPLE AND DISCUSSION

Choong and Skaar (1969) evaluated the D and S at $\bar{E}=0.5$ using their approximate method for yellow-poplar sapwood and heartwood specimens, dried in either the tangential or the radial direction. Two samples of different thicknesses were dried for each combination in an environmental chamber at 32 ± 0.2 °C and 40 percent relative humidity at an air speed of 3.3 m/s. Their results for sapwood in the tangential direction were $D=7.5\times10^{-6}$ cm²/s and $S=1.52\times10^{-4}$ cm/s. Similar results were obtained by Liu (1989) using one sample and the slope technique. The data for sapwood in the tangential

direction were chosen for the evaluation of D and S in the present study.

When the internal resistance (represented by the diffusion coefficient) and external resistance (represented by the surface emission coefficient) were examined over the entire drying period, the internal resistance in this case was the predominant factor affecting the drying rate, and the external resistance was negligible. For the thick sample ($a=1.431$ cm), the calculated optimum D was always 5.25×10^{-6} cm^2/s , whereas the calculated optimum L varied from 5,000 to 25,000, or S from 1.83×10^{-2} to 9.17×10^{-2} cm/s . Similarly, for the thin sample ($a=0.479$ cm), the calculated optimum D was always 4.28×10^{-6} cm^2/s , whereas the calculated optimum L varied from 6,000 to 30,000, or S from 5.36×10^{-2} to 2.68×10^{-1} cm/s . These variations in L or S are the result of the relative insensitivity of NMSS to their values. The change in NMSS from one iteration to the next, which is the closure criterion, becomes sensitive to the initial values of D and S . On the response surface of the NMSS as a function of $\log(D)$ and $\log(L)$ (Figure 3.1), a ridge exists along which the searching usually stopped. A careful search along the ridge revealed the minimum absolute value of NMSS was virtually at $L=\infty$, or $S=\infty$. These are the values included in the calculated results of L , S , D , and the NMSS summarized in Table 3.1. The variation of the NMSS along the ridge as a function of $\log(L)$ for both samples is given in Figure 3.2. The search was

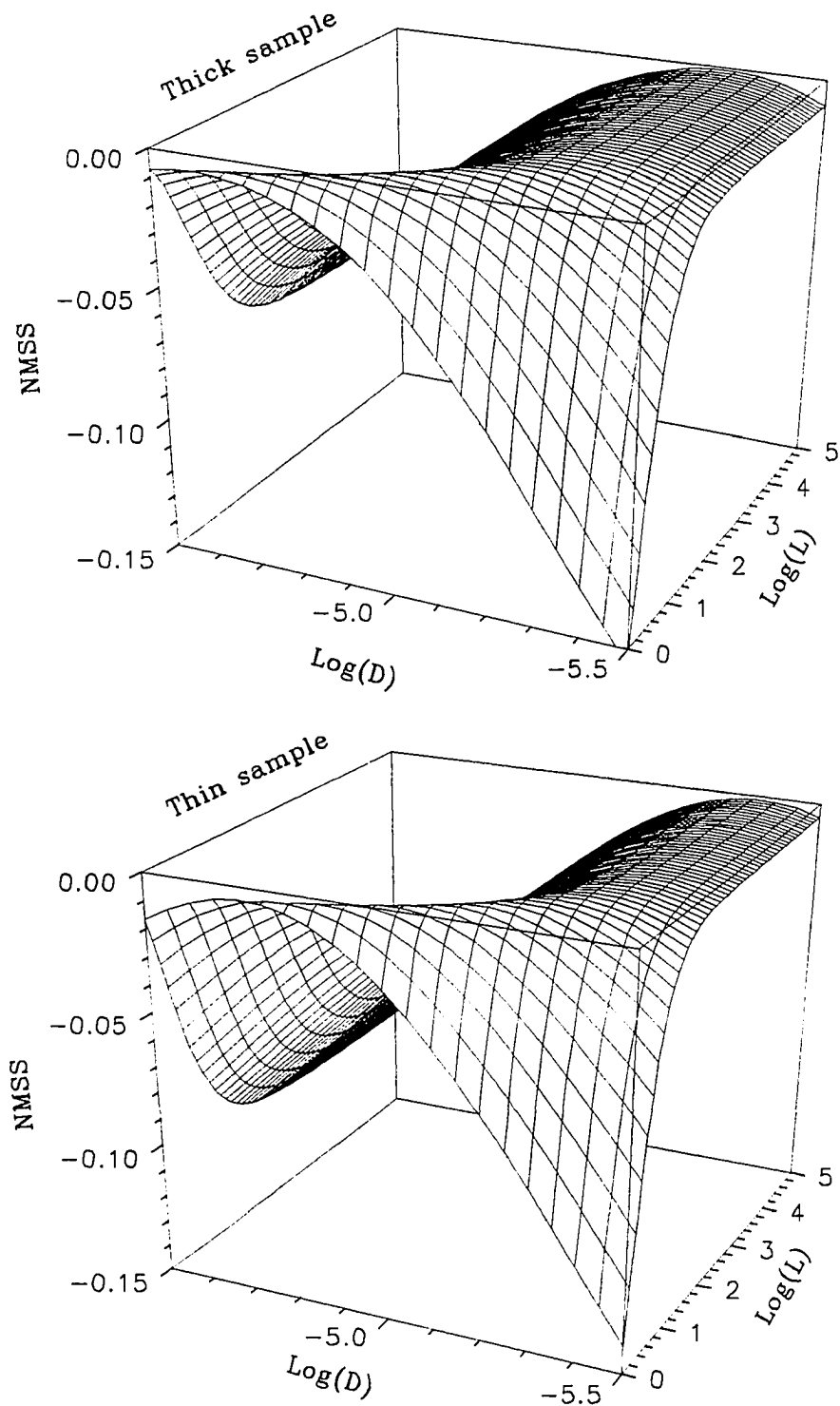


Figure 3.1. NMSS response surface as a function of $\text{Log}(D)$ and $\text{Log}(L)$ (Data from Choong and Skaar, 1969).

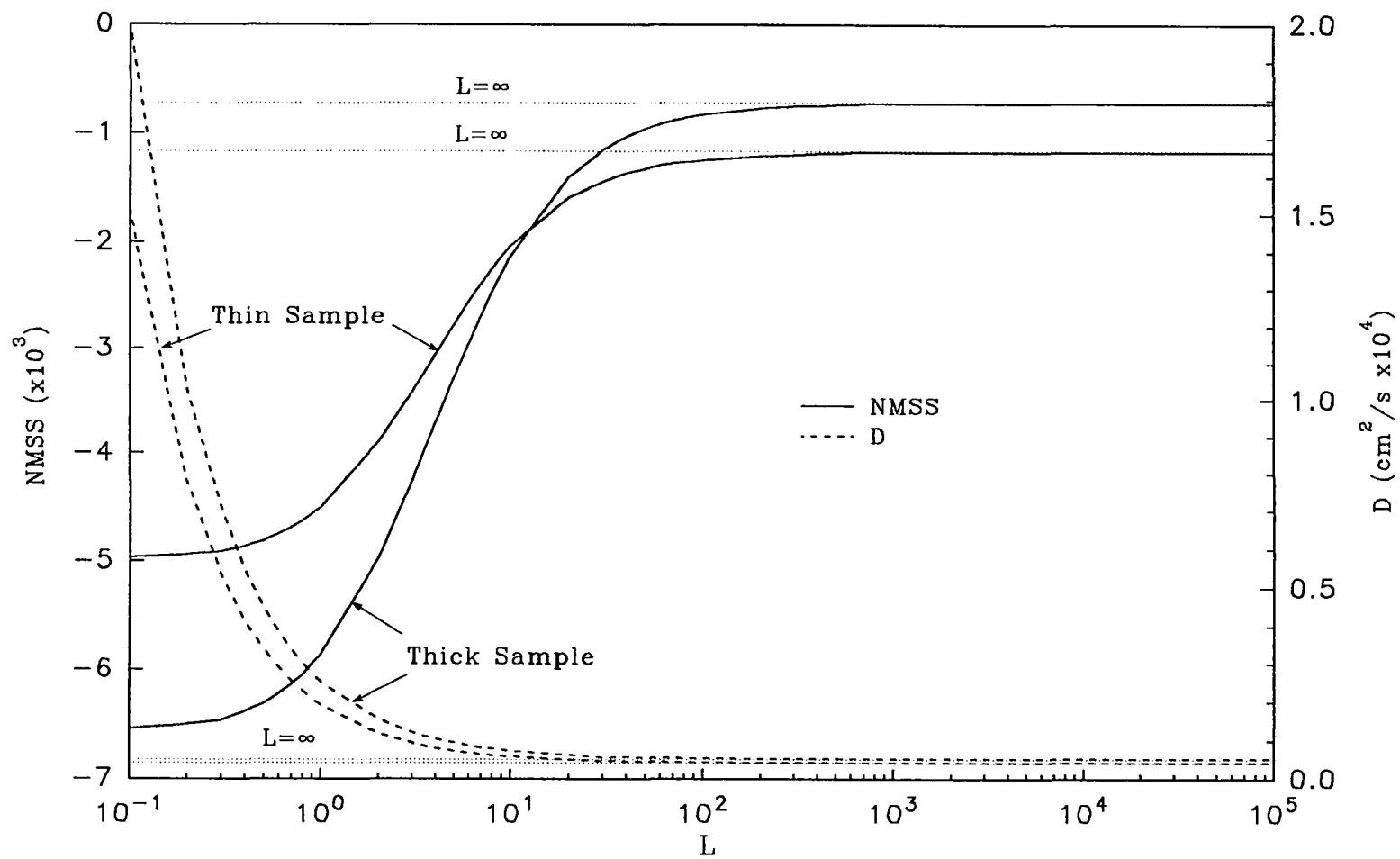


Figure 3.2. Variation of NMSS on the ridge of response surface as a function of D or L (Data from Choong and Skaar, 1969).

stopped because it had gone into the insensitive region where L was large, showing no further change in D or NMSS as L approached infinity.

Table 3.1. Comparison of values^a of L , S , D , and NMSS from the optimization technique with those from two approximate methods

Method	Half-thickness cm	L	S cm/sec	D cm ² /sec	NMSS data
Optimization technique	0.479	∞	∞	4.28N6	-1.16N3
	1.431	∞	∞	5.25N6	-7.22N4
.....					
Approximate method 1 ^b	0.479	9.7	1.52N4	7.50N6	-4.69N3
	1.431	29.1	1.52N4	7.50N6	-2.78N3
.....					
Approximate method 2 ^c	0.479	11.1	1.66N4	7.14N6	-4.25N3
	1.431	28.9	1.48N4	7.35N6	-2.47N3

^a The notation xNy means $x \times 10^y$.

^b Values of L , S , D are from Choong and Skaar (1969).

^c Values of L , S , D are from Liu (1989).

The application of the optimization technique significantly enhanced the predictability of the experimental data. The solid \bar{E} curves (Figure 3.3) are those which correspond to the optimum S and D , according to the minimum absolute NMSS criterion. The absolute values of the NMSS by the two approximate methods reported in Table 3.1 (Choong and Skaar 1969, Liu 1989) were more than 3.4 and 3.6 times larger than those resulting from the optimization technique for the

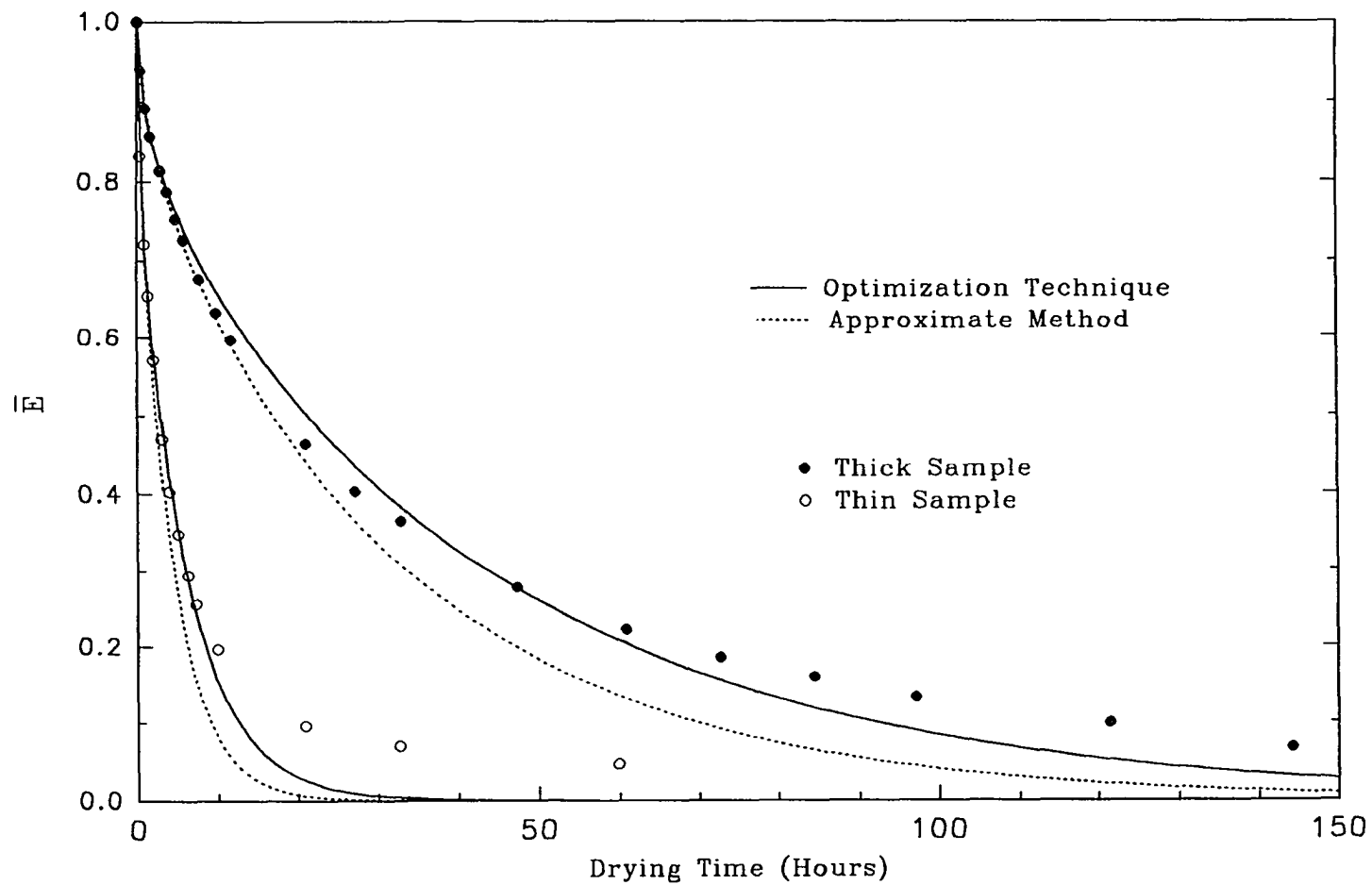


Figure 3.3. Comparison of calculated \bar{E} curves from the optimization technique with those from the approximate method based on two sample thicknesses (Data from Choong and Skaar, 1969).

thick and thin samples, respectively. Since they are based on data at $\bar{E}=0.5$, the approximate techniques fit well at that point, but poorly elsewhere. As can be seen in Figure 3.3, the dashed lines representing the approximate technique seriously underestimate the drying time necessary to reach low \bar{E} values.

The relationship of the optimum \bar{E} curves to the experimental data (Figure 3.3) indicates the diffusion coefficient was a function of MC. For both thick and thin samples, the experimental \bar{E} data are located below the optimum \bar{E} curve in the higher MC range and above it in the lower MC range, suggesting the diffusion coefficient decreased with the decrease of MC. This is consistent with the findings in the literature (Comstock 1963, Choong 1965).

The diffusion coefficient may depend on the sample thickness. The optimum D for the thin sample is 18 percent smaller than the thick sample (Table 3.1). A similar result was reported by Choong and Skaar (1969, 1972), who explained that the discrepancy in the "apparent" diffusion coefficients was due to a change in the external resistances of samples of different thicknesses. However, their air speed was 3.3 m/s, which is greater than the threshold value 3.0 m/s (Rosen 1978), above which the external resistance is negligible. Therefore, other factors, such as a time-dependent deformation of the cellulose-lignin matrix (Hart 1964) and drying stress, could contribute to this discrepancy.

CONCLUSIONS

- 1) The optimum values of the diffusion coefficient and the surface emission coefficient can be determined from a single drying curve using the optimization technique.
- 2) The drying curves calculated by the optimization technique gave a more accurate prediction to the experimental data than those by the existing approximate methods.
- 3) The diffusion coefficient determined by the optimization technique was the optimum average value over the entire drying period.
- 4) The diffusion coefficient was not constant, but a function of moisture content (MC). For the typical drying case studied, it had a relatively large value at the high MC, and a relatively small value at the low MC.

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CHAPTER IV

A NUMERICAL ANALYSIS TECHNIQUE TO EVALUATE THE MOISTURE-DEPENDENT DIFFUSION COEFFICIENT ON MOISTURE MOVEMENT DURING DRYING

INTRODUCTION

The diffusion model based on Fick's second law has been used to predict the removal of moisture from wood, since diffusion controls the movement during drying (Stamm 1964). It has been demonstrated in Chapter III that an accurate prediction of the drying curves can not be attained even though the optimum average (constant) values of the diffusion coefficient and the surface emission coefficient have been applied. When a constant diffusion coefficient is assumed, theoretical solutions to the diffusion model are readily available under simple boundary conditions, e.g., letting the surface moisture content (MC) drop immediately to equilibrium with the surroundings, or assuming a constant surface emission coefficient (Skaar 1954). However, as pointed out by many researchers (Moschler and Martin 1968, Bui *et al.* 1980, Steven *et al.* 1985), these simple assumptions usually do not hold; the diffusion coefficient is generally not a constant, and the surface MC changes gradually toward equilibrium with the surroundings. An accurate prediction of the distribution of moisture within wood cannot be achieved without considering the effect of wood MC on the diffusion coefficient.

When the diffusion coefficient is a function of MC, there is no closed form of theoretical solutions to the governing partial differential equation based on Fick's second law. If a constant diffusion coefficient and functional boundary conditions are assumed, the theoretical solutions expressed as

infinite Fourier series may exist, but are usually quite complicated (Crank 1975, Carslaw and Jaeger 1986). Therefore, in both cases, numerical analysis techniques are the most practical to evaluate the diffusion coefficient.

In the past, the moisture dependence of the diffusion coefficient has been evaluated based on the MC profile data obtained by the slicing technique. Moschler and Martin (1968) have given several polynomial relationships between the diffusion coefficient and MC at 40 °C for yellow poplar. These relationships were derived from MC profile data at specific times during the drying process. They could obtain a better prediction of the MC profiles, when substituting the derived expression into the diffusion equation, than by using other available diffusion coefficients from the literature. The best prediction could only be achieved when the samples were examined for a relatively long drying time. They attributed the discrepancy to several factors: the violation of initial and boundary conditions, experimental error caused by using the average MC distribution, lack of homogeneity on the macro or micro scale in wood, and the time-dependent, stress-related factor.

Stevens et al. (1985) presented a similar study on the relationship of the diffusion coefficient to MC at 32 °C for yellow poplar. Their empirical equation is in an exponential form with three adjustable parameters. The initial condition was obtained from the MC gradient after the samples had been

dried for a period of time to allow the surface MC to drop close to equilibrium with the surroundings. They reported that the experimentally determined relationship between the diffusion coefficient and MC could predict moisture gradients fairly accurately during isothermal nonsteady-state moisture movement, and the relationship between the diffusion coefficient and MC did not change over the course of desorption.

In the above studies, obtaining accurate data of MC profiles is essential for the calculation, which requires specific tools and laboratory techniques. Since only the average values of MC profiles are measured, an inherent error is introduced into the calculation. Inhomogeneity among samples may lead to inaccuracy, since variation in MC profiles among samples is confounded with the calculated parameters used to relate the diffusion coefficient to MC.

In order to reduce the experimental effort and eliminate the inherent error accompanied by the slicing technique, an alternate approach to evaluating the moisture-dependent diffusion coefficient using drying curves is suggested. This approach is based on the idea that the drying process is diffusion-controlled and the diffusion coefficient is moisture-dependent. Therefore, the diffusion model should satisfactorily predict the experimental drying curve, if a functional relationship between the diffusion coefficient and the MC, as well as the boundary condition of the model, is

appropriately defined. In this study, a linear relationship between the diffusion coefficient and MC is assumed because of its simplicity and informativeness. An exponential boundary condition is used to account for the fact that the surface equilibrium condition is not established instantaneously. This choice of the boundary condition is justified by Moschler and Martin's (1968) experimental data and Liu and Cheng's (1989) theoretical analysis.

The objective of this study was to determine if and to what extent the moisture-dependent diffusion coefficient and the change of surface moisture content affect the predictability of moisture removal during drying. The hypothesis is that, using a numerical analysis technique, a close prediction of the experimental drying curves could be achieved if the effect of the variation of the diffusion coefficient and surface moisture content was considered when solving the diffusion equation.

NUMERICAL ANALYSIS TECHNIQUE

The diffusion equation used in the present study was the common form of Fick's second law, which has been applied by many researchers (Moschler and Martin 1968, Rosen 1976, Stevens *et al.* 1985)

$$\frac{\partial m}{\partial t} = \frac{\partial}{\partial x} \left(D \frac{\partial m}{\partial x} \right) \quad (4.1)$$

where m is $MC/100$, t is drying time, x is the distance coordinate, and D is the diffusion coefficient, which is generally a function of m .

Several finite difference schemes can be used to solve Eq. 4.1 under given initial and boundary conditions. The classic explicit approximation, which has been used by previous researchers (Moschler and Martin 1968, Stevens *et al.* 1985), is simple and easily programmable, but the stability requirement must be strictly satisfied in order to obtain a convergent solution. In addition, it contains a relatively large truncation error of the time grid, of the order of Δt , or $O(\Delta t)$. To avoid the limitations of the classic explicit approximation, a linear three-level finite difference scheme was applied. This scheme is an implicit approximation, which is unconditionally stable, convergent, and with a smaller truncation error in the time grid, of $O(\Delta t)^2$.

The three-level finite difference scheme can be stated as follows (Lapidus and Pinder 1982):

$$\begin{aligned} (m_{i+1,j} - m_{i-1,j}) = \frac{2}{3}\rho \{ D_1 [(m_{i+1,j+1} - m_{i+1,j}) + (m_{i,j+1} - m_{i,j}) \\ + (m_{i-1,j+1} - m_{i-1,j})] - D_2 [(m_{i+1,j} - m_{i+1,j-1}) \\ + (m_{i,j} - m_{i,j-1}) + (m_{i-1,j} - m_{i-1,j-1})] \} \end{aligned} \quad (4.2)$$

where $\rho = \Delta t / (\Delta x)^2$, and i and j are time and space indices, respectively. In this study, the Δt and Δx were 0.01 hour and one-tenth of the sample half-thickness.

With the assumption of a linear relationship between D and m , D_1 and D_2 can be calculated according to the following equations:

$$D_1 = a_0 + a_1[(m_{i,j+1} + m_{i,j})/2] \quad (4.3)$$

$$D_2 = a_0 + a_1[(m_{i,j} + m_{i,j-1})/2] \quad (4.4)$$

where a_0 and a_1 are constant.

For the surface MC, m_s , an exponential decay from the initial MC to equilibrium with the surroundings was assumed, i.e.,

$$m_s = m_e + (m_i - m_e) \exp(-\beta t) \quad (4.5)$$

where m_i and m_e are the initial and equilibrium MC (on a fractional basis), and β is a surface parameter to account for the changing rate of surface MC.

The only restriction in the above equations is that the diffusion coefficient D should be greater than zero.

At the beginning of the calculation, the MC values on the first level are determined according to the initial condition. The values on the second level can be calculated according to the following equation:

$$(m_{i+1,j} - m_{i,j}) = \rho [D_1(m_{i+1,j+1} - m_{i+1,j}) - D_2(m_{i+1,j} - m_{i+1,j-1})] \quad (4.6)$$

When a set of parameter values in Eqs. 4.3, 4.4, and 4.5 are chosen, a theoretical drying curve can be generated. Each time the average MC of the sample is measured experimentally, the theoretical MC is calculated by applying Simpson's rule. The criterion for the optimum parameter values is that they

yield the minimum absolute value of *Negative Mean Sum of Squares* (NMSS), defined as $-\Sigma(\Delta m)^2/N$. Here Δm and N represent the difference in m between the theoretical drying curve and the experimental data, and the number of data points, respectively. The above definition of the NMSS is applicable to multi-drying curves as well. FORTRAN codes have been developed to facilitate the calculation of the NMSS for either a single drying curve or multi-drying curves. Several multivariable search methods (Pike 1986) may be applied to help find the optimum values of a_0 , a_1 , and β .

NUMERICAL EXAMPLES AND DISCUSSION

Four cases were examined, based on the following assumptions:

- Case 1. The diffusion coefficient is constant, and the surface MC drops immediately to equilibrium with the surroundings at the beginning of drying.
- Case 2. The diffusion coefficient is a linear function of moisture content, and the surface MC drops immediately to equilibrium with the surroundings at the beginning of drying.
- Case 3. The diffusion coefficient is constant, but the surface MC decays exponentially to equilibrium with the surroundings.

Case 4. The diffusion coefficient is a linear function of moisture content, and the surface MC decays exponentially to equilibrium with the surroundings.

In order to avoid frequent statements of the properties, the cases will be referred to by numbers unless mentioned otherwise. For convenience, the term "equilibrium boundary condition" refers to the condition when the surface MC drops immediately to equilibrium with the surroundings, and the term "exponential boundary condition" is defined by Eq. 4.5 where the surface MC decays exponentially to equilibrium with the surroundings.

DRYING BELOW THE FIBER SATURATION POINT (FSP)

Choong and Skaar (1969) evaluated the diffusion coefficient and the surface emission coefficient using their approximate method for yellow-poplar sapwood and heartwood specimens, dried in either the tangential or the radial direction. Two samples of different thicknesses were dried for each combination in an environmental chamber at a temperature of 32 ± 0.2 °C and 40 percent relative humidity (RH) with an air speed of 3.3 m/s. In Chapter III, it has been shown that, if the diffusion coefficient were treated as a constant, a close prediction to the drying data for yellow-poplar sapwood in the tangential direction over the entire drying period could not be achieved. For the convenience of

comparison, the same data were also used to analyze the performance of the current numerical analysis technique. In Choong and Skaar's experiment, the initial MC of the samples was about 25 percent. The equilibrium moisture content (EMC) was not given. Only the environmental conditions of the chamber were reported. From the relationship of EMC-temperature-relative humidity given in the Wood Handbook (U.S. Department of Agriculture 1987), the EMC was predicted to be 7.4 percent. The above initial MC and final EMC were assumed to be the same for both the thick sample, with a half-thickness $a=1.431$ cm, and the thin sample, with $a=0.479$ cm. Uniform initial MC and equilibrium surface MC with the surroundings were assumed to initiate the calculation.

In this example, only the results of Cases 1 and 2 are reported. This is because no optimum β values, as defined in Eq. 4.5, were found in Cases 3 and 4. With each increase in β , the absolute NMSS always decreased, although only to a small degree. The assumption that the surface MC drops immediately to equilibrium with the surroundings is valid because the final β values are large ($\beta > 1.5 \times 10^{-2}$ 1/s). Since β represents the external resistance to the moisture movement, an increase in air speed results in an increase in β . In the experiment, the air speed was relatively large (3.3 m/s) and greater than the threshold value 3.0 m/s (Rosen 1978), above which the external resistance is negligible.

The values of D and NMSS of Cases 1 and 2 are summarized in Table 4.1. To calculate the constant diffusion coefficient, a_1 in Eqs. 4.3 and 4.4 was set to zero with the boundary condition (Eq. 4.5) reduced to $m_g=m_e$ by setting β equal to ∞ . The optimum average diffusion coefficient with a minimum absolute value of the NMSS was searched. Its value for either the thick or the thin sample is practically the same as that obtained from the theoretical solution (Chapter III), indicating that this numerical technique is accurate and reliable. To find the linear relationship between D and MC, the optimum a_0 and a_1 in Eqs. 4.3 and 4.4 were searched.

Table 4.1. Summary of the values^a of D and NMSS for yellow-poplar sapwood^b

Sample type	Relation type	D ($\times 10^6 \text{cm}^2/\text{s}$)	NMSS
Thick ($a=1.431 \text{ cm}$)	Constant	5.24 (5.25) ^c	-2.20N5
	Linear	-2.476+0.6246(MC)	-2.45N6
.....			
Thin ($a=0.479 \text{ cm}$)	Constant	4.26 (4.28) ^c	-3.54N5
	Linear	-4.610+0.7082(MC)	-5.90N6

^a The notation xNy means $x \times 10^y$.

^b Data used for the calculation are from Choong and Skaar (1969).

^c The value in the bracket is obtained from a theoretical approach (Chapter III).

As illustrated in Figure 4.1, the application of the linear model enhances the predictability of the drying curves significantly. Compared with that of the constant diffusion

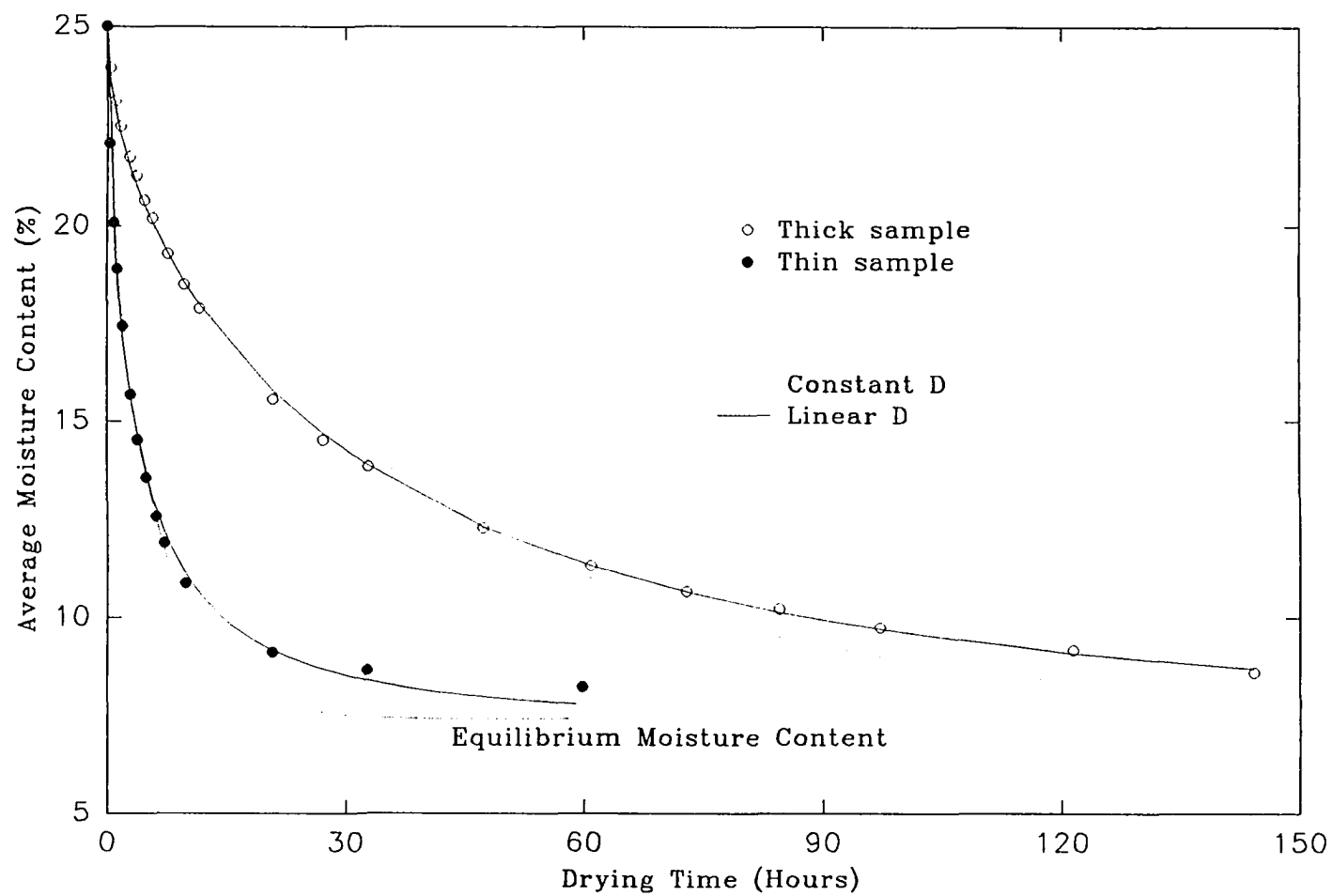


Figure 4.1. Predicted drying curves derived from constant diffusion coefficient and linear diffusion coefficient as a function of moisture content.

coefficient, the absolute value of the NMSS for either the thick or the thin sample decreases by one order of magnitude when the linear model is applied. This demonstrates that the diffusion coefficient is a function of the MC.

Sample thickness may affect the diffusion coefficient. The optimum constant diffusion coefficient for the thin sample is 18 percent smaller than that of the thick sample. As illustrated in Figure 4.2, the linear functions have different intercepts and slopes for the thick and thin samples. The intercept of the thin sample (Table 4.1) is about twice that of the thick sample. Choong and Skaar (1969, 1972) reported a similar result, explaining that the discrepancy in the "apparent" diffusion coefficients was due to differences in the external resistances. The discrepancy in the present study may not be explained by the external resistance alone, since the external resistance was found to be negligible. Other factors, such as a time-dependent deformation of the cellulose-lignin matrix (Hart 1964) and drying stress could, also be significant.

DRYING FROM ABOVE THE FIBER SATURATION POINT (FSP) TO BELOW THE FSP

Data from Chapter VI for the control samples of redwood (*Sequoia sempervirens*) and red oak (*Quercus* sp.) heartwood were used to study the moisture-dependent diffusion coefficient together with an exponential boundary condition.

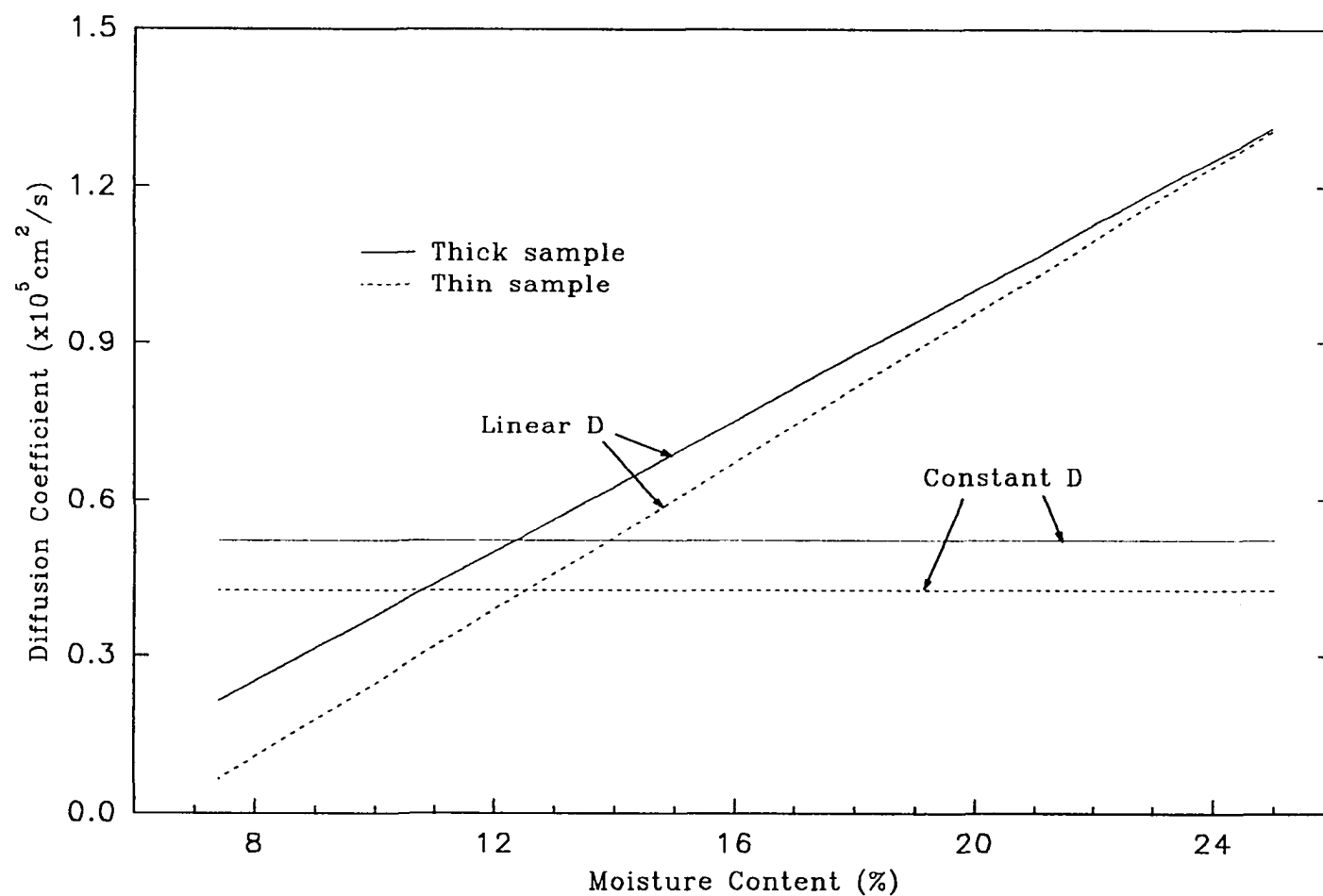


Figure 4.2. Comparison of constant diffusion coefficient and linear diffusion coefficient as a function of moisture content.

In these experiments, the samples were dried in an AMINCO environmental chamber from near saturation to 6 ± 1 percent MC (45 ± 0.5 °C, 30 percent RH) with an air speed of 2.0 m/s. The nominal half-thickness of the samples in the radial direction was $a = 0.75$ cm. Experimental data from three samples were used for each species. The initial and equilibrium MCs were determined from the average value of the three samples.

Table 4.2. Summary of the values^a of β , D , and NMSS for redwood heartwood and red oak heartwood^b

Species	Case	β ($\times 10^4$ 1/s)	D ($\times 10^6$ cm ² /s)	NMSS
Redwood	1	∞	11.78	-6.03N2
	2	∞	21.07-0.078(MC)	-5.69N2
	3	1.28	35.46	-5.09N3
	4	1.13	68.18-0.178(MC)	-4.41N3
.....				
Red oak	1	∞	6.93	-1.16N3
	2	∞	9.41-0.070(MC)	-1.05N3
	3	3.28	8.21	-7.64N4
	4	2.56	6.82+0.051(MC)	-7.45N4

^a The notation xNy means $x \times 10^y$.

^b Data used for the calculation are from Chapter VI.

The predicted drying curves, resulting from the optimization of the four cases, are shown in Figure 4.3 for redwood and in Figure 4.4 for red oak. The results of the optimization search are summarized in Table 4.2. As expected, the best prediction with the smallest absolute values of the NMSS was achieved by the conditions assumed in Case 4. However, almost the same accuracy could be achieved in Case 3.

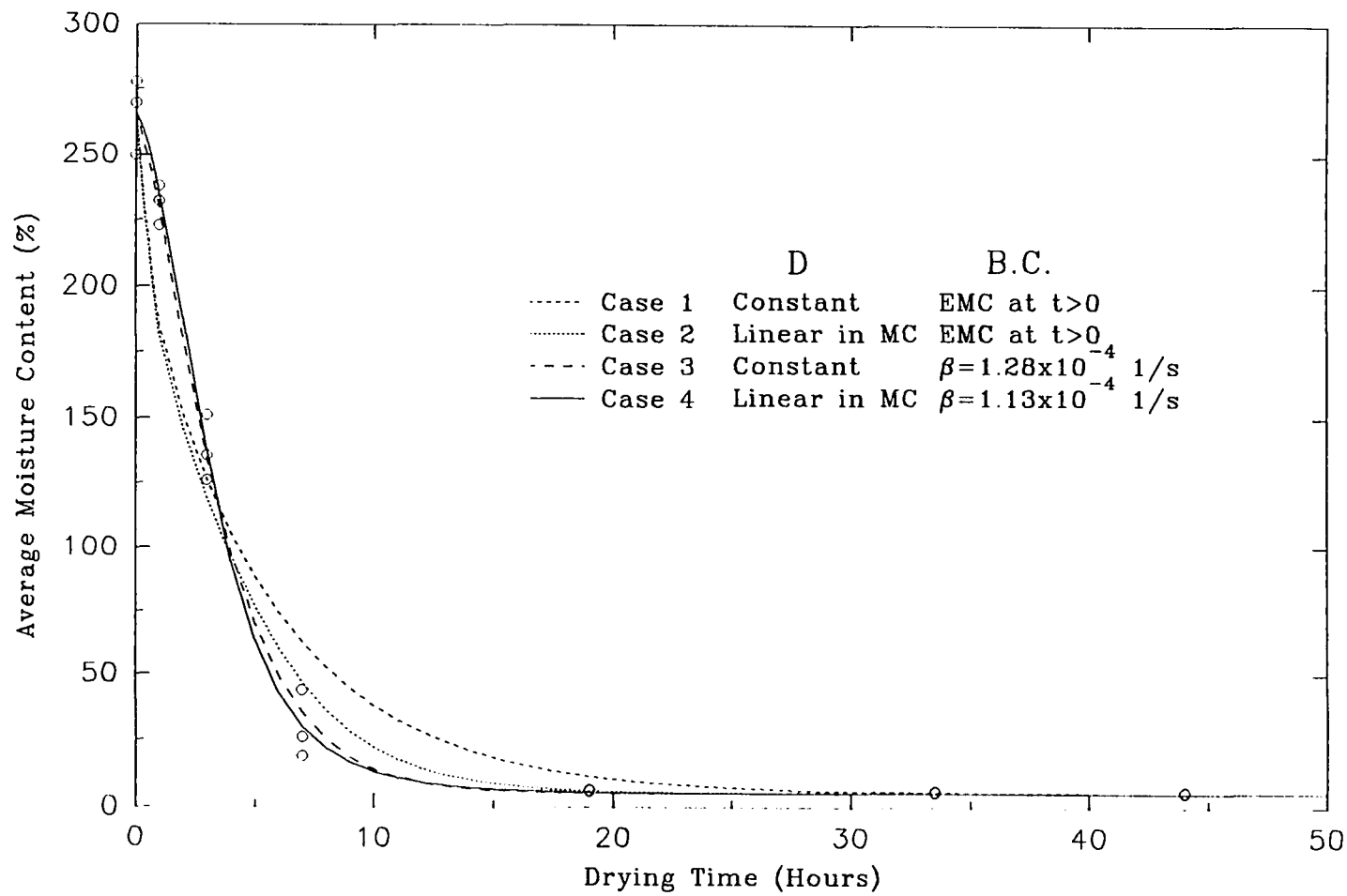


Figure 4.3. Comparison of drying curves of four cases for redwood heartwood.

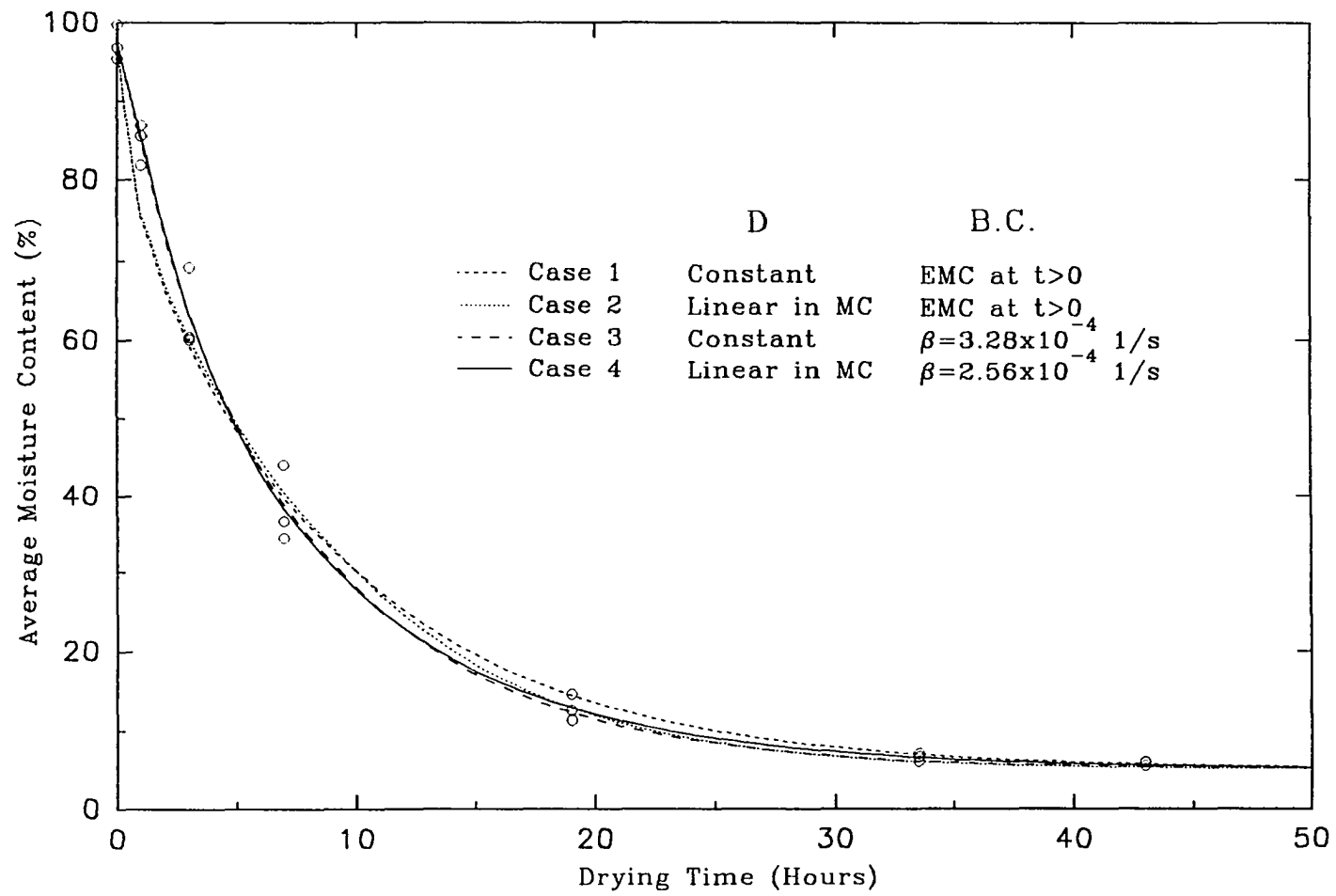


Figure 4.4. Comparison of drying curves of four cases for red oak heartwood.

The relative magnitude of the NMSS values indicates that the enhancement of the prediction on drying curves is mainly due to using the exponential boundary, rather than the equilibrium boundary, condition. For instance, while the value of NMSS of Case 3 for redwood heartwood is 1.2 times greater than that of Case 4, the values of Cases 1 and 2 are 13.7 and 12.9 times larger, respectively. For red oak heartwood, the enhancement of the prediction is relatively small.

In this example, the diffusion coefficient can be considered a constant. Even though the calculated diffusion coefficient of Case 4 changes with MC, its use in the diffusion model does not result in significant improvement in the predictability of drying curves. The removal of moisture above the FSP is controlled by moisture diffusion at the wood surface. Based on the kinetic theory, Stamm (1964) explained that moisture diffuses through the cell wall by molecular jumps from one sorption site to another. When wood shrinks, the total number of sorption sites decreases; therefore, the diffusion coefficient decreases. However, a constant diffusion coefficient can prevail when the average moisture content is above the FSP. As drying progresses, the surface layers of the wood are the first to dry below the FSP, but are restrained from shrinking fully because the inner part of wood, which is still above the FSP, lags in shrinking. Consequently, the wood in the surface layers is stretched. Barkas (1949) reported an increase in EMC when wood is

subjected to tension stress, which increases the sorption sites for bound water. The net effect of tension stress and shrinkage results is that the number of sorption sites remains about the same, with the diffusion coefficient staying constant.

CONCLUSIONS

- 1) The parameters in the linear function of D to MC under an exponential boundary condition can generally be determined from drying curves using the optimization technique.
- 2) The predictability of moisture removal during drying can be improved by using the moisture-dependent diffusion coefficient or the exponential boundary condition. Their relative importance depended on the initial moisture content of the wood and drying conditions.

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CHAPTER V

EFFECT OF STEAMING AND HOT-WATER SOAKING ON THE MOVEMENT OF MOISTURE IN SOUTHERN HARDWOODS DURING DRYING

INTRODUCTION

A common goal of research in wood drying is to reduce the long drying times involved in wood processing without unacceptable degradation. The attempt to advance drying technology by optimizing process variables such as temperature, relative humidity, and air velocity has not been effective in most hardwoods, which are considered refractory or difficult to dry. To attain this goal, the blockage of the moisture passageways within wood must be minimized.

Steaming is one of several predrying treatment techniques which has been used to open the moisture passageways and increase the drying rate of wood. Steaming has been reported by Mackay (1971) to increase the diffusivity of two Australian hardwoods, by 10 to 12 percent in the green condition, and by 30 to 40 percent at 22 percent moisture content. Steaming also has been reported by several researchers to decrease drying time (Ellwood and Erickson 1962, Kininmonth 1973) and increase drying rate (Sharma and Bali 1969, Simpson 1975, Alexion *et al.* 1990) in wood. However, the effect of steaming on the drying rate depends on species. Simpson (1975) reported that among four species investigated, oaks responded best. In some, the drying rate increased more than 50 percent.

Removal or redistribution of water-soluble extractives has been considered among the likely reasons for the increase in drying rate. Kininmonth (1971) showed that steaming

changed the continuous layer of extractives lining the cell lumina and pit membrane in a New Zealand hardwood (*Nothofagus fusca*) into a generally discontinuous layer, which appeared as cracked and blistered. Alexion et al. (1990) described a similar effect of steaming on regrown *Eucalyptus*. Chen and Workman (1980) reported that steaming black walnut heartwood partially reduced its extractives content, since the condensed water always showed a black color. Simpson (1975) reported that while steaming sweetgum heartwood resulted in an increase in the drying rate by about 20 to 30 percent, steaming sweetgum sapwood had no beneficial effect. Sweetgum heartwood, which has a considerable amount of polymeric phenolic glycosides, is relatively impermeable (Rowe and Conner 1979), and steaming may remove some of the polymers.

Simpson (1975) has pointed out certain precautions to take when the effects of steaming are examined. The temperature of the steamed samples is usually higher than that of the unsteamed ones, and the moisture contents of the steamed and unsteamed samples may be different before drying is begun. Therefore, for the purpose of comparison, the experiment must be designed to minimize the above variables.

Hot-water soaking is a direct method of removing the water-soluble extractives from wood. Like steaming treatment, hot-water soaking also partially hydrolyzes some of the cell wall substances, such as hemicelluloses (Rowe and Conner 1979). Hot-water extraction increases not only the

permeability of southern pine (Fogg 1968), but also the shrinkage and equilibrium moisture content of domestic and tropical woods (Nearn 1955, Choong and Achmadi 1991). There is, however, no published data in the literature describing the effect of hot-water soaking on wood drying.

The objective of this study was to determine if and to what extent steaming and hot-water soaking treatments enhance the movement of moisture in certain southern hardwood species above, as well as below, the fiber saturation point (FSP).

EXPERIMENTAL PLAN AND PROCEDURES

STUDY PLAN

The heartwood of six species, namely red oak (*Quercus* sp.), white oak (*Quercus* sp.), elm (*Ulmus americana*), sweetgum (*Liquidambar styraciflua*), willow (*Salix nigra*), and cottonwood (*Populus* sp.) was selected for this study. These woods were chosen for their structure (ring- and diffuse-porous hardwoods), commercial importance, and relatively slow drying rate. Movement of moisture during drying was restricted to one of the three structural directions of wood, i.e., longitudinal, tangential, or radial. Five treatments were:

- A) Control,
- B) Steaming in the green condition for 1 hour at 100 °C,
- C) Steaming in the green condition for 5 hours at 100 °C,
- D) Hot-water soaking for 10 hours at 70 °C, and

E) Steaming near the FSP for 1 hour at 100 °C.

The samples were dried in two phases: Phase 1 -- Drying in the Free Water Range, from near saturated condition to near the FSP, and Phase 2 -- Drying in the Hygroscopic Range, from near the FSP to the final equilibrium moisture content (EMC). Samples undergoing Treatment E were dried only in the hygroscopic range, since their MCs were already near the FSP. A completely randomized design with two replications was applied in this study.

PROCEDURE

All sample boards were obtained in the green condition from the Plaquemine Hardwood Lumber Company in Plaquemine, Louisiana. For each species, two flat-sawn boards containing heartwood about 5 cm thick and 20 cm wide were selected. They were cut into 2-meter long sections, wrapped with Visqueen plastic sheet, and then stored in a cool room.

From each board, two slabs 2.0 cm thick in the structural (longitudinal, tangential, radial) direction being measured were prepared. To ensure that the movement of moisture followed true structural direction, an adjusting miter gauge was used to define the cutting direction. Each sample had nominal dimensions of 2.5cm × 2.5cm × 2.0cm, and was marked according to a specified numbering system. There were altogether 180 samples.

After cutting, the samples were submerged in cold water to keep them in the green condition. For the steaming treatments, the samples were randomly chosen from each corresponding group and placed inside a small pressure retort. High pressure steam generated in an electric steam boiler was continuously released into the retort. The hot-water soaking was done in a thermostatically-controlled water container equipped with a stirrer.

After treatment, all samples, except those for Treatment E, were subjected to periodic vacuum at 56-cm Hg for one hour under water, then released to atmospheric pressure. This process was carried out three times per day, and the interval from the end of pressure recovery to the beginning of the next vacuum was at least two hours. After 10 days, only a few bubbles could be observed on the samples under vacuum; therefore, they were considered saturated. The samples were then coated with Dow's Saran F-120 resin on the four 2.5cm x 2.0cm faces to provide a uni-directional movement of moisture. After coating, the samples were again submerged in water inside desiccators to keep them saturated.

In Phase 1 (Drying Above the FSP), an AMINCO environmental chamber was adjusted to 45 °C and 97 percent relative humidity (RH), corresponding to a nominal 25 percent EMC. The air velocity in the drying chamber was maintained at about 2 meters per second. The samples were divided into two subgroups because the AMINCO chamber was not large enough to

accommodate all the samples at once, and a considerable amount of time would be needed to measure all samples at one time. After the first sample group had been dried to near the FSP, the samples in the first subgroup were then taken from the AMINCO chamber to a BLUE-M chamber set to the same conditions, so that the samples in the second subgroup could be placed in the AMINCO chamber. Thus, the BLUE-M was used to keep the samples in a "holding" condition of about 25 percent EMC until they were ready for Phase 2 Drying. After the samples in the second subgroup had been dried to near the FSP, the samples for Treatment E were steamed in the pressure retort before they were coated with Dow's Saran F-120 resin.

In Phase 2 (Drying Below the FSP), the drying conditions in the AMINCO chamber were adjusted to 45 °C and 30 percent RH, corresponding to a nominal 6 percent EMC. After the samples had been dried to the final EMC, they were oven-dried in a Supermate oven at 103 °C for three days, and their moisture contents (MC's) were calculated based on their oven-dry weights.

For each sample, the values of \bar{E} (the fraction of evaporable moisture present in wood) were determined from the MC data. These were then used to calculate the average diffusion coefficients by the optimization method described in Chapter II. A general linear model was used to evaluate the effects of treatments, species, and directions on moisture

movement. Statistical analysis was made for each of the two phases of drying separately.

RESULTS AND DISCUSSION

PHASE 1 (DRYING ABOVE THE FSP)

The average diffusion coefficient in each direction-treatment-species combination is given in Table 5.1. An analysis of variance for the diffusion coefficients (Table 5.2) shows that the treatment effect was significant, but that it was not as large as the effect of direction or species. Since the F value of the treatments is one order higher than that of treatment-species interaction, the treatment effect was generally the same for all directions and species. The result of pair-wise t-tests (LSD) for treatments (Table 5.3) indicates that all the predrying treatments increased the diffusion coefficients. Treatment C and Treatment D do not show any difference between themselves, but they were more effective than Treatment B.

Steaming before drying should open the passageways for capillary movement in wood. Since the samples were soaked in water after steaming, there were no confounded effects due to differences in initial temperature and moisture content. The effect of steaming is mainly due to the redistribution of blocking substances (without their removal) from wood, and by the distortion of materials lining the cell lumina and pits areas (Kininmonth 1971, Kubinsky 1971, Alexion et al. 1990).

Table 5.1. Average diffusion coefficients^a of six hardwoods for Phase 1 (Drying Above the FSP)

Direction	Treatment	Species					Cottonwood
		Red oak	White oak	Elm	Sweetgum	Willow	
		----- x10 ⁻⁶ cm ² /s -----					
Longitudinal	A	0.522	0.445	0.407	0.438	0.332	0.437
	B	0.528	0.454	0.409	0.448	0.341	0.455
	C	0.556	0.477	0.439	0.482	0.356	0.502
	D	0.598	0.480	0.417	0.477	0.362	0.499
.....							
Radial	A	0.388	0.324	0.346	0.401	0.273	0.423
	B	0.466	0.346	0.353	0.417	0.281	0.431
	C	0.510	0.371	0.363	0.425	0.319	0.463
	D	0.502	0.377	0.366	0.436	0.299	0.468
.....							
Tangential	A	0.378	0.340	0.314	0.391	0.293	0.411
	B	0.442	0.367	0.340	0.418	0.312	0.418
	C	0.458	0.379	0.351	0.433	0.330	0.470
	D	0.490	0.377	0.364	0.439	0.334	0.463

^a Each value represented the mean of two replicates.

The process of rearranging the blocking substances takes time. Steaming for 1 hour for the size of the samples in this study may not be long enough to fully open the passageways.

Table 5.2. Analysis of variance of average diffusion coefficients for Phase 1 (Drying Above the FSP)

Source	DF	MS	F	P
DIR	2	6.507×10^{-2}	224.07	0.001
TREAT	3	1.939×10^{-2}	66.78	0.001
SPECIES	5	8.729×10^{-2}	300.58	0.001
CHM ^a	1	6.385×10^{-4}	2.20	0.143
DIRxTREAT	6	2.797×10^{-4}	0.96	0.456
DIRxSPECIES	10	3.073×10^{-3}	10.58	0.001
TREATxSPECIES	15	8.627×10^{-4}	2.97	0.001
DIRxTREATxSPECIES	30	2.258×10^{-4}	0.78	0.775
Error	71	2.904×10^{-4}	--	--

^a Batch effect of drying samples at different time in the AMINCO chamber.

Table 5.3. Comparison of the effect of treatments on average diffusion coefficients by pair-wise t-tests (LSD) at 0.05 significance level in Phase 1 (Drying Above the FSP)

Treatment	D	C	B	A
Mean ($\times 10^6 \text{cm}^2/\text{s}$)	<u>0.430</u>	<u>0.427</u>	0.401	0.381

Partially removing the hot-water soluble extractives and effectively rearranging them in the wood during the relatively long treating period are two possible explanations for the better performance of Treatment D than Treatment B. Since the samples came in direct contact with hot-water, the water-

soluble extractives could diffuse into the surrounding water under a concentration gradient. However, not all the hot-water soluble extractives could be extracted out of the samples by soaking in circulating hot water for 10 hours; therefore extractives redistribution also contributed to the increase in the diffusion coefficients. Some species showed better response to hot-water soaking than steaming. As shown in Figure 5.1, the average diffusion coefficient of hot-water soaking for red oak was higher than that for steaming. This also accounts for the significant effect of treatment-species interaction (Table 5.2).

PHASE 2 (DRYING BELOW THE FSP)

The average diffusion coefficients in each direction, treatment, and species combination are given in Table 5.4. An analysis of variance for the diffusion coefficients (Table 5.5) shows that the treatment effect was significant, although it was not as large as the effect of direction or species. Since its F value is at least one order higher than those of treatment-direction and treatment-species interactions, the treatment effect also tends to be the same for all directions and species. There is a significant ($P < 0.05$) treatment-direction interaction, which arises, as indicated in Figure 5.2, from the fact that the increase of the average diffusion coefficients by various predrying treatments in the

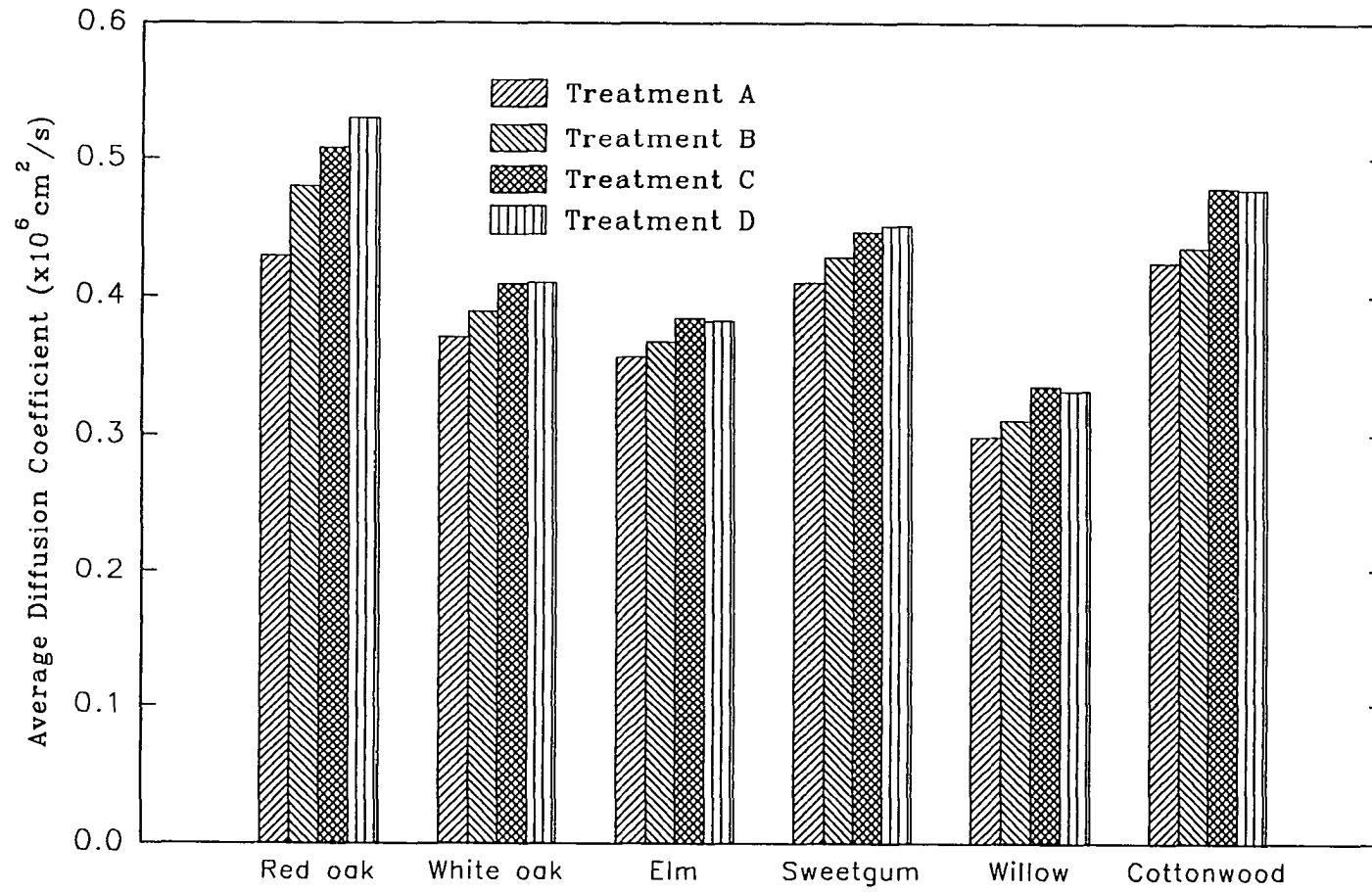


Figure 5.1. Average diffusion coefficients for the four treatments in Phase 1 (Drying Above the FSP).

Table 5.4. Average diffusion coefficients^a of six hardwoods for Phase 2 (Drying Below the FSP)

Direction	Treatment	Species					
		Red oak	White oak	Elm	Sweetgum	Willow	Cottonwood
		----- x10 ⁻⁶ cm ² /s -----					
Longitudinal	A	9.05	8.03	18.97	14.45	15.66	20.35
	B	10.67	10.22	21.59	21.25	20.71	25.30
	C	14.24	11.73	24.11	22.62	26.91	32.94
	D	13.29	10.74	27.58	24.27	24.94	28.25
	E	11.35	9.48	19.25	18.92	17.65	29.98
.....							
Radial	A	1.44	2.41	9.11	4.80	9.96	5.46
	B	1.74	2.99	10.38	5.44	11.37	6.38
	C	2.22	3.12	14.80	9.73	10.61	6.99
	D	1.72	2.89	10.77	5.07	15.40	8.33
	E	2.10	2.39	12.13	7.84	11.62	7.99
.....							
Tangential	A	1.26	1.36	6.47	4.80	5.91	4.62
	B	1.50	1.55	6.70	4.85	6.86	5.52
	C	1.56	2.63	10.81	5.22	7.23	8.38
	D	2.11	3.75	12.78	4.87	6.35	5.19
	E	1.30	2.75	9.08	4.78	7.41	5.86

^a Each value represented the mean of two replicates.

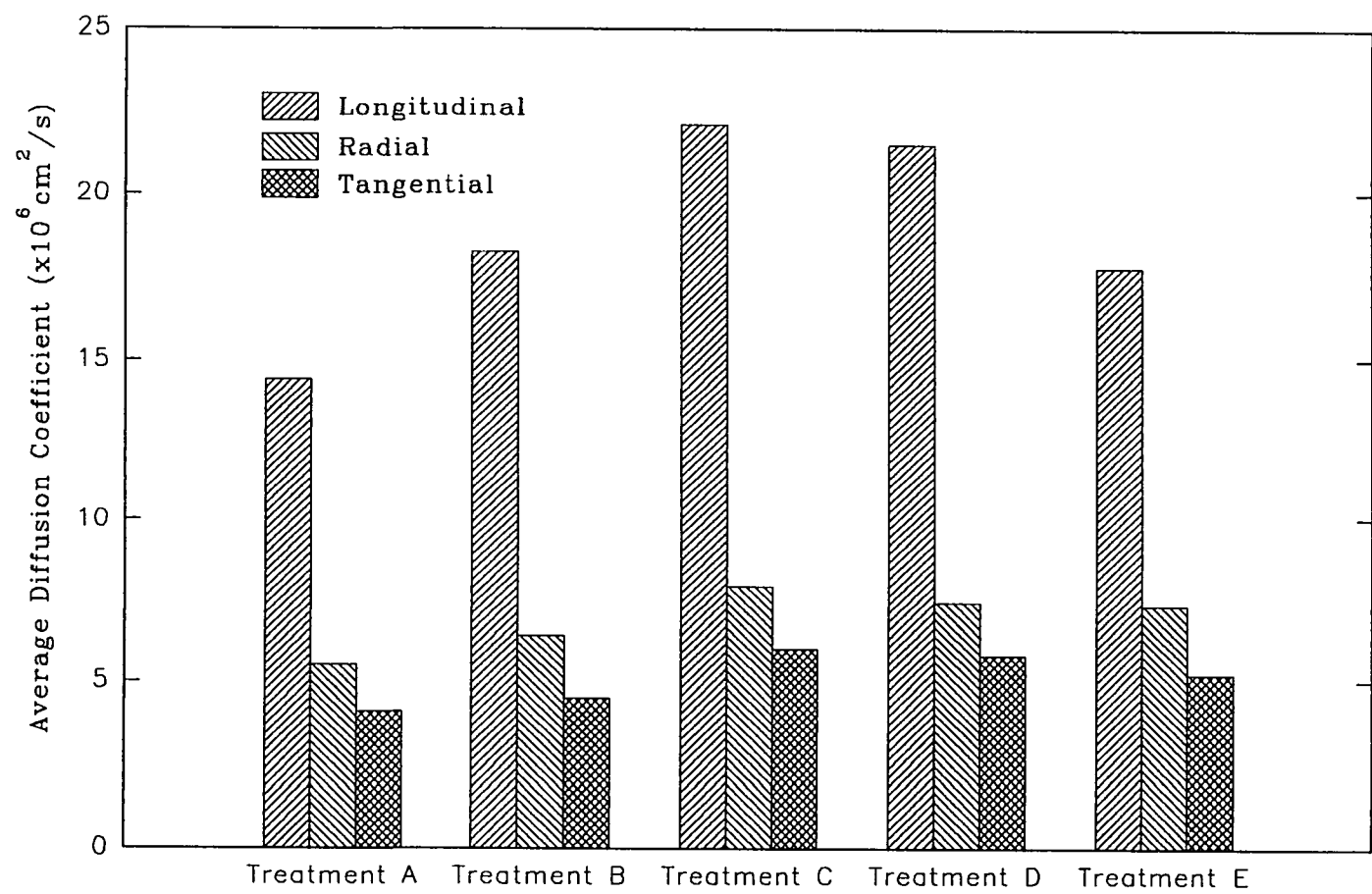


Figure 5.2. Average diffusion coefficients of six hardwoods for Phase 2 (Drying Below the FSP).

longitudinal direction is larger than that in either the radial or the tangential direction.

Table 5.5. Analysis of variance of average diffusion coefficients for Phase 2 (Drying Below the FSP)

Source	DF	MS	F	P
DIR	2	3.185x10 ³	480.83	0.001
TREAT	4	8.840x10 ¹	13.35	0.001
SPECIES	5	5.278x10 ²	79.68	0.001
CHM ^a	1	3.172x10 ⁻¹	0.05	0.827
DIRxTREAT	8	2.170x10 ¹	3.28	0.003
DIRxSPECIES	10	7.862x10 ¹	11.87	0.001
TREATxSPECIES	20	4.731	0.71	0.802
DIRxTREATxSPECIES	40	5.192	0.78	0.803
Error	89	6.624	--	--

^a Batch effect of drying samples at different time in the AMINCO chamber.

Table 5.6. Comparison of the effect of treatments on average diffusion coefficients by pair-wise t-tests (LSD) at 0.05 significance level in Phase 2 (Drying Below the FSP)

Treatment	C	D	E	B	A
Mean (x10 ⁶ cm ² /s)	<u>11.99</u>	<u>11.57</u>	<u>10.10</u>	<u>9.72</u>	<u>8.01</u>

The results of pair-wise t-tests (LSD) for treatments are given in Table 5.6. Except for Treatment B, all other predrying treatments resulted in a significant increase in the diffusion coefficients. The t-tests do not show any difference of effects among Treatments C, D, and E. It also does not show a difference between Treatments A and B.

The increase in the diffusion coefficients by hot-water soaking and steaming in the green condition is not only due to the opening up of capillary passageways, but also to the reduction in resistance of moisture moving through the cell walls. Moisture movement below the FSP is a diffusion phenomenon. Choong (1965), through model calculations, showed that the pit openings are important to moisture diffusion only at low moisture contents, and Siau (1984) indicated that the contribution of the pit openings to diffusion at high moisture content may be neglected. Therefore, the main resistance to the movement of moisture comes from the cell walls. Soaking and steaming treatments redistributed the water-soluble substances in the cell walls, and as a consequence, the cell walls became more accessible to water, which facilitated moisture movement.

CONCLUSIONS

- 1) Steaming and hot-water soaking treatments improved the movement of moisture in southern hardwood species during drying.
- 2) Steaming in the green condition for 5 hours was more effective in increasing the diffusivity than steaming for 1 hour.
- 3) Hot-water soaking for 10 hours was as effective as steaming in the green condition for 5 hours.

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CHAPTER VI

EFFECT OF BULKING AGENTS ON DRYING PROPERTIES OF WOOD

INTRODUCTION

Bulking agents are substances which penetrate the structure of wood, "bulk" the amorphous region of its lignocellulose cell walls, and thus stabilize its dimensions against changes with variation in moisture content. Bulking agents may be natural substances such as wood extractives which are formed during the transition of sapwood into heartwood. They may also be chemicals artificially introduced into wood in a chemical modification process.

Bulking agents may affect moisture transport in two ways: by mechanically blocking the macro- and micro-passageways of moisture in wood, and/or by changing the hygroscopicity of wood (Cooper 1974, Taylor 1974, Spalt 1979). Both affect the diffusion of water molecules ("jumping" as suggested by Stamm 1964) from one sorption site to another. In industrial drying practice, the initial moisture content (MC) of wood is high, and, therefore, capillary movement occurs. Hart (1964) and Choong *et al.* (1974) suggested that moisture flow above the fiber saturation point (FSP) is dependent upon permeability since it occurs mainly through the continuous void structure. They reported a relationship between permeability and diffusivity.

While extensive research has been carried out to study the effect of bulking agents on equilibrium moisture content (EMC) and dimensional stability of wood (Nearn 1955, Stamm 1964, Spalt 1979, Choong and Barnes 1969), there is limited

information describing how and to what extent bulking agents affect moisture transport properties of wood. There is some evidence that bulking agents in wood affect moisture movement. For instance, Resch and Ecklund (1964) surveyed 12 variables affecting the drying rate of redwood and found that extractives were the most influential in reducing the drying rate. Comstock (1965) and Fogg (1968) reported that extracting softwoods by hot water and chemicals to remove part of the extractives could increase longitudinal permeability. While bulking chemicals prove effective in enhancing dimensional stability, their effect on moisture movement has not been appropriately explored.

This experiment was designed to examine the effect of bulking agents in wood on the movement of moisture during drying. For extractives, the effect was examined by comparing the difference of moisture diffusivities between control and extracted samples. For bulking chemicals, it was done by comparing control with impregnated samples. For the purpose of understanding where bulking agents are located and how they block the moisture passages in wood, shrinkages and gas permeabilities for the same treatments were also measured. The hypothesis was that the blockage of the moisture passages by extractable substances or bulking chemicals would result in a reduction in drying rate.

EXPERIMENTAL PLAN AND PROCEDURES

STUDY PLANMoisture Movement

To study the effect of hot-water soluble extractives, the heartwood of redwood (*Sequoia sempervirens*, a softwood), and the heartwood of red oak (*Quercus* sp., a hardwood), were selected because they contained relatively high amounts of extractives. Hot-water soaking at 70 °C was the only treatment. The amount of material extracted from the samples was determined by "freeze-drying." A randomized block design with three replications was applied to reduce the variation due to different cutting positions.

To study the effect of bulking chemicals, the sapwood of southern pine (*Pinus* sp., a softwood) and the sapwood of yellow poplar (*Liriodendron tulipifera*, a hardwood) were chosen because these woods contained relatively low amounts of extractives, which could be further reduced by a cold water soaking process. Polyethylene glycol (PEG 600) and a natural condensed tannin were the bulking chemicals used to treat the samples. A randomized block design with three replications was also applied. The amount of PEG impregnated into the samples was estimated from the change in solvent concentration, and the amount of tannin impregnated into the wood was determined directly from the reduction of its initial amount in the treating solution.

Wood samples were dried from near saturation to EMC, and moisture movement was restricted to the radial direction. The diffusion coefficients were calculated with the technique described in Chapter IV to separate the effect of surface moisture content.

Shrinkage

The three variables examined were radial (S_r), tangential (S_t), and volumetric (S_v) shrinkages. S_v was taken as the summation of S_r and S_t .

Gas Permeability

The same species and treatment processes as described in the drying experiment were applied. Gas permeabilities were measured in both longitudinal and radial directions using six replications for each treatment-species-direction combination because of the relatively large variation in the permeability measurement.

PROCEDURES

Moisture Movement

Air-dry redwood samples were obtained from the Louisiana-Pacific Corporation in Samoa, California. The other three species (southern pine, red oak, yellow poplar) were obtained in the green condition from a local sawmill in Denham Springs. For each species, only one board about 6 meters in length and 5 cm in thickness was used.

The cutting scheme for the preparation of wood samples is shown in Figure 6.1. The samples were obtained from 3 blocks at the top, middle, and bottom of the board. For redwood heartwood and red oak heartwood, a 2.0-cm thick slab was cut from each block, from which two samples 2.0cm × 2.0cm × 1.5cm in dimensions were obtained. An adjusting miter gage was used to define true radial direction of flow. For southern pine and yellow-poplar, a 2.0-cm thick slab was also cut from each block, but three samples were obtained from the slab.

In redwood and red oak, half of the samples was subjected to cold water soaking in a beaker inside a desiccator. A vacuum was applied for an hour, and then water was gradually added to the beaker while still under vacuum. Vacuum and atmospheric pressure treatment was applied every three hours until the samples sank in water. Then each sample was placed in a 60-ml wide-mouth plastic jar with tight cover, which was filled with water. The jars were placed in a thermostatically-controlled water bath for the hot-water soaking treatment at 70 °C. After 72 hours, each of the samples was transferred to a 30-ml plastic jar and kept wet. Meanwhile, the extracted solution inside each of the 60-ml jar was poured into a Pyrex round bottom flask. The amount of extractives in each flask was determined by freezing the solution in an alcohol bath at about -40 °C, and then drying in a freeze dryer.

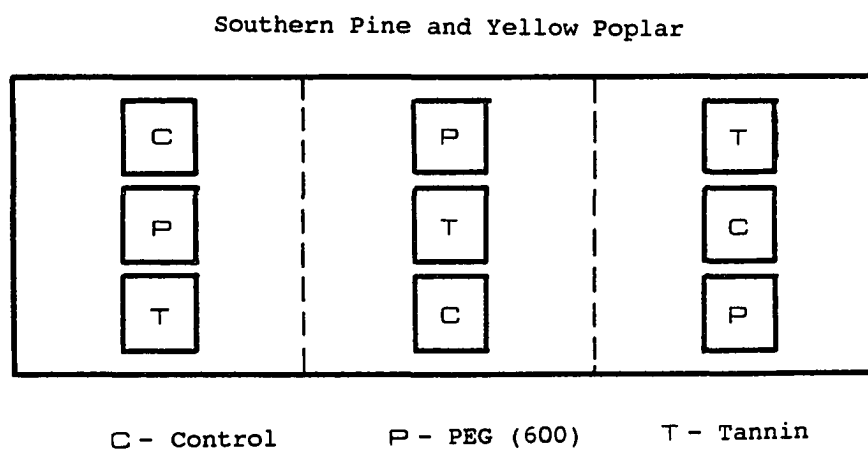
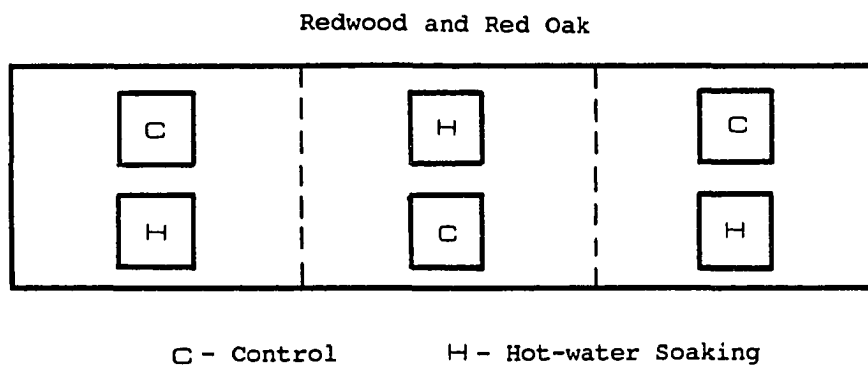


Figure 6.1. Cutting scheme for wood samples used to study the effects of natural extractives, and PEG (600) and tannin treatments on moisture movement during drying.

The other halves of the samples of redwood and red oak were also subjected to vacuum and atmospheric pressure treatment in a desiccator. Each time before vacuum was applied, the water in the beaker was poured out, and new water was gradually added at the end of the one-hour vacuum period. This process was necessary to reduce the removal of cold water extractives from the samples. After the samples had sunk in water, they were individually wrapped in aluminum film. Droplets of water were added to keep the samples wet.

Southern pine sapwood and yellow poplar sapwood were soaked in cold water for about two months to remove the cold water extractives. For those which were treated with PEG 600, each sample was submerged in a 60-ml plastic jar with 50 percent PEG concentration for 15 days. At the end of the treatment, the refractive index of the solution was measured with a RFM-90 automatic refractometer. The concentration of the solution was obtained from the calibration line shown in Figure 6.2. The amount of PEG could be calculated by multiplying the concentration by the total weight of the solution. The amount of PEG penetrating the sample was the difference between the PEG weight at the beginning and that at the end of the treatment process.

The tannin used in this study was obtained from Dr. R.W. Hemingway of the USDA Southern Forest Experiment Station. The molecular weight of the tannin was relatively small, and had an average degree of polymerization of between 3 and 4

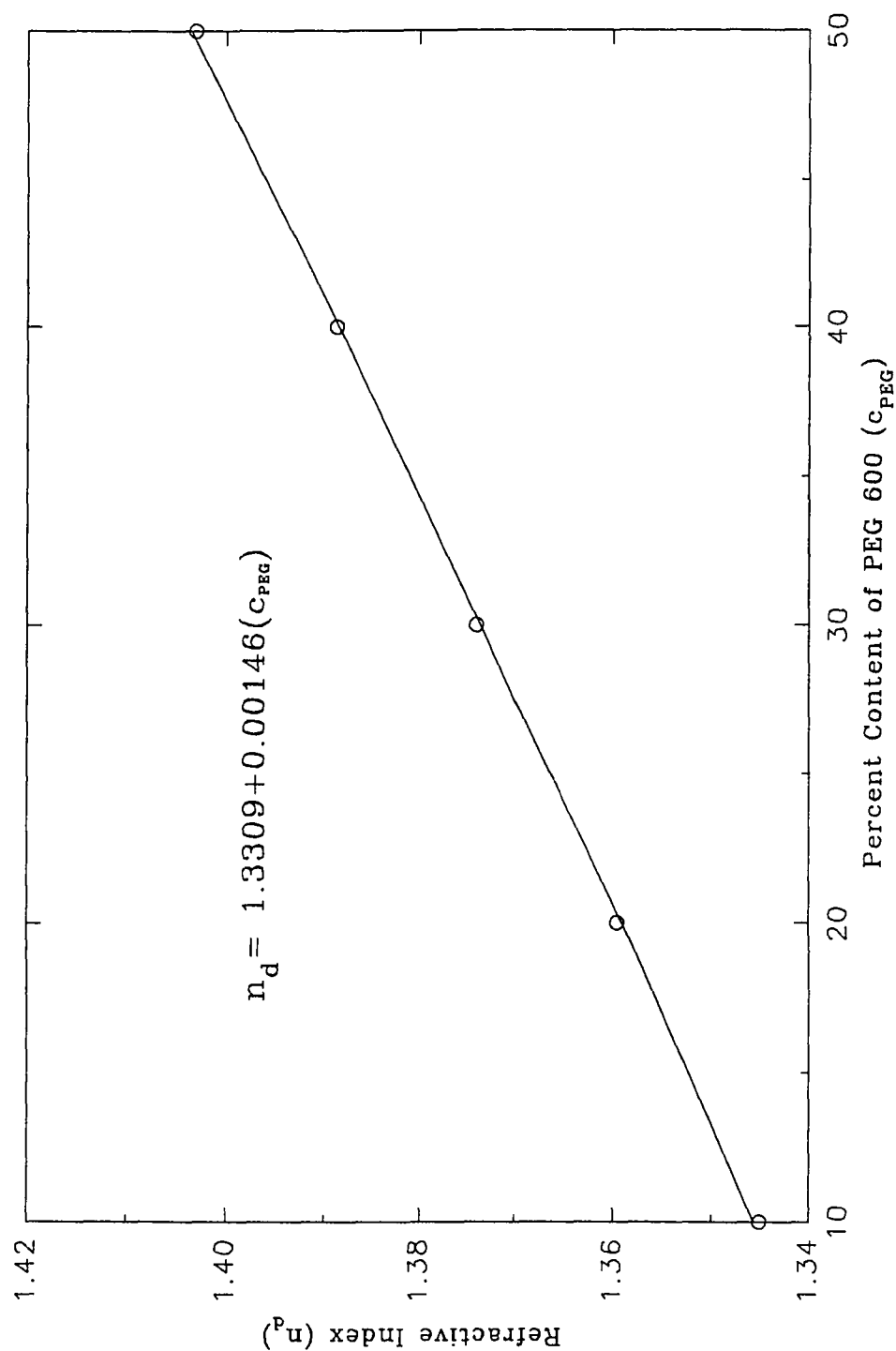


Figure 6.2. Calibration curve of refractive index (n_d) as a function of percent content of PEG (600).

flavonoid units. For treatment with tannin, each sample was submerged in a 60-ml jar containing an approximately 30 percent tannin solution for 15 days. At the end of treatment, the tannin solution in each jar was dried in an oven at 103 °C. From the known weights of the tannin at the beginning and the end of the treatment process, the amount of tannin penetrating the sample could be calculated.

After treatments, all the samples were coated with Dow's Saran F-120 resin to ensure uni-directional moisture movement. They were dried from near saturation to 6 percent MC (45 °C, 30 percent RH) inside an AMINCO environmental chamber with an air velocity of 2 m/s. Throughout the drying period the samples were weighed periodically to monitor moisture loss and the weights were recorded. The diffusion coefficients were then calculated from the drying curves.

At the end of the drying period, redwood and red oak samples were sliced into thin wafer pieces. The wafers from each sample were placed inside 60-ml jars, and soaked in hot water for 72 hours. The amount of extractives in solution in each jar was determined by freeze-drying. The wafers were then oven-dried at 103 °C to determine their moisture contents based on their extractive-free weight.

Shrinkage

Shrinkage was measured on the same samples used for calculating the diffusion coefficient by measuring the dimensions in the radial and tangential directions when

weighing. The shrinkages were plotted as a function of MC. The formula for calculating the percent shrinkage S is

$$S = \frac{D_i - D_f}{D_i} \times 100 \quad (6.1)$$

where D_i is the initial dimension and D_f is the final dimension of a sample at about 6 percent MC.

Gas Permeability

For each species, two boards measuring about 6 meters in length and 5 cm in thickness were obtained from the same source as those in the drying experiment. The cutting scheme for preparing the wood samples is shown in Figure 6.3. A split-plot randomized block design was used to reduce the variation due to different board and cutting positions. From each board, a 35-cm long block was cut and air-dried for two weeks in the laboratory. For redwood and red oak, six 1.5-cm thick slabs (three in the longitudinal direction and three in the radial direction) were cut from each block; and from each slab, two permeability samples were obtained using a 1.59-cm Greenlee plug cutter. For southern pine and yellow poplar, six 1.5-cm thick slabs were also cut from each block, but three permeability samples were obtained from each slab.

These samples were subjected to the same treatments (i.e. hot-water soaking, PEG, and tannin bulking) as described above for the drying samples. Instead of a single sample, three samples were placed in each 60-ml plastic jar, and

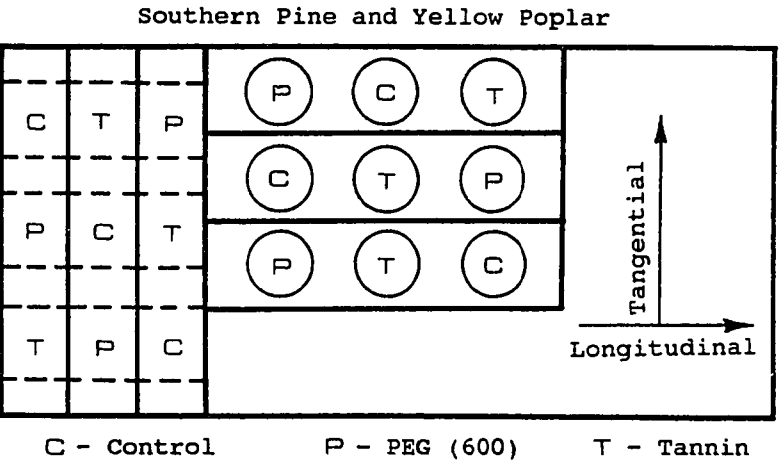
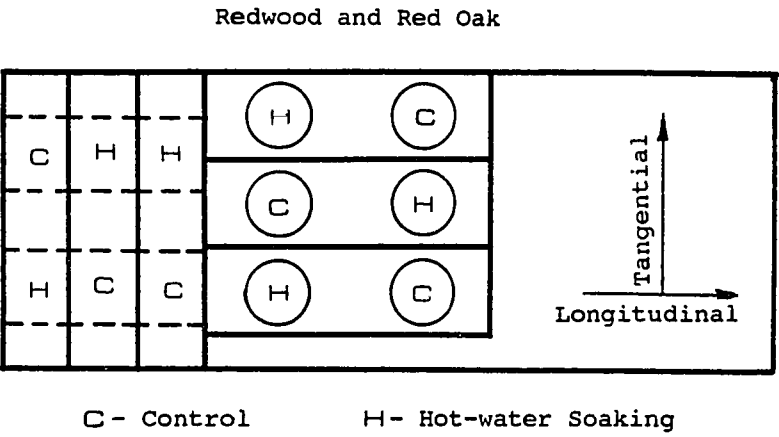


Figure 6.3. Cutting scheme for wood samples used to study the effects of natural extractives, and PEG (600) and tannin treatments on gas permeability.

corresponding average values of extractives and bulking chemical contents for three samples were obtained. When the treatments had been completed, each sample was coated with a layer of Dow's Saran F-120 resin on the cylindrical surface to reduce the possibility of gas leak during measurement. All the samples were then conditioned in the AMINCO environmental chamber at 35 °C and 80 percent RH for about one month to obtain an EMC of about 15 percent.

Gas permeability measurements were made using an apparatus similar to that described by Choong *et al.* (1974). A schematic diagram of the apparatus is illustrated in Figure 6.4, which includes a sample holder made of a stainless steel barrel fitted inside with a neoprene rubber sleeve. The space between the barrel and the sleeve was filled with water and connected to a vacuum and pressure system. The permeability sample was placed between two stainless steel plugs with a 0.32 cm pore through the center of each, which was sealed inside the rubber sleeve in the sample holder by hydrostatic pressure equal to at least twice the upstream pressure so that gas could not flow around the sample.

Nitrogen gas was passed through a filter and two nullamatic regulators. One regulator was maintained at 75 psig (6.1 atm) to control the sleeve pressure holding the sample inside the steel barrel; the other regulator controlled the upstream flow to the sample. All samples were measured at a mean pressure \bar{P} of 1.2 atm. The upstream pressure, P_1 , and

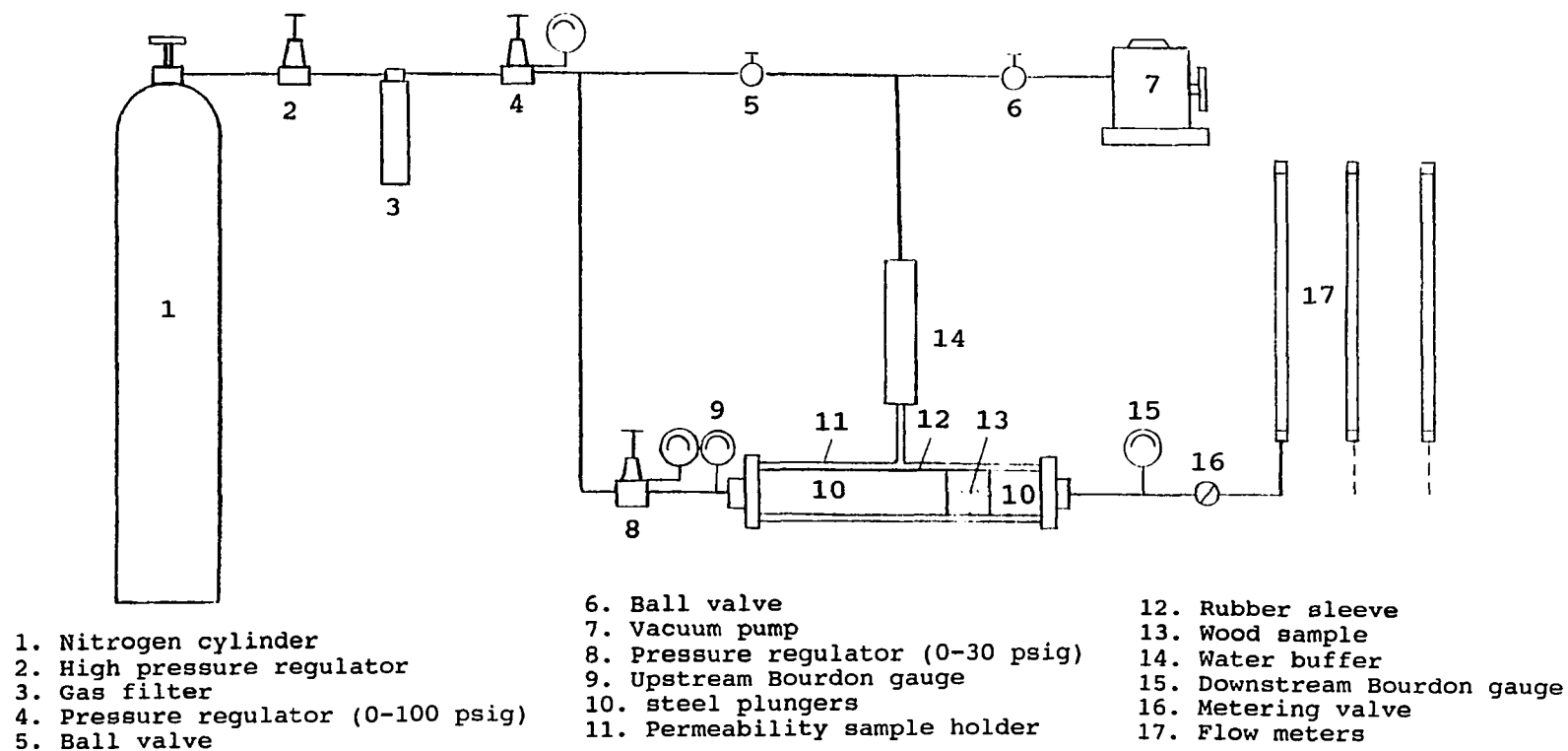


Figure 6.4. Schematic diagram of gas permeability apparatus.

downstream pressure, P_2 , were adjusted to give the same mean pressure. The apparent permeability, K , was calculated using Darcy's law for gas, as shown below.

$$K = \frac{Q_a \mu L P_a}{A F \Delta P} \quad (6.2)$$

Q_a is the flow rate at atmospheric pressure P_a , μ is the viscosity of nitrogen, A is the cross sectional area of the sample, L is the length of the sample, and ΔP is the pressure drop ($P_1 - P_2$). The temperature in the testing room was 25 ± 0.5 °C, and the atmospheric pressure was about 1 atm. Therefore, μ should be about 0.0178 centipoise.

Following the procedure described above, the amount of residual extractives in the samples was determined, and the MC was calculated based on the extractive-free weight.

RESULTS AND DISCUSSION

MOISTURE MOVEMENT

Water-soluble Extractives

The drying curves for redwood and red oak heartwood are shown in Figure 6.5. As described in Chapter IV, when the samples were dried from a high MC above the fiber saturation point (FSP) to the final equilibrium moisture content (EMC) below the FSP, the effect of surface moisture content must be incorporated in the diffusion model in order to obtain a good prediction of the experimental drying curves. In this study,

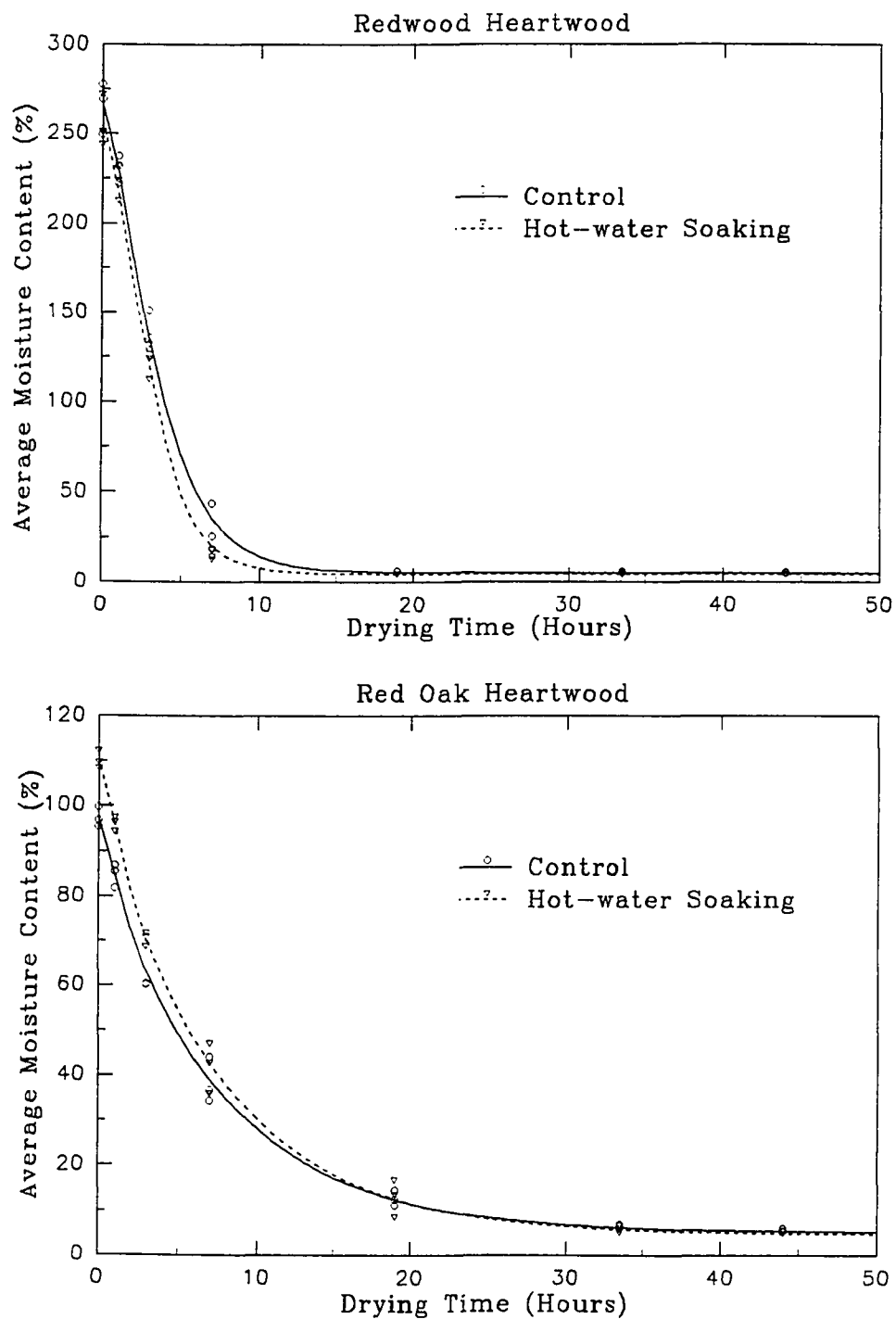


Figure 6.5. Effect of hot-water soaking on moisture removal during drying for redwood heartwood and red oak heartwood.

the diffusion coefficient was assumed to be constant and the surface MC was assumed to decrease exponentially toward the final EMC. The optimum surface parameters (β) differed among control and treated samples of the same species, but the variation was within 3 percent. Therefore, it was convenient to calculate all diffusion coefficients with the same β value of that species.

The average values of the surface parameter β and the diffusion coefficient D are summarized in Table 6.1. An analysis of variance (Table 6.2) shows that the diffusion coefficients of redwood differ significantly from those of red oak. Hot-water soaking increased the diffusivity significantly ($P < 0.05$). The significant treatment-species interaction ($P < 0.10$) indicated that the response of redwood to hot-water soaking differed from that of red oak. As shown in Table 6.1, hot-water soaking could increase the diffusion coefficient for redwood by 21 percent, but it could increase it only 5 percent for red oak.

When the samples were treated based on the procedures described above, the initial MCs could not be the same. For instance, the initial MCs of the treated red oak samples were higher than the control samples. As a result, the MCs of the treated samples could be somewhat higher than that of the control ones. Nevertheless, the calculated average diffusion coefficient of the treated samples could still be larger than that of the control samples since the diffusion coefficient is

independent of initial moisture content. This is because the drying rate was larger for the treated samples, and more moisture could be removed in a given time.

Table 6.1. Average values of D and β for control and hot-water soaked samples of redwood heartwood and red oak heartwood

Species	Treatment	β ($\times 10^4$ 1/s)	D ($\times 10^6$ cm ² /s)
Redwood	Control	1.32	34.25
	Hot-water	1.32	41.60
Red oak	Control	3.33	8.14
	Hot-water	3.33	8.55

Table 6.2. Analysis of variance of diffusion coefficients for control and hot-water soaked samples of redwood heartwood and red oak heartwood

	Source	DF	MS	F	P
	SPECIES	1	2.626×10^3	147.70	0.007
Error A:	BLOCK	2	1.778×10^1	2.56	0.157
	TRT	1	4.509×10^1	6.50	0.044
	TRT x SPECIES	1	3.612×10^1	5.21	0.063
	Error B	6	6.937	--	--

Hot-water soaking for 72 hours, as shown in Table 6.3, extracted out most of the water-soluble extractives. However, the diffusion coefficients increased only slightly. Therefore, a large portion of water-soluble extractives probably stayed in locations in wood other than moisture passageways.

Table 6.3. Average amount of water-soluble extractives in control and hot-water soaked drying samples

Species	Amount of Water-soluble Extractives (%)	
	control	treatment
Redwood	13.68	1.57
Red oak	6.48	0.35

Bulking Chemicals

The drying curves for southern pine and yellow poplar sapwood are shown in Figure 6.6. The optimum surface parameters (β) were similar for samples within the same species-treatment combination, but generally differed among treatments. Therefore, the same β value was used to calculate the diffusion coefficients of the same species-treatment combination.

The average values of the surface parameter β and the diffusion coefficient D are summarized in Table 6.4. An analysis of variance (Table 6.5) shows that the treatment effect was highly significant, but other effects were not. As shown in Table 6.4, the diffusion coefficients of the control samples were an order of magnitude higher than those of treated samples. The reduction in the diffusion coefficients in treated samples may be due to the blockage of the macro-passages. When the wood was in the saturated condition, there was a considerable amount of free water in the voids; therefore, the macro-passages were very important for moisture

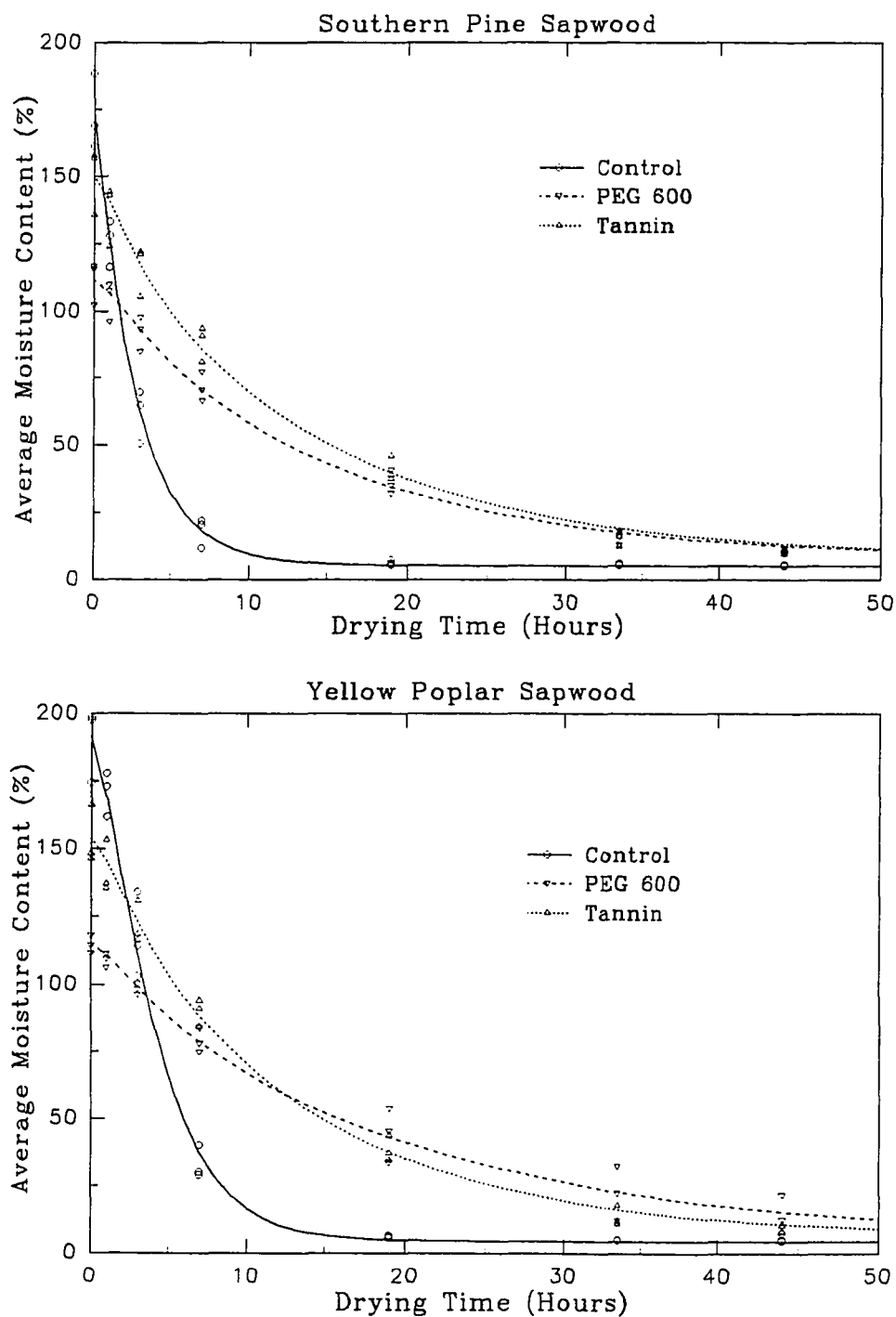


Figure 6.6. Effect of PEG (600) and tannin treatments on moisture removal during drying for southern pine sapwood and yellow poplar sapwood.

Table 6.4. Average values of D and β for control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood

Species	Treatment	β ($\times 10^4$ 1/s)	D ($\times 10^6$ cm ² /s)
Southern pine	Control	4.72	24.01
	PEG (600)	1.19	4.47
	Tannin	1.86	4.73
Yellow poplar	Control	1.19	26.94
	PEG (600)	1.03	3.50
	Tannin	1.42	5.05

Table 6.5. Analysis of variance of diffusion coefficients for control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood

	Source	DF	MS	F	P
	SPECIES	1	2.614	0.26	0.659
Error A:	BLOCK	2	9.899	0.96	0.415
	TRT	2	8.865 $\times 10^3$	86.05	0.001
	TRT \times SPECIES	2	5.917	0.57	0.581
	Error B	10	1.030 $\times 10^1$	--	--

to move toward the wood surface. Since the chemicals penetrated into the wood through the passages, they could deposit in the void spaces and block the passages. Consequently, the movement of moisture during drying slowed down, and the diffusion coefficient decreased. Table 6.6 shows the amount of bulking chemicals retained by the samples. Tannin content is much lower than PEG content, but these chemicals behaved similarly as shown in Figure 6.6 and Table

6.4. Therefore, a portion of the PEG was found in locations in wood other than the moisture passageways.

Table 6.6. Average amount of bulking chemicals in impregnated drying samples

Species	Type	Amount of Bulking Chemicals (%)
Southern pine	PEG (600)	66.79
Southern pine	Tannin	37.15
Yellow poplar	PEG (600)	70.88
Yellow poplar	Tannin	28.32

Shrinkage

Water-soluble Extractives

The samples began to shrink when the average MC was still quite high because the surface MC dropped below the FSP first. They began to shrink normally after the average MC reached the FSP. Therefore, the shrinkage-MC relationships for redwood and red oak can be shown as linear regression lines (Figure 6.7) obtained from the straight line part of the shrinkage data.

An analysis of variance (Table 6.7) indicates that the shrinkage of red oak in either radial or tangential direction differs from that of redwood; the former, as shown in Table 6.8, is larger than the latter because red oak is denser than redwood. As expected, the shrinkage in the tangential direction was larger than that in the radial direction. The

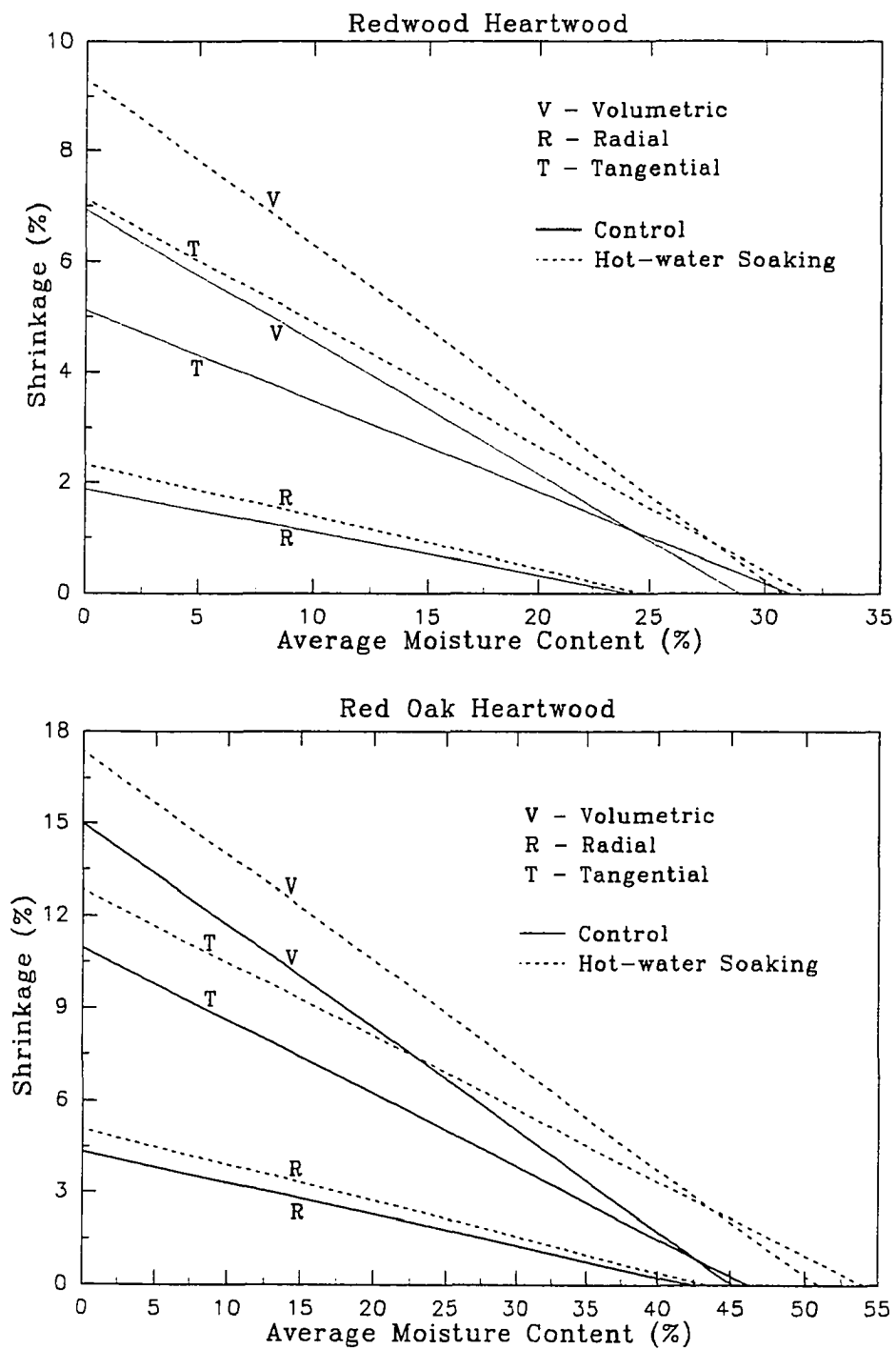


Figure 6.7. Effect of hot-water soaking on radial, tangential, and volumetric shrinkages in redwood heartwood and red oak heartwood.

Table 6.7. Analysis of variance of radial and tangential shrinkages for control and hot-water soaked samples of redwood heartwood and red oak heartwood

	Source	DF	MS	F	P
	SPECIES	1	1.300×10^2	150.41	0.007
	BLOCK	2	1.063×10^{-1}	0.12	0.891
Error A:	SPECIESxBLOCK	2	8.642×10^{-1}	0.82	0.503
	TRT	1	8.644	8.20	0.046
	SPECIESxTRT	1	1.264×10^{-2}	0.01	0.918
Error B:	SPECIESxTRT*BLOCK	4	1.054	4.77	0.029
	DIR	1	1.927×10^2	872.66	0.001
	SPECIESxDIR	1	1.720×10^1	77.90	0.001
	TRTxDIR	1	2.286	10.35	0.012
	SPECIESxTRTxDIR	1	2.607×10^{-2}	0.12	0.740
	Error C	8	2.208×10^{-1}	--	--

Table 6.8. Average shrinkages of redwood and red oak samples in radial and tangential directions

Unit: %	Radial	Tangential
Redwood	2.10	6.08
Red oak	5.06	12.4

tangential/radial (T/R) ratio is 2.5 for red oak and 2.9 for redwood. Several anatomical characteristics are believed responsible for this differential shrinkage, including presence of ray tissue, frequent pitting on radial walls, domination of summerwood in the tangential direction, and differences in the amount of cell wall material between tangential and radial directions. The species-direction

interaction is significant because the difference between radial and tangential shrinkages for red oak is larger than that for redwood.

Hot-water soaking increased the shrinkages, but its effect, as shown in Table 6.7, was not as large as that due to species or direction. The treatment-direction interaction is significant because the increase in shrinkage in the tangential was larger than that in the radial direction (Table 6.9).

Table 6.9. Average shrinkages of control and hot-water soaked samples in radial and tangential directions

Unit: %	Radial	Tangential
Control	3.29	8.34
Hot-water soaking	3.87	10.2

Table 6.10. Analysis of variance of volumetric shrinkages for control and hot-water soaked samples of redwood heartwood and red oak heartwood

Source	DF	MS	F	P
SPECIES	1	2.600×10^2	168.87	0.001
TRT	1	1.729×10^1	11.23	0.010
TRT x SPECIES	1	2.528×10^{-2}	0.02	0.901
Error	8	1.540	--	--

The analysis of variance of volumetric shrinkages (Table 6.10) indicates that the effects of both species and treatments were significant, and that there was no interaction

between them. The overall average shrinkage after hot-water soaking (14.0 percent) was 20 percent larger than that of the untreated control (11.6 percent). Hot-water soaking treatment resulted in the decrease of water-soluble extractives in the cell walls and made them less "bulked."

Bulking Chemicals

Linear regression lines (Figure 6.8) show the shrinkage-MC relationships for southern pine and yellow poplar. An analysis of variance (Table 6.11) indicates that the treatment effect is highly significant, at least one order higher than any other single effect. As expected, impregnating woods with bulking chemicals causes a reduction in shrinkage. The significant treatment-direction interaction indicates that the shrinkage pattern of each treatment in the radial and tangential directions is not the same. As shown in Table 6.12, while the tannin-treated woods followed a trend in shrinkage similarly to those of the untreated control, the PEG-treated woods showed almost no shrinkage.

The shrinkage of southern pine in either radial or tangential direction (Table 6.11) differed from that of yellow poplar; the former, as shown in Table 6.13, was smaller than the latter. Generally, the higher the density of the sample, the more it will shrink. The density of southern pine is slightly higher than that of yellow poplar. However, the average shrinkage values of southern pine are somewhat less than those of yellow poplar. This phenomenon can be explained

Table 6.11. Analysis of variance of radial and tangential shrinkages for control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood

	Source	DF	MS	F	P
	SPECIES	1	6.612×10^{-1}	54.85	0.018
	BLOCK	2	1.254×10^{-1}	10.41	0.088
Error A:	SPECIESxBLOCK	2	1.206×10^{-2}	0.10	0.914
	TRT	2	8.728×10^1	726.67	0.001
	SPECIESxTRT	2	2.170×10^{-2}	0.18	0.838
Error B:	SPECIESxTRT*BLOCK	8	1.201×10^{-1}	1.63	0.215
	DIR	1	1.308×10^1	177.35	0.001
	SPECIESxDIR	1	3.614×10^{-1}	4.90	0.047
	TRTxDIR	2	3.404	46.16	0.001
	SPECIESxTRTxDIR	2	2.585×10^{-1}	3.51	0.073
	Error C	12	7.374×10^{-1}	--	--

Table 6.12. The average shrinkages of control and chemical impregnated samples in radial and tangential directions

Unit: %	Radial	Tangential
Control	4.41	6.38
PEG (600)	0.30	0.29
Tannin	3.54	5.19

Table 6.13. Average shrinkages of southern pine or yellow poplar samples in radial and tangential directions

Unit: %	Radial	Tangential
Southern pine	2.71	3.72
Yellow poplar	2.78	4.19

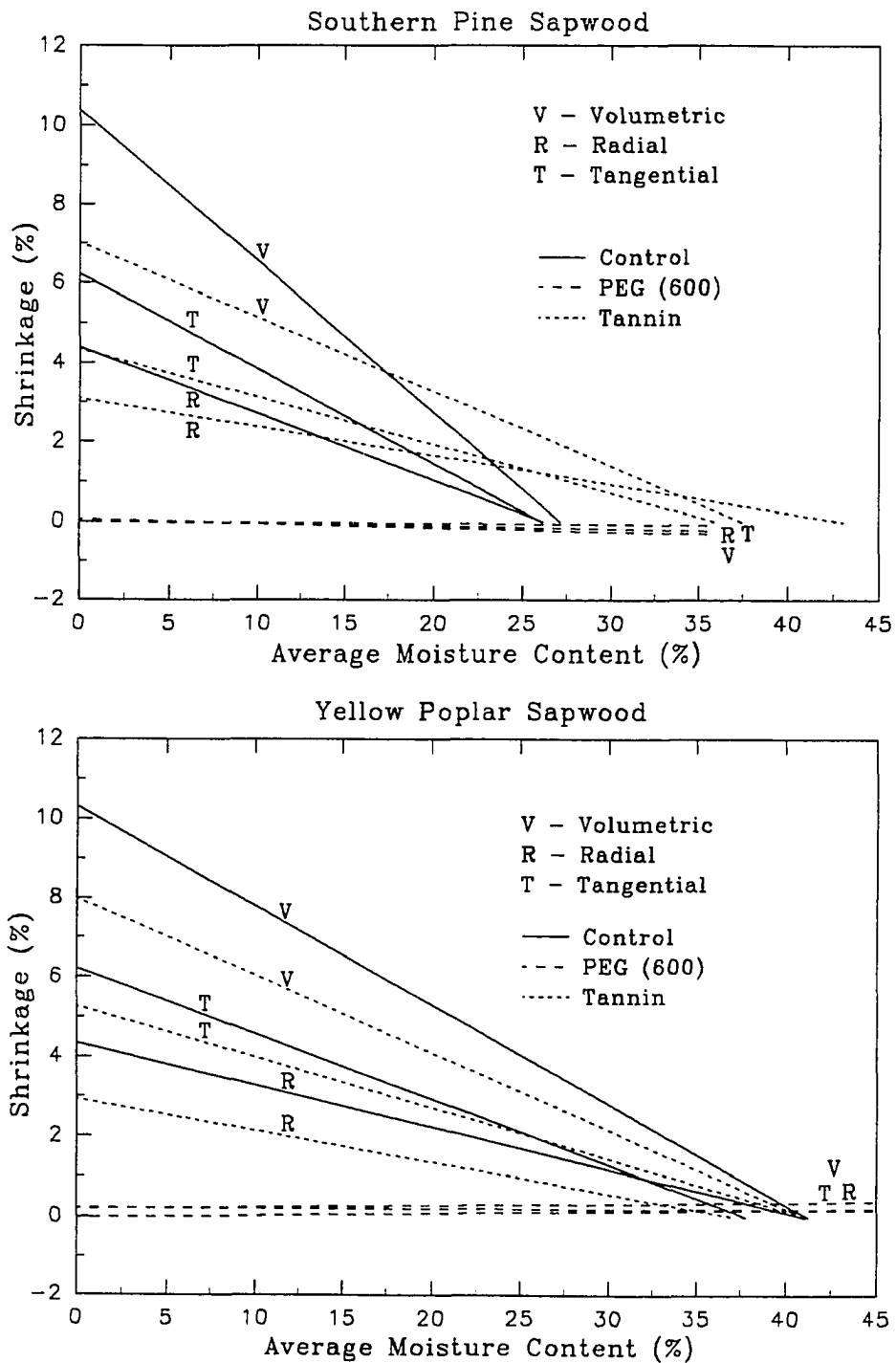


Figure 6.8. Effect of PEG (600) and tannin treatments on radial, tangential, and volumetric shrinkages in southern pine sapwood and yellow poplar sapwood.

from the bulking effect of tannin in southern pine. As shown in Table 6.6, the amount of tannin in the southern pine samples was about 30 percent higher than that in the yellow poplar samples; therefore, the southern pine samples should shrink less. Treatment with PEG is irrelevant because the PEG effectively bulked the wood; consequently there was no change in shrinkage. The shrinkage in the tangential direction is also larger than that in the radial direction. The species-direction interaction is significant because the T/R ratio for yellow poplar is larger than that for southern pine.

Table 6.14. Analysis of variance of volumetric shrinkages for control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood

Source	DF	MS	F	P
SPECIES	1	1.323	6.42	0.026
TRT	2	1.746x10 ²	847.45	0.001
TRTxSPECIES	2	4.339x10 ⁻²	0.21	0.816
Error	12	2.060x10 ⁻¹	--	--

The analysis of variance of volumetric shrinkages (Table 6.14) indicates that the treatment effect is the most important of all effects, although the species effect is also significant ($P < 0.05$). The overall average shrinkage values of the control, PEG-treated, and tannin-treated woods are 10.8, 0.6, and 8.7 percent, respectively. In terms of Anti-Shrinkage Efficiency (ASE), defined as

$$ASE(\%) = \left(\frac{S_c - S_t}{S_c} \right) 100 \quad (6.3)$$

where S_c and S_t are the average shrinkages of the untreated control and treated samples, respectively, the values of ASE are 95 and 19 percent for PEG and tannin, respectively. Since change in shrinkage behavior has been interpreted as evidence of bulking agents in the cell wall (Tarkow and Krueger 1961), it is evident that the PEG molecules could penetrate the cell walls more effectively than the tannin molecules.

GAS PERMEABILITY

Water-soluble Extractives

The results of analysis of variance are summarized in Tables 6.15 and 6.16. In the longitudinal direction (Table 6.15), the treatment effect was not significant ($P > 0.05$). However, the effect of the boards within species was significant ($P < 0.05$), suggesting that the measured permeabilities differed between the two boards within a species. As shown in Table 6.17, the variation mainly comes from the two boards of redwood, with an average value of permeabilities in one board about 2.7 times larger than that in another board. This is expected because permeability in the longitudinal direction, which can be 1000 times greater than that in the radial direction, is affected by minute changes in the micro-capillary structure of wood, and great variation in longitudinal permeability values has been

Table 6.15. Analysis of variance of permeabilities for control and hot-water soaked samples of redwood heartwood and red oak heartwood in the longitudinal direction

	Source	DF	MS	F	P
	SPECIES	1	11.596	4.34	0.173
Error A:	BOARD(SPECIES)	2	2.671	22.11	0.043
	TRT	1	1.352	11.19	0.079
	SPECIESxTRT	1	1.301	10.77	0.082
Error B:	TRTxBOARD(SPECIES)	2	0.121	7.11	0.006
	Error C	16	0.017	--	--

Table 6.16. Analysis of variance of permeabilities for control and hot-water soaked samples of redwood heartwood and red oak heartwood in the radial direction

	Source	DF	MS	F	P
	SPECIES	1	1.733×10^{-4}	1.62	0.331
Error A:	BOARD(SPECIES)	2	1.069×10^{-4}	1.80	0.193
	TRT	1	4.395×10^{-4}	7.42	0.014
	SPECIESxTRT	1	1.955×10^{-4}	3.30	0.086
	Error B	18	5.927×10^{-5}	--	--

Table 6.17. Average permeabilities of redwood and red oak samples within a board in the longitudinal direction

Unit: Darcy	Board 1	Board 2
Redwood	0.775	2.110
Red oak	0.049	0.056

reported (Choong *et al.* 1989) even within a single board. Also, the macro-passageway in the longitudinal direction may be heavily blocked in one board but not in another. Since the

effect of the interaction between treatments and boards within species was significant, the response of permeabilities to the treatments is different from one board to another. This suggests that boards with similar permeabilities need to be sorted in order to detect the effect of hot-water soaking in the longitudinal direction. In the radial direction (Table 6.16), treatment effect was significant ($P < 0.05$), but other effects were not; different boards of the two species had similar permeability and response to hot-water soaking. The average permeability value of the treated woods was 0.024 darcy, which is 60 percent higher than that (0.015 darcy) of the untreated woods. Hot-water soaking removed most of the water-soluble extractives (Table 6.18); therefore, the permeability increased. The increase, which was significant only in the radial direction, may suggest that the passages in the cell walls are more readily blocked by water-soluble extractives.

Table 6.18. Average amount of water-soluble extractives in control and hot-water soaked permeability samples

Species	Amount of Water-soluble Extractives (%)	
	control	treatment
Redwood	14.69	1.71
Red oak	6.49	0.83

Bulking Chemicals

The results of analysis of variance are summarized in Tables 6.19 and 6.20. In the longitudinal direction (Table 6.19), the effects of both species and treatments are significant ($P > 0.05$). As suggested by the significant species-treatment interaction, each species responded to the chemical impregnation differently. Table 6.21 shows the average values of the species-treatment combination. For southern pine, the PEG and tannin treatments obviously reduced the longitudinal permeability. For the yellow poplar, tannin treatment reduced the permeability, but PEG treatment resulted in a somewhat higher average value than that of the untreated control, with a difference of 0.006 darcy. However, since the standard deviation is 0.018 darcy, the difference in the average values should be accounted for by random variation.

In the radial direction (Table 6.20), the effects of both species and treatments are significant, but there was no interaction between them. The average permeability value (0.037 darcy) of southern pine was 3 times larger than that of yellow poplar (0.012 darcy). The average permeability values were 0.054, 0.013, and 0.006 darcy for the control, PEG-treated, and tannin-treated woods, respectively. Both PEG and tannin treatments reduced the permeabilities of the woods significantly.

The amounts of bulking chemicals retained by the samples are summarized in Table 6.22. The PEG levels were much higher

Table 6.19. Analysis of variance of permeabilities for control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood in the longitudinal direction

	Source	DF	MS	F	P
	SPECIES	1	1.726×10^2	260.82	0.004
Error A:	BOARD(SPECIES)	2	6.618×10^{-1}	1.47	0.332
	TRT	2	3.233×10^1	71.81	0.001
	SPECIES \times TRT	2	3.207×10^1	71.23	0.001
Error B:	TRT \times BOARD(SPECIES)	4	4.503×10^{-1}	0.55	0.698
	Error C	24	8.133×10^{-1}	--	--

Table 6.20. Analysis of variance of permeabilities for control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood in the radial direction

	Source	DF	MS	F	P
	SPECIES	1	5.424×10^{-3}	111.04	0.009
Error A:	BOARD(SPECIES)	2	4.885×10^{-5}	0.05	0.951
	TRT	2	8.184×10^{-3}	8.37	0.001
	SPECIES \times TRT	2	2.267×10^{-3}	2.32	0.117
	Error B	28	9.783×10^{-4}	--	--

Table 6.21. Average permeabilities of control and chemical impregnated samples of southern pine sapwood and yellow poplar sapwood in the longitudinal direction

Unit: Darcy	Southern pine	Yellow poplar
Control	8.11	0.0295
PEG (600)	3.23	0.0360
Tannin	1.88	0.0094

than the tannin levels. However, the tannin-treated woods had permeability values similar to, or even lower, than those of

the PEG-treated woods, indicating tannin blocked the macro-passageways more effectively. PEG also blocked the macro-passageway, but it also expanded the cell wall and kept the wood in the swollen condition. Thus blockage by PEG was not as effective as by tannin.

Table 6.22. Average amount of bulking chemicals in impregnated permeability samples

Species	Type	Amount of Bulking Chemicals (%)
Southern pine	PEG (600)	66.82
Southern pine	Tannin	38.31
Yellow poplar	PEG (600)	71.65
Yellow poplar	Tannin	25.38

LOCATION OF WATER-SOLUBLE EXTRACTIVES

Tarkow and Krueger (1961) have suggested a simple way to predict the amount of extractives in the cell wall from shrinkage data. They stated that if extractives were removed with hot water and the sample was dried and remeasured, its new volume should be smaller than before the extractives were removed, by an amount approximately equal to the volume of extractives removed from the wood substance. The loss in volume multiplied by the density of the extractives yields the weight of the hot-water extractives which were present in the cell wall. In their experiment, they found that the average

specific gravity of extractives in redwood is about 1.4. This is slightly lower than the specific gravity of the wood substance, which is 1.45-1.55 (Stamm 1964). If 1.4 is accepted as the approximate specific gravity of extractives for all species, then the percent extractives within the cell wall can be conveniently estimated. For example, from Figure 6.7 for redwood, the average volumetric shrinkage was 6.9 percent for the control samples, and 9.3 percent for the samples treated with hot water. Therefore, the reduction in volume was 2.4 percent, or 0.144 cm^3 . The calculated average amount of extractives in the cell wall then is 0.20 gram. The measured average amount of extractives in redwood was 0.39 gram; therefore, 51 percent extractives should be present in the cell wall of redwood. Similarly, from Figure 6.7 for red oak, the reduction in volume was 2.2 percent, or 0.132 cm^3 . The calculated average extractives in the cell wall then is 0.18 gram, and the measured average amount of extractives in red oak was 0.25 gram. Therefore, 72 percent of the extractives should be present in the cell walls of red oak.

Tarkow and Krueger (1961) also indicated that about three-fourths of the total amount of extractives was present in the cell walls of redwood. However, the present study shows that the value for redwood is one-third lower. Tarkow and Krueger's technique may not be accurate because residual growth stresses could cause appreciable errors (Arganbright 1971); nevertheless it is a simple and economical method to

estimate the amount of hot-water soluble extractives in the cell walls as well as void spaces of wood.

RELATIVE EFFICIENCY, ϕ , OF BULKING CHEMICALS

The amount of bulking chemicals impregnated into wood can be evaluated from the relative efficiency, ϕ ;

$$\phi = \frac{A_a}{A_m} \quad (6.4)$$

where A_a is the actual amount of bulking chemicals which are actually impregnated into wood and A_m is the maximum amount of bulking chemicals which can theoretically penetrate the wood. The value A_a can be measured directly through the techniques described in the experimental procedure. Assuming that the bulking chemicals are equally accessible to all the water molecules, then A_m can be calculated by the following equation:

$$A_m = \frac{W_f}{W_j + W_i} A_t \quad (6.5)$$

where W_i and W_f are the amount of water in the wood sample at the beginning and at the end of treatment, respectively; W_j is the amount of water in the jar in which the wood sample is submerged; and A_t is the total amount of bulking chemicals in the jar. The average values of relative efficiency, ϕ , for various samples are summarized in Table 6.23. The average specific gravities for the four species are given in Table

6.24, which was used as reference for predicting the amount of water in wood.

Table 6.23. Average values of relative efficiency of bulking chemical impregnation in southern pine sapwood and yellow poplar sapwood

Species	Bulking agent	W_i (g)	W_f (g)	A_m (g)	A_s (g)	ϕ
Southern pine	PEG (600)	4.91	3.18	2.55	1.91	0.75
Southern pine	Tannin	4.91	4.21	1.54	1.06	0.69
Yellow poplar	PEG (600)	4.87	2.92	2.35	1.84	0.78
Yellow poplar	Tannin	4.87	4.00	1.46	0.75	0.51

Table 6.24. Average specific gravities of extractive-free samples of four species

Wood type	Sample size	SG	Std
Redwood	3	0.297	0.015
Red oak	3	0.547	0.006
Southern pine	3	0.403	0.021
Yellow poplar	3	0.377	0.021

As an example, the value ϕ for southern pine is 0.75, which was derived from Eqs. 6.4 and 6.5. The initial average amount of water W_i in the control samples was 4.91 grams, and the final average amount of water W_f in the PEG samples was 3.18 grams. The amount of water in the jar W_j and the amount of PEG A_t were both 20 grams; therefore, the maximum amount

of PEG A_m which could penetrate the wood should be 2.55 grams, and the measured average amount of PEG A_a in southern pine was 1.91 grams. Hence, the value ϕ can be calculated from the ratio of A_a and A_m .

More PEG than tannin could penetrate wood (Tables 6.6 and 6.22) because the initial concentration of PEG was much higher (50 percent) than that of tannin (30 percent). It is very difficult to obtain high concentration of tannin solution without precipitation during treatment; but it is possible for more tannin to enter wood by adjusting the pH value of the solution with chemicals and hence reducing the precipitation. PEG is more efficient in its penetration because its ϕ value is high.

It is also possible to predict the locations of bulking chemicals in wood using the technique of Tarkow and Krueger. For example, the specific gravity of PEG 600 is 1.128 (Aldrich 1986). From Figure 6.8 for southern pine, the decrease in volumetric shrinkage was 10.6 percent, or 0.636 cm^3 . The calculated average amount of PEG in a drying sample is 0.72 gram. Compared with the value of A_a given in Table 6.23, the predicted PEG concentration is 62 percent in the cell voids, and 38 percent in the cell wall. Since the total amount of bulking chemicals penetrated into wood was much higher than that of natural extractives, the blocking effect, as expected, was much more significant. As a result, the corresponding diffusivities and permeabilities also decreased significantly.

CONCLUSIONS

- 1) Hot-water soaking to remove most of water-soluble extractives slightly increased the diffusivities of redwood and red oak heartwood. Impregnating southern pine and yellow poplar sapwood with polyethylene glycol or tannin considerably decreased their diffusivities.
- 2) Hot-water soaking increased radial, tangential, and volumetric shrinkages. Impregnating woods with the two bulking chemicals decreased the shrinkages, but the PEG treatment was more effective than the tannin treatment.
- 3) Hot-water soaking increased gas permeabilities in the radial direction, but did not show an increase in the longitudinal direction due to great variations between boards. Impregnating woods with the two bulking chemicals decreased their permeabilities significantly.
- 4) Based on volumetric shrinkage data, more water-soluble extractives in redwood and red oak heartwood stayed in the cell walls rather than the void spaces.

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SUMMARY AND CONCLUSIONS

A thorough understanding of the movement of moisture in wood during drying requires that all contributing factors be considered. Although natural extractives are minor chemical components of wood, they affect dryability. However, the effect on moisture movement is not large, therefore the diffusion coefficient must be determined accurately to obtain the true effect over the entire drying period.

This dissertation emphasized two aspects of the above problem. First, solutions of the diffusion model under various assumptions were studied using instrumentation and optimization techniques. Then, the effects of extractives were examined by comparing the diffusion coefficients among untreated and treated samples. The mechanism of the effect of extractives on the movement of moisture inside wood during drying was explained from shrinkage and permeability data.

In the preliminary study, an instrumentation method using the "continuous" measuring system has been demonstrated useful in reducing the experimental errors, and to record the drying process accurately. The drying rates of wood were readily derived from the recorded data of moisture removal. The application of this system alone, however, may not allow the average diffusion coefficient, which characterizes the overall drying process, to be found if it is calculated by the approximate method in the Boltzmann equation.

Optimization techniques proved useful for finding the appropriate solutions to the diffusion model under various assumptions. Based on the least squares principle, these solutions predict optimum drying curves over the entire drying period. The methods developed in this study allow the optimum diffusion coefficients to be calculated either theoretically or numerically. The effect of surface moisture content on diffusion can also be determined from the calculation.

When the optimum average diffusion coefficient was used as an index to evaluate the effects of several predrying treatments on the drying rate, the results indicated that steaming and hot-water soaking increased the diffusion coefficients of southern hardwoods above, and below, the fiber saturation point; but their effects were not as large as those due to differences in species and structural directions. Hot-water soaking tended to have the same effect as prolonged steaming. The increase in the diffusivity was due to removal and rearrangement of the extractives in wood.

The effects of bulking agents on the drying properties of wood suggested that the change in wood drying rate was mainly due to blockage of the macro-passages in the void spaces of wood. Hot-water soaking, which removed most of the water-soluble extractives, slightly increased the diffusion coefficients of redwood and red oak heartwood. Hot-water soaking also increased the gas permeability in the radial direction, but not in the longitudinal direction. The

shrinkage data indicate that most of the water-soluble extractives stayed inside the cell walls and did not block the passage of water. Bulking chemicals impregnated into southern pine and yellow poplar sapwood blocked the macro-passageways; as a result the moisture diffusion coefficients decreased significantly.

APPENDIX A

FORTRAN PROGRAM FOR LOCATING OPTIMUM AVERAGE DIFFUSION COEFFICIENT


```

C PROGRAM FOR CALCULATING OPTIMUM AVERAGE DIFFUSION COEFFICIENT (ADC)
C
C FEATURES
C UNI-DIRECTIONAL OPTIMUM ADC OF A RECTANGULAR WOOD SAMPLE
C CALCULATED BY ASSUMING NEGLIGIBLE SURFACE RESISTANCE. GOLDEN
C SECTION SEARCH TECHNIQUE AND LEAST SQUARES CRITERION ARE APPLIED.
C ALL TERMS IN PARAMETER DECLARATION CAN BE ADJUSTED FOR EACH
C INDIVIDUAL SITUATION.
C
C ..... ARGUMENT LIST .....
C
C DFC  -- ADC ARRAY
C DLB  -- LOWER BOUND OF CHOSEN ADC
C DSN  -- ADC ARRAY SUBSCRIPT
C DUB  -- UPPER BOUND OF CHOSEN ADC
C EMS  -- E CALCULATED FROM MEASURED DATA
C ETR  -- E CALCULATED FROM TRIAL DATA
C HFT  -- HALF THICKNESS OF A SAMPLE IN CENTIMETER
C ITEM -- NUMBER OF DATA POINTS
C SS   -- SUM OF SQUARE OF THE DIFFERENCE BETWEEN EMS AND ETR
C TIME -- DRYING TIME DATA
C TSN  -- TIME ARRAY SUBSCRIPT
C SUMAE - FUNCTION FOR CALCULATING AVERAGE E
C SUMSQ - SUBROUTINE FOR CALCULATING SUM OF SQUARE
C
C ..... DECLARATIONS .....
C      PARAMETER (HFT=1.27,DLB=1.E-8,DUB=1.E-4,ITEM=9)
C      INTEGER TSN, DSN
C      REAL TIME(30),EMS(30),ETR(4,30),SS(4),DFC(4)
C
C ..... READ MEASURED DATA IN .....
C      READ *, (TIME(I),EMS(I),I=1,ITEM)
C
C ..... CALCULATE ADCS .....
C      DFC(1)=DLB
C      DFC(2)=DUB
C      DFC(3)=DFC(1)+(DFC(2)-DFC(1))*0.381966
C      DFC(4)=DFC(1)+(DFC(2)-DFC(1))*0.618034
C
C ..... CALCULATE SUM OF SQUARE .....
C      DO DSN=1,4
C        DO TSN=1,ITEM
C          ETR(DSN,TSN)=SUMAE(HFT,TIME,DFC,TSN,DSN)
C        ENDDO
C        CALL SUMSQ(SS,EMS,ETR,DSN,ITEM)
C      ENDDO
C
C ..... CALCULATE OPTIMUM ADC.....
C      DO WHILE ((DFC(2)-DFC(1)).GT.1.E-10)
C        IF (SS(3).GT.SS(4)) THEN
C          DFC(1)=DFC(3)
C          DFC(3)=DFC(4)
C          DFC(4)=DFC(1)+(DFC(2)-DFC(1))*0.618034
C          SS(1)=SS(3)
C          SS(3)=SS(4)
C          DSN=4
C          DO TSN=1,ITEM
C            ETR(DSN,TSN)=SUMAE(HFT,TIME,DFC,TSN,DSN)
C          ENDDO
C          CALL SUMSQ(SS,EMS,ETR,DSN,ITEM)
C        ELSE
C          DFC(2)=DFC(4)

```

```

      DFC(4)=DFC(3)
      DFC(3)=DFC(1)+(DFC(2)-DFC(1))* .381966
      SS(2)=SS(4)
      SS(4)=SS(3)
      DSN=3
      DO TSN=1,ITEM
        ETR(DSN,TSN)=SUMAE(HFT,TIME,DFC,TSN,DSN)
      ENDDO
      CALL SUMSQ(SS,EMS,ETR,DSN,ITEM)
    ENDIF
  ENDDO
C
C ..... PRINT OUT RESULTS .....
C   IF (SS(3).GT.SS(4)) THEN
C     PRINT *, 'LOWER ADC LIMIT: ',DFC(3)
C     PRINT *, 'UPPER ADC LIMIT: ',DFC(2)
C     PRINT *, 'OPTIMUM ADC:',DFC(4)
C     PRINT *, 'SS AT OPTIMUM ADC:',SS(4)
C   ELSE
C     PRINT *, 'LOWER ADC LIMIT: ',DFC(1)
C     PRINT *, 'UPPER ADC LIMIT: ',DFC(4)
C     PRINT *, 'OPTIMUM ADC:',DFC(3)
C     PRINT *, 'SS AT OPTIMUM ADC:',SS(3)
C   ENDIF
C   STOP
C   END
C
C -----
C   FUNCTION SUMAE(HFT,TIME,DFC,TSN,DSN)
C
C   AVERAGE E IS CALCULATED BASED ON THEORETICAL FOURIER SERIES
C   SOLUTION OF DIFFUSION EQUATION.
C
C   ..... DECLARATIONS .....
C     INTEGER TSN,DSN
C     REAL TIME(*),DFC(*)
C     REAL CTERM,EXPTM,TERM
C
C   ..... INITIALIZATION .....
C     SUMAE=0.
C     TERM=1.
C     I=0
C
C   ..... CALCULATE AVERAGE E .....
C     DO WHILE ((I.LT.100).AND.(TERM.GE.1.E-30))
C       I=I+1
C       CTERM=(ACOS(-1.))**2*((2.*I-1.))**2
C       EXPTM=900.*CTERM*TIME(TSN)*DFC(DSN)/(HFT**2)
C       IF (EXPTM.GT.100.) EXPTM=100.
C       EXPTM=EXP(-EXPTM)
C       TERM=8.*EXPTM/CTERM
C       SUMAE=SUMAE+TERM
C     ENDDO
C     RETURN
C     END
C
C -----
C   SUBROUTINE SUMSQ(SS,EMS,ETR,DSN,ITEM)

```

```
C
C SUM OF SQUARE IS CALCULATED FOR ADC.
C
C ..... DECLARATIONS .....
C          INTEGER DSN,ITEM
C          REAL EMS(*),ETR(4,*),SS(*)
C          REAL LSS
C
C ..... CALCULATE SUM OF SQUARE .....
C          LSS=0.
C          DO I=1,ITEM
C              LSS=LSS+(EMS(I)-ETR(DSN,I))**2
C          ENDDO
C          SS(DSN)=LSS
C          RETURN
C          END
```

APPENDIX B

FORTRAN PROGRAM FOR DETERMINING DIFFUSION COEFFICIENT AND SURFACE EMISSION COEFFICIENT

```

C PROGRAM FOR CALCULATING THE OPTIMUM DIFFUSION
C COEFFICIENT D AND SURFACE EMISSION COEFFICIENT S
C
C FEATURES
C UNI-DIRECTIONAL OPTIMUM D AND S OF A RECTANGULAR
C WOOD SAMPLE ARE CALCULATED BASED ON NEWMAN'S
C THEORETICAL SOLUTION. CONJUGATE GRADIENT SEARCH
C TECHNIQUE AND LEAST SQUARES CRITERION ARE APPLIED.
C
C ..... ARGUMENT LIST .....
C
C A      -- HALF THICKNESS VARIABLE
C AD     -- D VALUE
C AL     -- L VALUE
C EEV    -- E CALCULATED FROM MEASURED DATA
C FMS    -- FINAL MEAN SUM OF SQUARES
C G      -- GRADIENT ARRAY
C HLFT   -- HALF THICKNESS OF A SAMPLE
C ITN    -- NUMBER OF DATA POINT VARIABLE
C LD     -- LOG(L) AND LOG(D) ARRAY
C NP     -- NUMBER OF DATA POINTS
C NMSS   -- NEGATIVE MEAN SUM OF SQUARES
C S      -- STOPPING CRITERION VARIABLE
C TIME   -- DRYING TIME DATA
C
C ..... DECLARATIONS .....
C      PARAMETER (NP=20,HLFT=1.431)
C      COMMON TIME(30),EEV(30),A,ITN
C      INTEGER ITN
C      REAL TIME,EEV,A,LD(2),G(2),NMSS,S,AL,AD,FMS
C
C THE FOLLOWING INITIAL VALUES FOR LOG(L) AND LOG(D)
C MAY BE CHANGED.
C      DATA LD/1.,-5./
C
C ..... INITIALIZATION .....
C      ITN=NP
C      A=HLFT
C      READ *,(TIME(I),EEV(I),I=1,ITN)
C
C ..... SEARCH FOR OPTIMUM L AND D
C      DO J=1,20
C        CALL GRAD(LD,G)
C        S=0.
C        DO I=1,2
C          S=S+G(I)*G(I)
C        ENDDO
C        IF (S.LT..00001) GOTO 1
C        CALL DIR(G)
C        CALL LSRCH(LD,G)
C        AL=10.**LD(1)
C        AD=10.**LD(2)
C        PRINT *, 'ITERATION ',J,'L=',AL,'D=',AD,'S=',S
C      ENDDO
C
C PRINT OUT THE FINAL SEARCHING RESULT
C      1 FMS=NMSS(LD)
C      PRINT *, 'FINAL MEAN SUM OF SQUARE',FMS
C      PRINT *, 'DIFFUSION COEFFICIENT =',AD
C      PRINT *, 'SURFACE EMISSION COEFFICIENT=',AL*AD/A
C      STOP
C      END

```

```

C
C -----
      SUBROUTINE GRAD(LD,G)
C
C CALCULATE THE SEARCHING GRADIENT
C
C ..... DECLARATIONS .....
      REAL LD(2),LDV(2),G(2),H,NMSS
C
C ..... CALCULATION .....
      H=.0001
      LDV(1)=LD(1)+H
      LDV(2)=LD(2)
      G(1)=(NMSS(LDV)-NMSS(LD))/H
      LDV(1)=LD(1)
      LDV(2)=LD(2)+H
      G(2)=(NMSS(LDV)-NMSS(LD))/H
      RETURN
      END
C
C -----
      SUBROUTINE DIR(D)
C
C COMPUTE THE CONJUGATE GRADIENT
C
C ..... DECLARATIONS .....
      REAL D(2),Q(2),R(2),S,NMSS,C
      DATA K/1/,R/2*0./,Q/2*1./
C
C ..... INITIALIZATION .....
      S=0.
      NMSS=0.
C
C ..... SEARCHING BEGINS .....
      DO I=1,2
        S=S+D(I)*D(I)
        NMSS=NMSS+Q(I)*Q(I)
      ENDDO
      C=S/NMSS
      DO I=1,2
        Q(I)=D(I)
        D(I)=D(I)+C*R(I)
        R(I)=D(I)
      ENDDO
      K=K+1
      IF(K.LT.7) RETURN
      K=1
      R(1)=0.
      R(2)=0.
      RETURN
      END
C
C -----
      SUBROUTINE LSRCH (LD,D)
C
C SEARCH OUT X+TD UNTIL DECREASE
C
C ..... DECLARATIONS .....
      INTEGER I,IX
      REAL LD(2),D(2),Y(2),Z(2),W(2)
      REAL T,NMSS,ZZ,SSQY,SSQZ
C

```

```

C ..... INITIALIZATION .....
      T=.001
      DO I=1,2
        Y(I)=LD(I)+T*D(I)
        Z(I)=LD(I)+.33*T*D(I)
      ENDDO
      SSQY=NMSS(Y)
      SSQZ=NMSS(Z)

C
C ..... DEFINE THE SEARCHING AREA .....
      DO WHILE ((NMSS(Y).GE.NMSS(Z)))
        T=T*2.
        DO I=1,2
          ZZ=Y(I)
          Y(I)=LD(I)+T*D(I)
          LD(I)=Z(I)
          Z(I)=ZZ
        ENDDO
        SSQY=NMSS(Y)
        SSQZ=NMSS(Z)
      ENDDO

C
C ..... LOCATE THE OPTIMUM POINT WITHIN ABOVE AREA
      DO IX=1,15
        DO I=1,2
          W(I)=LD(I)+.382*T*D(I)
          Z(I)=LD(I)+.618*T*D(I)
        ENDDO
        T=T*.618
        SSQY=NMSS(W)
        SSQZ=NMSS(Z)
        IF (NMSS(Z).GE.NMSS(W)) THEN
          LD(1)=W(1)
          LD(2)=W(2)
        ENDIF
      ENDDO
      RETURN
      END

C
C -----
      REAL FUNCTION NMSS(LD)

C
C CALCULATE NEGATIVE MEAN SUM OF SQUARES OF THE
C DIFFERENCE BETWEEN PREDICTED AND EXPERIMENTAL E VALUES.
C
C ..... DECLARATIONS .....
      COMMON TIME(30),EEV(30),A,ITN
      INTEGER ITN
      REAL TIME,EEV,A
      REAL TM,CEV,DFV,LLD(2),LSS,E

C
C ..... INITIALIZATION .....
      LLD(1)=10.**LD(1)
      LLD(2)=10.**LD(2)
      LSS=0.

C
C ..... CALCULATE THE SUM OF SQUARES .....
      DO I=1,ITN
        TM=TIME(I)
        CEV=E(LLD,TM,A)
        DFV=EEV(I)-CEV
        IF (ABS(DFV).LT.1.E-30) DFV=1.E-30

```

```

        LSS=LSS+DFV**2
        ENDDO
        NMSS=-LSS/ITN
        RETURN
        END
C
C -----
        FUNCTION E(LD,T,A)
C
C CALCULATE A VALUE OF E
C
C ..... DECLARATIONS .....
        REAL LD(*),T,A,BETA
        REAL L,D,CTM,ETM,TERM,B,SUM
C
C ..... INITIALIZATION .....
        L=LD(1)
        D=LD(2)
        TERM=1.
        I=0
        SUM=0.
C ..... CALCULATE E .....
        DO WHILE ((I.LT.100).AND.(TERM.GE.1.E-30))
            I=I+1
            B=BETA(L,I)
            CTM=2.*L**2/B**2/(B**2+L**2+L)
            ETM=B**2*D*3600.*T/A**2
            IF (ETM.GT.100.) ETM=100.
            ETM=EXP(-ETM)
            TERM=CTM*ETM
            SUM=SUM+TERM
        ENDDO
        E=SUM
        RETURN
        END
C
C -----
        FUNCTION BETA(L,N)
C
C CALCULATE THE POSITIVE ROOT
C
C ..... DECLARATIONS .....
        INTEGER N
        REAL L,PI,FL,FM,FU,BL,BM,BU
        REAL ROOT,B,LL
        ROOT(B,LL)=LL-B*TAN(B)
C
C ..... SEARCH THE INTERVAL IN WHICH A BETA IS LOCATED .....
        PI=ACOS(-1.)
        BL=(1.*N-1.5)*PI
        BU=(1.*N-.5)*PI
        IF (BL.LT.0.) BL=0.
        FL=1.
        FU=-1.
C
C ..... CALCULATE THE BETA .....
        DO I=1,15
            BM=(BL+BU)/2.
            FM=ROOT(BM,L)
            IF (FL*FM.LT.0.) THEN
                FU=FM
                BU=BM
            
```



```
ELSE
  FL=FM
  BL=BM
ENDIF
ENDDO
BETA=(BL+BU)/2.
RETURN
END
```

APPENDIX C.1

AVERAGE VALUES OF INITIAL AND EQUILIBRIUM MOISTURE CONTENT OF SIX SOUTHERN HARDWOODS

Species	Treatment	Longitudinal						Radial			Tangential		
		Initial MC	Final ^a		Initial MC	Final ^b		Initial MC	Final		Initial MC	Final	
			MC(1)	MC(2)		MC(1)	MC(2)		MC(1)	MC(2)		MC(1)	MC(2)
Red oak	A	107.34	32.60	5.15	105.50	34.31	6.52	112.16	35.93	6.13	112.16	35.93	6.13
	B	102.40	29.26	5.30	103.27	34.65	6.04	108.25	34.40	6.69	108.25	34.40	6.69
	C	118.27	31.12	5.09	116.37	33.61	5.54	107.88	35.24	6.31	107.88	35.24	6.31
	D	111.29	31.45	4.82	104.32	33.64	6.20	112.20	34.61	6.20	112.20	34.61	6.20
	E	111.89	30.39	5.34	106.12	33.50	5.88	105.93	34.62	6.93	105.93	34.62	6.93
White oak	A	120.44	30.04	5.88	109.60	30.87	6.77	109.85	30.61	6.80	109.85	30.61	6.80
	B	122.74	30.56	5.67	121.75	31.14	6.21	120.45	32.17	6.29	120.45	32.17	6.29
	C	127.13	30.64	5.73	119.15	31.54	7.01	117.93	31.72	6.05	117.93	31.72	6.05
	D	119.27	29.47	5.62	112.41	30.69	6.42	119.41	31.16	6.31	119.41	31.16	6.31
	E	121.83	30.02	5.71	113.96	30.75	6.41	116.41	31.98	6.00	116.41	31.98	6.00
Elm	A	149.59	30.31	4.96	156.34	28.77	5.24	146.69	32.60	5.52	146.69	32.60	5.52
	B	152.88	29.94	5.31	158.83	31.58	5.33	144.81	32.80	5.30	144.81	32.80	5.30
	C	146.65	30.10	4.77	144.99	30.68	5.29	154.20	32.03	5.17	154.20	32.03	5.17
	D	151.76	30.72	5.20	151.42	31.34	5.16	148.39	33.12	5.26	148.39	33.12	5.26
	E	155.21	29.16	5.16	147.42	30.19	5.42	151.59	30.12	5.41	151.59	30.12	5.41

^a Equilibrium moisture content at 45° and 97 percent RH.

^b Equilibrium moisture content at 45° and 30 percent RH.

Continued

Continued												
Species	Treatment	Longitudinal				Radial			Tangential			
		Initial MC	Final MC(1)	Final MC(2)	Initial MC	Final MC(1)	Final MC(2)	Initial MC	Final MC(1)	Final MC(2)		
Sweetgum	A	158.05	30.80	5.25	138.95	31.21	6.16	143.01	32.35	6.08		
	B	158.98	29.47	5.20	152.24	32.64	6.01	152.32	30.31	6.17		
	C	163.25	29.27	4.90	159.41	31.24	5.92	147.84	30.52	6.01		
	D	153.00	30.50	4.77	143.62	31.34	5.79	146.85	30.56	6.27		
	E	159.87	30.45	5.50	151.21	29.90	5.75	139.70	29.91	5.77		
Willow	A	257.23	30.39	5.13	251.87	31.95	5.93	256.65	29.86	6.06		
	B	256.63	32.75	5.29	254.47	30.84	5.91	258.27	31.38	5.95		
	C	251.30	31.94	5.32	260.48	31.91	5.29	249.45	33.36	5.77		
	D	257.52	29.29	5.50	250.52	30.94	5.97	248.48	31.79	5.86		
	E	249.89	30.86	5.35	257.26	31.75	5.77	250.88	32.83	5.91		
Cottonwood	A	218.31	29.45	5.37	191.64	30.17	5.53	189.34	32.48	5.85		
	B	185.88	32.16	4.83	189.21	31.86	5.26	197.43	31.61	5.67		
	C	201.30	30.07	5.19	175.86	30.15	5.29	205.33	31.84	5.49		
	D	184.32	29.81	4.81	187.98	31.65	5.40	199.38	32.98	5.64		
	E	221.88	30.18	5.18	175.91	31.88	5.25	190.27	31.79	5.56		

APPENDIX C.2
AVERAGE \bar{E} VALUES OF SIX SOUTHERN HARDWOODS

Red Oak

Drying Phase	TRT ^a	DIR ^b	Drying Time (Hours)																715	907
			0	1	3	7	13	19	31	43	67	91	139	187	235	331	427	523		
1	A	L	1.00	.99	.97	.96	.92	.87	.82	.73	.57	.46	.32	.26	.23	.20	.17	.15	.09	.06
		R	1.00	.97	.97	.96	.93	.90	.84	.78	.65	.56	.44	.35	.31	.26	.21	.16	.10	.07
		T	1.00	.99	.97	.95	.91	.88	.79	.73	.64	.58	.46	.37	.33	.28	.22	.18	.11	.07
		L	1.00	.99	.97	.95	.89	.85	.77	.71	.56	.46	.34	.26	.23	.20	.18	.15	.11	.07
	B	R	1.00	.99	.97	.95	.92	.89	.82	.76	.64	.54	.39	.31	.25	.20	.15	.12	.06	.04
		T	1.00	.97	.95	.92	.88	.84	.76	.72	.62	.54	.42	.33	.29	.24	.19	.15	.09	.06
		L	1.00	.99	.98	.96	.90	.86	.77	.71	.57	.46	.32	.24	.21	.19	.16	.13	.08	.05
		R	1.00	.99	.97	.94	.91	.87	.79	.75	.61	.50	.35	.27	.24	.18	.14	.12	.09	.07
	C	T	1.00	.97	.95	.93	.87	.83	.77	.72	.60	.54	.42	.33	.28	.22	.16	.14	.10	.07
		L	1.00	.99	.99	.97	.90	.87	.79	.71	.55	.41	.29	.23	.20	.17	.14	.12	.07	.05
		R	1.00	.96	.96	.93	.90	.86	.80	.74	.59	.52	.32	.27	.24	.21	.17	.15	.09	.06
		T	1.00	.98	.96	.93	.88	.85	.75	.69	.58	.52	.41	.32	.26	.20	.15	.13	.09	.06

		Drying Time (Hours)												56	72	104
		0	2	4	6	8	12	16	24	32	40					
A	L	1.000	.793	.646	.520	.432	.309	.214	.051	.011	.010	.007	.005	.004		
	R	1.000	.894	.850	.791	.770	.716	.641	.581	.542	.491	.404	.336	.223		
	T	1.000	.923	.882	.829	.802	.734	.656	.582	.543	.500	.437	.390	.286		
	L	1.000	.714	.572	.465	.359	.272	.186	.095	.053	.028	.010	.006	.004		
B	R	1.000	.924	.874	.822	.788	.713	.637	.535	.460	.386	.313	.282	.210		
	T	1.000	.928	.878	.818	.785	.718	.670	.558	.494	.459	.376	.325	.225		
	L	1.000	.705	.487	.350	.267	.174	.124	.058	.022	.012	.008	.006	.004		
	R	1.000	.877	.807	.749	.714	.646	.572	.477	.418	.354	.275	.229	.145		
C	T	1.000	.876	.831	.772	.755	.702	.632	.576	.527	.470	.380	.313	.204		
	L	1.000	.737	.572	.377	.300	.183	.061	.031	.017	.010	.005	.003	.002		
	R	1.000	.877	.817	.762	.734	.675	.605	.539	.495	.449	.360	.300	.195		
	T	1.000	.885	.836	.787	.755	.685	.618	.515	.432	.356	.261	.207	.120		
D	L	1.000	.674	.561	.419	.346	.256	.163	.082	.042	.022	.009	.006	.004		
	R	1.000	.963	.818	.747	.695	.621	.581	.484	.421	.381	.296	.249	.161		
	T	1.000	.886	.845	.790	.776	.728	.662	.612	.569	.516	.431	.366	.253		
	L	1.000	.793	.646	.520	.432	.309	.214	.051	.011	.010	.007	.005	.004		

^a Treatments.

^b Directions of moisture movement during drying.

Continued

Continued

White Oak

Drying Phase	TRT	DIR	Drying Time (Hours)																	
			0	1	3	7	13	19	31	43	67	91	139	187	235	331	427	523	715	907
1	A	L	1.00	.98	.97	.96	.94	.92	.86	.81	.70	.60	.43	.30	.23	.17	.12	.11	.07	.05
		R	1.00	.97	.97	.95	.92	.90	.85	.81	.73	.67	.55	.47	.41	.29	.19	.13	.07	.05
		T	1.00	.98	.96	.84	.90	.85	.79	.75	.67	.61	.51	.43	.39	.30	.24	.18	.10	.06
		L	1.00	.98	.97	.96	.93	.91	.85	.80	.69	.60	.40	.28	.23	.18	.14	.11	.07	.05
	B	R	1.00	.98	.96	.96	.93	.89	.83	.78	.69	.63	.51	.44	.38	.28	.19	.15	.08	.05
		T	1.00	.98	.97	.94	.91	.88	.81	.75	.68	.61	.51	.42	.36	.26	.19	.14	.07	.04
		L	1.00	.98	.97	.94	.91	.89	.82	.77	.66	.57	.40	.28	.23	.17	.13	.12	.07	.05
		R	1.00	.98	.97	.94	.91	.88	.82	.77	.69	.63	.52	.43	.35	.24	.16	.11	.08	.05
	C	T	1.00	.98	.97	.96	.91	.89	.81	.77	.68	.62	.51	.40	.34	.25	.17	.12	.06	.03
		L	1.00	.97	.96	.94	.91	.88	.82	.76	.65	.56	.41	.29	.24	.18	.12	.10	.06	.04
		R	1.00	.97	.96	.94	.91	.88	.82	.77	.68	.62	.50	.40	.34	.25	.17	.13	.08	.05
		T	1.00	.99	.97	.95	.92	.89	.83	.77	.69	.63	.51	.41	.35	.24	.15	.12	.06	.04
2	A	L	1.000	.754	.630	.630	.484	.436	.355	.264	.156	.095	.095	.095	.055	.020	.010	.004	.004	.004
		R	1.000	.866	.794	.794	.729	.695	.621	.549	.450	.383	.383	.383	.322	.257	.227	.172	.172	.172
		T	1.000	.946	.904	.904	.857	.823	.758	.705	.582	.498	.498	.498	.465	.387	.347	.256	.256	.256
		L	1.000	.790	.595	.595	.409	.343	.252	.204	.121	.069	.069	.069	.045	.024	.018	.013	.013	.013
	B	R	1.000	.854	.776	.776	.710	.669	.591	.509	.402	.335	.335	.335	.269	.193	.156	.100	.100	.100
		T	1.000	.958	.898	.898	.832	.802	.710	.595	.528	.499	.499	.499	.463	.386	.317	.209	.209	.209
		L	1.000	.669	.554	.554	.410	.336	.246	.155	.077	.039	.039	.039	.021	.009	.006	.004	.004	.004
		R	1.000	.840	.774	.774	.698	.662	.584	.489	.397	.337	.337	.337	.275	.185	.130	.068	.068	.068
	D	T	1.000	.902	.843	.843	.782	.737	.652	.582	.433	.329	.329	.329	.283	.193	.156	.092	.092	.092
		L	1.000	.783	.585	.585	.396	.330	.238	.192	.112	.063	.063	.063	.041	.023	.018	.013	.013	.013
		R	1.000	.940	.785	.785	.707	.650	.565	.516	.406	.336	.336	.336	.292	.210	.172	.107	.107	.107
		T	1.000	.834	.749	.749	.685	.630	.535	.469	.334	.266	.266	.266	.217	.134	.107	.069	.069	.069
E	L	1.000	.782	.644	.644	.523	.420	.264	.187	.053	.019	.019	.019	.015	.007	.007	.006	.006	.006	
	R	1.000	.855	.785	.785	.723	.689	.620	.542	.463	.409	.409	.409	.349	.265	.211	.125	.125	.125	
	T	1.000	.826	.749	.749	.670	.633	.559	.476	.394	.363	.363	.363	.338	.279	.224	.138	.138	.138	

Continued

Continued

Elm

Drying Phase	TRT	DIR	Drying Time (Hours)																	
			0	1	3	7	13	19	31	43	67	91	139	187	235	331	427	523	715	907
1	A	L	1.00	.99	.98	.97	.94	.92	.87	.82	.72	.60	.43	.32	.28	.22	.17	.12	.06	.04
		R	1.00	.99	.97	.97	.95	.93	.89	.85	.78	.70	.56	.43	.32	.23	.16	.11	.05	.03
		T	1.00	.99	.98	.96	.94	.90	.86	.83	.76	.70	.58	.47	.40	.28	.19	.14	.08	.05
		L	1.00	.98	.97	.96	.94	.91	.86	.81	.70	.59	.43	.32	.28	.23	.17	.13	.07	.05
	B	R	1.00	.99	.98	.97	.94	.91	.84	.81	.72	.66	.52	.40	.35	.26	.19	.13	.06	.04
		T	1.00	.99	.99	.97	.96	.93	.88	.84	.74	.66	.51	.39	.34	.27	.20	.15	.08	.05
		L	1.00	.98	.98	.97	.94	.90	.85	.81	.68	.58	.41	.30	.26	.21	.15	.11	.05	.03
		R	1.00	.98	.98	.98	.97	.97	.91	.86	.78	.66	.50	.37	.30	.22	.16	.12	.06	.04
	C	T	1.00	.98	.98	.97	.95	.93	.89	.85	.76	.67	.53	.41	.33	.24	.17	.12	.05	.03
		L	1.00	.99	.99	.96	.92	.90	.86	.81	.72	.61	.45	.33	.28	.21	.14	.10	.05	.03
		R	1.00	.99	.98	.97	.95	.93	.88	.82	.73	.65	.47	.37	.32	.25	.17	.14	.07	.05
		T	1.00	.99	.99	.97	.93	.91	.86	.83	.73	.64	.50	.38	.33	.25	.17	.13	.07	.05

Drying Time (Hours)

	0	2	4	6	8	12	16	24	32	40	56	72	104
A	L	1.000	.647	.408	.262	.179	.100	.069	.032	.011	.008	.007	.004
	R	1.000	.745	.625	.505	.424	.306	.186	.096	.064	.040	.020	.017
	T	1.000	.754	.658	.561	.512	.408	.301	.202	.144	.097	.053	.042
B	L	1.000	.606	.378	.220	.150	.095	.056	.031	.022	.018	.014	.009
	R	1.000	.703	.570	.461	.390	.270	.165	.083	.059	.042	.033	.020
	T	1.000	.771	.675	.589	.526	.410	.313	.178	.098	.040	.006	.003
C	L	1.000	.528	.364	.203	.134	.081	.032	.018	.012	.009	.006	.004
	R	1.000	.663	.510	.368	.278	.162	.059	.013	.014	.012	.012	.010
	T	1.000	.758	.614	.486	.407	.248	.095	.012	.004	.003	.003	.002
D	L	1.000	.563	.300	.155	.084	.036	.031	.020	.007	.007	.006	.004
	R	1.000	.697	.572	.452	.383	.261	.149	.069	.049	.033	.026	.023
	T	1.000	.670	.529	.402	.329	.210	.112	.462	.036	.023	.015	.010
E	L	1.000	.611	.430	.254	.194	.124	.066	.029	.020	.012	.007	.006
	R	1.000	.700	.561	.428	.340	.219	.107	.040	.028	.019	.015	.013
	T	1.000	.715	.593	.479	.414	.300	.195	.109	.100	.042	.007	.004

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Continued

Continued

Sweetgum

Drying Phase	TRT	DIR	Drying Time (Hours)																	
			0	1	3	7	13	19	31	43	67	91	139	187	235	331	427	523	715	907
1	A	L	1.00	.99	.99	.97	.94	.91	.86	.82	.74	.64	.48	.33	.24	.13	.09	.08	.04	.02
		R	1.00	.99	.99	.98	.95	.92	.88	.84	.74	.65	.49	.35	.28	.18	.11	.09	.06	.05
		T	1.00	.99	.99	.98	.96	.94	.88	.83	.73	.65	.50	.38	.31	.19	.12	.08	.04	.03
		L	1.00	.99	.98	.96	.93	.92	.87	.83	.73	.65	.48	.32	.22	.11	.08	.07	.05	.03
	B	R	1.00	.99	.99	.97	.95	.93	.89	.84	.74	.66	.49	.33	.24	.14	.11	.09	.06	.05
		T	1.00	.99	.99	.97	.96	.94	.88	.84	.73	.65	.47	.33	.24	.16	.11	.10	.06	.04
		L	1.00	.99	.99	.97	.93	.90	.85	.80	.68	.59	.43	.27	.19	.13	.12	.09	.05	.03
		R	1.00	.99	.99	.98	.96	.93	.88	.82	.70	.62	.45	.33	.25	.18	.13	.09	.05	.03
	C	T	1.00	.98	.98	.96	.93	.90	.86	.82	.72	.62	.47	.34	.25	.16	.11	.08	.04	.03
		L	1.00	.98	.98	.96	.93	.91	.85	.80	.72	.61	.42	.26	.18	.14	.11	.10	.06	.04
		R	1.00	.99	.98	.97	.94	.92	.87	.83	.74	.66	.49	.35	.22	.11	.08	.07	.05	.04
		T	1.00	.99	.99	.98	.95	.91	.87	.81	.69	.62	.44	.32	.27	.17	.10	.08	.04	.03
2	A	L	1.000	.664	.502	.332	.272	.189	.114	.051	.029	.015	.007	.006	.004					
		R	1.000	.812	.715	.637	.584	.488	.391	.270	.202	.142	.090	.079	.056					
		T	1.000	.811	.716	.644	.583	.478	.405	.263	.196	.153	.085	.072	.051					
		L	1.000	.609	.382	.225	.154	.098	.058	.032	.023	.018	.014	.012	.009					
	B	R	1.000	.881	.701	.604	.530	.420	.353	.226	.156	.125	.077	.074	.060					
		T	1.000	.845	.762	.682	.622	.512	.423	.252	.142	.102	.040	.035	.026					
		L	1.000	.642	.392	.182	.115	.054	.049	.038	.027	.022	.019	.017	.013					
		R	1.000	.712	.583	.477	.407	.288	.182	.096	.068	.047	.042	.030	.022					
	C	T	1.000	.814	.729	.640	.585	.468	.353	.230	.167	.118	.077	.070	.053					
		L	1.000	.527	.362	.201	.132	.080	.032	.018	.012	.009	.007	.006	.004					
		R	1.000	.819	.729	.632	.569	.472	.359	.255	.195	.142	.077	.046	.019					
		T	1.000	.834	.746	.656	.598	.490	.410	.253	.168	.128	.065	.052	.033					
D	L	1.000	.612	.431	.254	.194	.124	.067	.029	.020	.012	.007	.006	.004						
	R	1.000	.839	.643	.533	.449	.325	.254	.132	.078	.063	.043	.032	.022						
	T	1.000	.781	.686	.593	.545	.451	.354	.258	.226	.208	.176	.147	.101						
	E																			

Continued

Willow

Continued

VITA

Yong Chen was born on March 18, 1955 in Beijing, the People's Republic of China. He received a B.S. degree in Wood Science and Technology from Nanjing Forestry University in China in January, 1981. Mr. Chen then worked in Nanjing Forestry University as a teacher for five years. He received a M.S. degree in Wood Science and Technology from the University of Minnesota in December, 1988. Mr. Chen is presently a candidate for the Ph.D degree in Forestry, majoring in Wood Science and Technology.

DOCTORAL EXAMINATION AND DISSERTATION REPORT

Candidate: Yong Chen

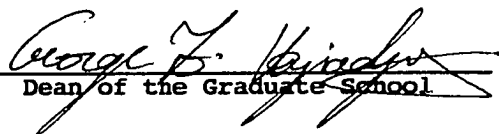
Major Field: Forestry

Title of Dissertation: APPLICATION OF OPTIMUM SOLUTIONS OF A DIFFUSION MODEL TO EVALUATE THE EFFECTS OF EXTRACTIVES IN WOOD DRYING

Approved:

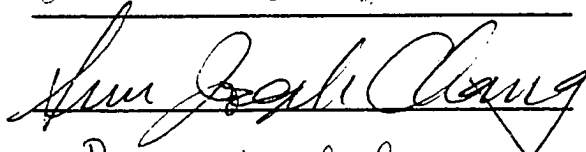
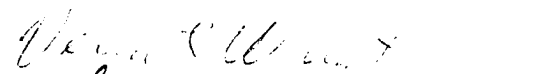
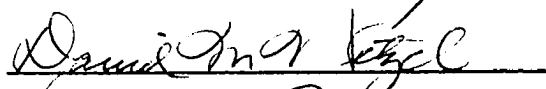
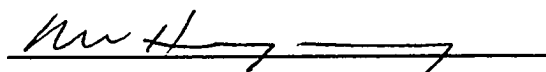


Major Professor and Chairman



Dean of the Graduate School

EXAMINING COMMITTEE:



Barun K. S. Gupta

Date of Examination:

December 10, 1993