

EQUILIBRIUM MOISTURE CONTENT OF WOOD IN HIGH-TEMPERATURE PRESSURIZED ENVIRONMENTS

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(Received December 1999)

ABSTRACT

Experiments were conducted on the water sorption characteristics of three wood species, for both juvenile wood and mature wood, at conditions above and below 100°C. A pressurized chamber was constructed for this purpose. At 50°C, equilibrium moisture content (EMC) behavior deviated only slightly from predictions based on the published data for Sitka spruce. At 160°C, the sorption behavior was distinctly different from the 50°C data, or any extrapolation from published low-temperature data. The data suggested that a change in the sorptive properties of the wood occurred as temperature and moisture conditions exceeded the glass transition temperature for lignin. At 50°C, juvenile wood tended to equilibrate at a higher moisture content than mature wood. At 160°C, however, juvenile wood exhibited a markedly lower EMC than mature wood. Thermal degradation of wood was detected during the experiments. Reduction in the sorptive behavior as a result of thermal degradation is proposed as a possible explanation for differences in EMC behavior for juvenile and mature wood at 160°C.

Keywords: EMC (equilibrium moisture content), wood, thermal degradation, high-temperature, sorption.

INTRODUCTION

This research effort focused on investigations of the wood-water relationship at conditions relevant to the hot pressing of wood-based composites. Very little information has been documented on the equilibrium moisture content (EMC) behavior of wood in environments of elevated temperature and pressure. Such data are essential in understanding the influence of moisture on the viscoelastic behavior of wood. The specific objectives of this study were: to develop apparatus and techniques suitable for collecting sorption isotherm data at high temperatures and pressures,

and to characterize the EMC vs. relative humidity behavior of selected wood species in environments relevant to hot pressing.

BACKGROUND

During the hot pressing of wood-based composites, internal mat temperature has been measured in excess of 150°C and relative humidity calculated to reach or exceed 75% (Humphrey and Bolton 1989; Kamke and Casey 1989; Kamke and Wolcott 1991). In some cases, saturation occurs (Kamke and Johnson 1994). Such severe processing conditions are adequate to transcend the glass transition of the viscoelastic wood polymers (Kelly et al. 1987) and have been reported to change the properties of the wood component (Geimer et

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al. 1985; Price 1976; Casey 1987). Casey (1987) found as much as a 30% increase in specific dynamic bending modulus for flakes that were tested before and after being consolidated in a flakeboard panel. Such results suggest that there is much to gain from an improved understanding of wood-water relations in hot-pressing environments and how they bear upon material properties.

The relationship that exists between a wood element and the water vapor in its local environment is likely the single most significant factor affecting its behavior in both manufacture and service. Wood-water relations in ambient to moderate conditions have been well addressed in excellent texts by Skaar (1972, 1988) and Siau (1971, 1984, 1995). The data compiled by the U.S. Forest Products Laboratory (1999) for the equilibrium moisture content (EMC) of Sitka spruce as a function of relative humidity, in oscillating desorption at temperatures from 0 to 130°C, have become the foundation from which nearly all EMC predictions are made. Simpson (1971, 1973, 1980) has evaluated theoretical sorption models using statistical regression analysis and determined that, while several exhibit an excellent fit to the Forest Products Laboratory EMC data, none can accurately predict the heat of sorption of wood. Avramidis (1989) evaluated four empirical moisture sorption models that incorporate temperature as an independent variable in addition to relative humidity. He found that they are as effective in fitting the FPL data as the theoretical sorption models presented by Simpson (1973), and simpler to apply to data representing a range of temperatures. The Hailwood-Horrobin model (Hailwood and Horrobin 1946) with coefficients determined by Simpson (1973) is probably the most widely used equation for predicting EMC of wood.

Experimental data on the EMC behavior of wood at temperatures above 100°C are not nearly as well represented in the literature. Keylwerth (1949), Grumach (1951), and Kohlman and Malmquist (1952) have collected EMC data in pure steam at temperatures up

to 154°C. High-temperature EMC values in pressurized, saturated environments have been determined through a variety of methods by Hann (1966), Strickler (1968), Lutz (1974), Engelhardt (1979), and Resch et al. (1988). These studies employed a range of techniques, and while they reported contradictions in both the shape of the EMC vs. relative humidity curve and the existence of adsorption-desorption hysteresis, they all clearly showed that the nature of the wood-water relations changed significantly as temperature and pressure increased. With the exception of the study by Lutz (1974), all of these researchers have suggested some influence of wood degradation at high temperatures. Using a gravimetric technique for moisture content determination, Engelhardt (1979) and Resch et al. (1988) attempted to correct for this loss of mass. This phenomenon agrees with research on wood stability as reviewed by Hillis (1984); however, the time frame necessary for such degradation to occur makes it more of a concern in the characterization of long-term EMC experiments than in the manufacture of wood-based composites.

High-temperature EMC predictions obtained through various methods of extrapolation from low-temperature data have also been presented by Keylwerth (1949), Sturany (1952), Kauman (1956), and Ladell (1957). Rosen (1980) derived psychrometric relationships for superheated steam environments and discussed their influence on the EMC of wood. Simpson and Rosen (1981) provided a review of previous high temperature EMC work and presented a method of extrapolation at atmospheric pressure based on a fit of the Hailwood-Horrobin equation to EMC data for Sitka spruce (U.S. Forest Products Laboratory 1999).

EXPERIMENTAL

Mature log sections of yellow-poplar (*Liriodendron tulipifera*), loblolly pine (*Pinus taeda*), and aspen (*Populus tremuloides*) were obtained and cut into cants. These cants were

further separated into juvenile, mature, and intermediate portions. Juvenile wood was defined as the wood inside the tenth growth ring and mature wood as that material outside the thirtieth growth ring, as per Kretschmann and Bendsten (1992). The wood material was then end-sealed and allowed to air-dry at ambient laboratory conditions for approximately 12 months.

Equilibrium moisture content data were collected and used to create desorption isotherms at 50°C and 160°C in order to provide information across the range of temperatures found in wood-based composites manufacture. Different gravimetric methods were employed for the 50°C and 160°C isotherm experiments. Below 100°C, experiments could be conducted at atmospheric pressure. Above 100°C, a pressurized vessel was required to obtain water vapor pressure values in excess of atmospheric pressure.

For the 50°C desorption, juvenile, mature, and intermediate wood specimens of yellow-poplar and loblolly pine were machined to dimensions of approximately 8 cm (tangential) by 5 cm (radial) by 2 cm (longitudinal). Due to the smaller size of the aspen log, aspen specimens were longer in the longitudinal direction, and smaller in the transverse dimensions. Specimens for the 160°C experiments were created to maximize surface area, and thus speed up the desorption process. Wood strips approximately 5 cm (tangential) by 0.4 cm (radial) by 25 cm (longitudinal) were separated by 4-mm wooden spacers and bound together using thin chromel wire (Fig. 1). This configuration comprised one specimen.

At 50°C, desorption was carried out in a one m³ environment cabinet with recirculating, forced-air flow. Dry-bulb and wet-bulb thermocouples were used to monitor temperature and humidity of the environment to within 0.1°C and 0.5% relative humidity.

For the 50°C experiments, the air-dried specimens were first allowed to equilibrate at 50°C and 95% relative humidity within the chamber. Specimens were then subjected to an initial desorption to 25% relative humidity, af-

ter which they were again reconditioned to 95% relative humidity. Henceforth, EMC data were collected as the specimens equilibrated in a humidified environment, which was reduced in a stepwise fashion, in 5% increments, to 25% relative humidity. The final step was oven-drying the specimens at 103°C in a convection oven. Specimens were maintained at each relative humidity step until weight loss was less than 0.1% per day, as determined by weighing on a laboratory balance. Weight measurements required the removal of the specimens from the chamber.

A special pressurized sorption apparatus was created to conduct the EMC experiments at 160°C. A test chamber that could be pressurized was necessary to generate wood EMC conditions near fiber saturation at temperatures above 100°C (Fig. 1). This device consisted of a 50-l stainless steel vessel, which could withstand internal pressures in excess of six atmospheres. It had computer control of temperature and internal pressure. A computer algorithm compared the current internal conditions with user-defined setpoints. Based on this information, digital outputs were used to energize or de-energize electric resistance heating elements, control the high-pressure pump for make-up water, and to open a solenoid valve for venting.

Temperature control was achieved using three electronic resistance heaters mounted on the exterior of the pressure vessel. The primary and secondary heaters were incased in cast aluminum jackets that conformed to the circumference of the cylindrical vessel. Both the primary and secondary heaters were 100 ampere, 220 volt units. The primary heater, surrounding the lower part of the vessel, was run at 100% power; and the secondary heater, surrounding the upper portion of the vessel, was operated at 50% power. The tertiary heater was a flat ring-shaped unit fixed to the bottom of the cylinder. This heater was rated at 220 volts, 10 amperes and operated at 100% power. The primary and tertiary heaters were controlled to maintain a constant setpoint temperature measured on the external surface of

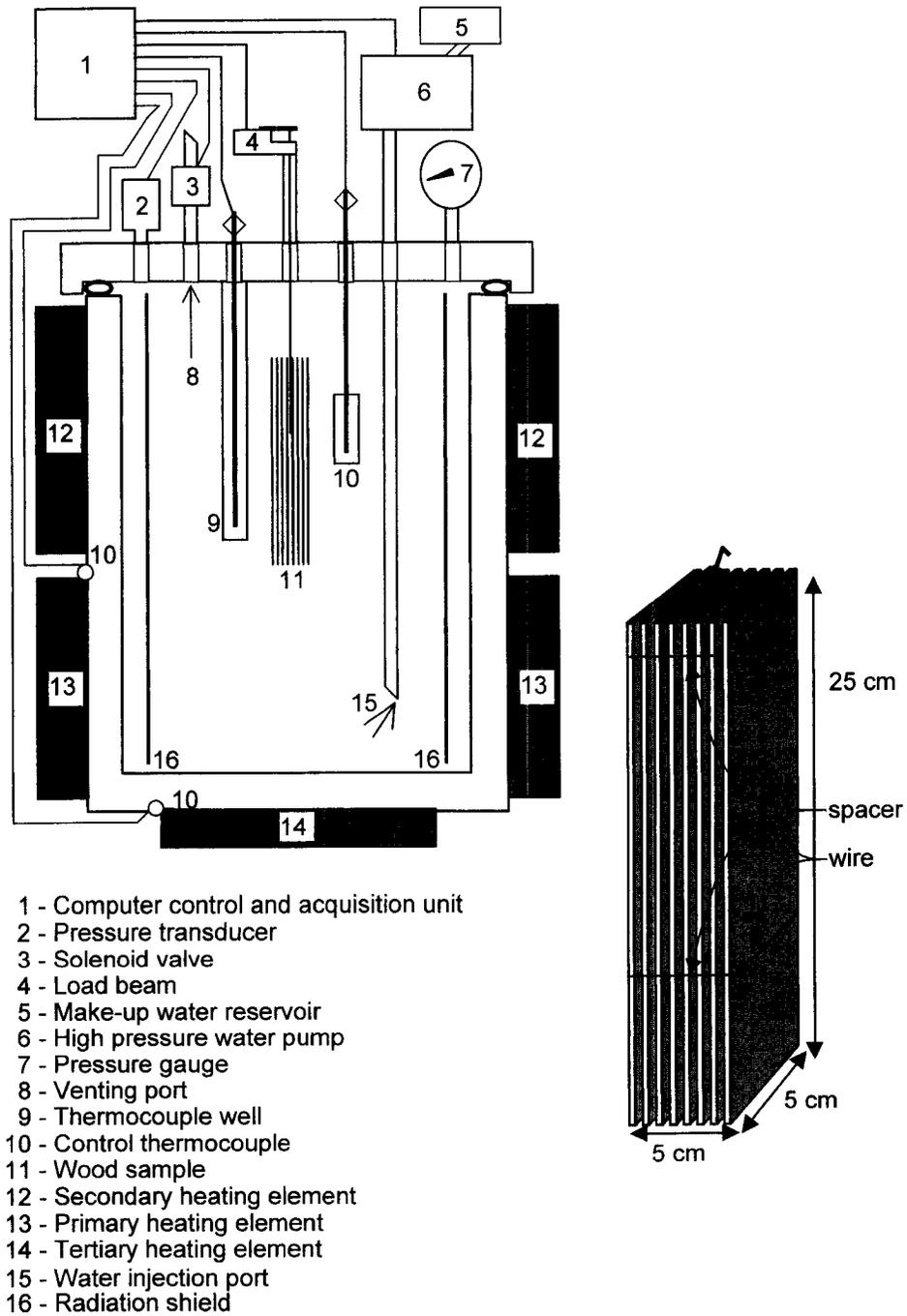


FIG. 1. Schematic drawing of pressurized sorption apparatus and illustration of 160°C desorption specimen.

the cylinder. The secondary heater was controlled to maintain a constant specimen temperature that was measured with a type-K thermocouple embedded in a piece of wood adjacent to the sorption specimen. After the initial heat-up sequence, most of the temperature control was accomplished with the secondary heater.

EMC data, were collected by monitoring the mass of a wood specimen (Fig. 1) within the sorption vessel as the total pressure, and thus the relative humidity of the internal water vapor environment was reduced in a stepwise fashion from saturation to atmospheric pressure. Constant temperature of the specimen was maintained throughout. A paramount concern was protecting the sensitive weighing instrumentation from the corrosive steam environment. To do this, an external weighing apparatus was created to monitor the mass of the specimen while it was suspended inside the chamber (Fig. 2). This device consisted of a load beam (0 to 227 g range max, 0 to 10 volt output, 0.25% of full-scale combined error) mounted on the outside of the vessel. The load beam was attached to the specimen by means of a chromel wire (0.025-mm diameter), which passed through a small orifice (0.030-mm diameter) in the lid. The small leak inherent to this design served to improve circulation within the vessel, and was compensated for by introducing de-oxygenated, distilled make-up water. The loss of water vapor through the orifice created a natural circulation of water vapor from the bottom of the vessel, past the specimen, and out the top. A stainless steel cylinder served as a shield to protect the specimen from direct heat radiation from the vessel wall. This allowed the specimen temperature to closely match the temperature of the water vapor atmosphere.

Prior to the desorption experiments, the load beam was calibrated with weights of known mass. The influence of temperature and pressure was determined by hanging a brass weight on the weighing mechanism and performing a complete desorption cycle. This was done using weights reflecting the upper (sat-

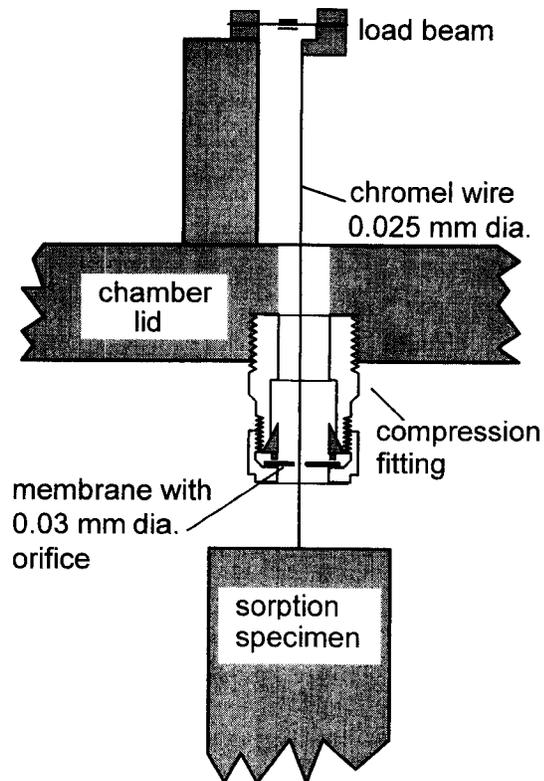


FIG. 2. Schematic drawing of weighing mechanism for the pressurized sorption apparatus.

urated) and lower (dry) limits of mass for the desorption specimens.

The control sequence steps for the high-temperature desorption experiments are shown in Table 1. Preliminary experiments were carried out to determine the soak times necessary at each humidity step. The time increments used in the work by Resch et al. (1988) were employed as guidelines and adjusted based on observations with the wood species and specimen geometry used in this study. It was also necessary to determine the amount of wood degradation that took place during each humidity step. This provided a framework for subsequent correction of EMC data due to mass loss. Specimens for the mass loss tests were assembled, weighed, and hydrated at 95% relative humidity. A matched specimen was used to calculate an approximate oven-dry weight. Specimens were placed in the

TABLE 1. Control sequence for high-temperature desorption experiments.

Step	Control Setpoints			Rel. Hum. (%)
	Time (min.)	Temp. (°C)	Pressure (kPa, psia)	
Heat-up				
1	75 ^a	160	583.8 (84.7)	95
2	10 ^a	160	583.8 (84.7)	95
Desorption				
3	45	160	583.8 (84.7)	95
4	10	160		
5	45	160	549.3 (79.7)	89
6	20	160		
7	45	160	480.4 (69.7)	78
8	20	160		
9	45	160	411.4 (59.7)	67
10	20	160		
11	45	160	342.4 (49.7)	56
12	20	160		
13	45	160	273.5 (39.7)	44
14	20	160		
15	45	160	204.5 (29.7)	33
16	20	160		
17	45	160	135.5 (19.7)	22
18	20	160		
19	45	160	101.0 (14.7)	16
Cool Down				
20	225	20	101.0 (14.7)	—

^a approximate time.

sorption vessel with three liters of water, and the vessel was sealed and heated to 160°C. The pressure was increased to the level necessary for the first relative humidity step (95%), and the mass of the specimen was recorded throughout the duration of that step. The chamber was then depressurized and the specimen's weight was recorded, after which the specimen was oven-dried at 103°C and weighed again. This procedure was repeated for all the relative humidity steps listed in Table 1. Due to concerns of wood degradation in the severe environments, specimens were not subjected to an initial adsorption—desorption cycle at 160°C, as in the 50°C experiments.

Specimens were assembled from the air-dried wood and then hydrated in an environment of 95% relative humidity at 25°C before placement in the pressurized sorption appara-

tus. A small piece of each specimen was oven-dried at both 103°C and 160°C to determine the initial moisture content using a gravimetric technique. This information was then used to approximate the initial oven-dry weight. Three liters of liquid water were added before the prehydrated specimens were suspended from the weighing mechanism and sealed inside the vessel. The vessel was heated at approximately 2°C per minute until the isotherm temperature of 160°C was reached. Once the internal pressure had attained a level corresponding to 95% relative humidity (587 kPa, 85 psia at 160°C), the desorption sequence was initiated. At this time, essentially all of the air initially in the vessel had been displaced by water vapor. The isotherm temperature was then maintained as the internal pressure was reduced to atmospheric pressure in seven steps. Figure 3 illustrates the targeted environmental conditions for the high-temperature experiments as well as characteristic temperature and relative humidity values recorded during the desorption. Some fluctuation in relative humidity and temperature about the setpoint conditions was evident. The relative humidity is tied closely to temperature through the saturated water vapor pressure, and thus minor fluctuations in temperature, inherent to the heating system employed, resulted in subsequent variations in relative humidity. Occasionally such variation resulted in equilibrium not being achieved at a given step, in which case the control sequence continued to the next step and EMC data at that condition were omitted for that specimen. Specimens were judged to have reached equilibrium when their observed mass was unchanged for 5 minutes.

Once the desorption experiment was complete and the apparatus had cooled, specimens were removed, weighed, and placed in a convection oven at 160°C to dry. This oven-dry mass was compared to the initial oven-dry mass to determine the amount of mass lost during desorption due to hygro-thermal degradation. Together with the results of preliminary mass-loss experiments, this value was used to correct the oven-dry mass for subse-

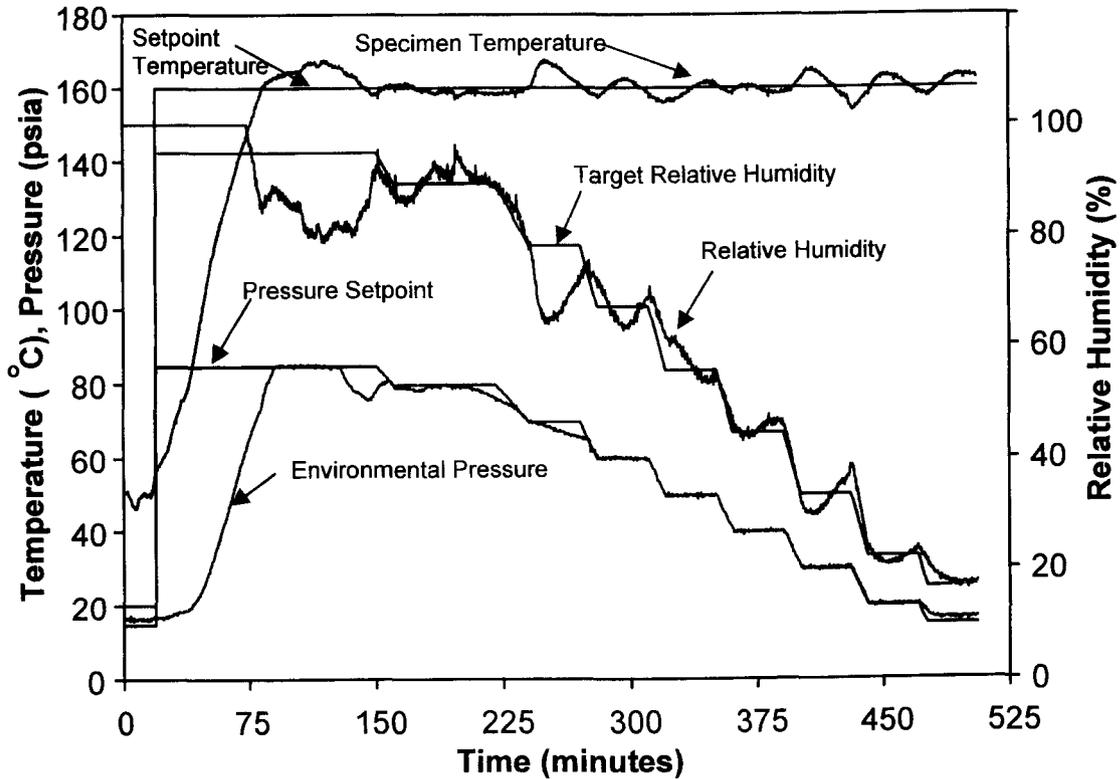


FIG. 3. Environmental conditions and specimen temperature for a desorption experiment at 160°C.

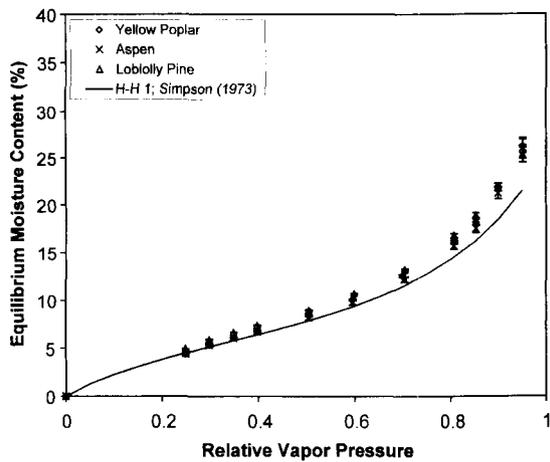


FIG. 4. Desorption isotherms for yellow-poplar, aspen, and loblolly pine at 50°C. Data points represent an average of all wood types. Error bars indicate one standard deviation. Line is EMC predicted by the Hailwood-Horrobin 1-hydrate model (H-H 1) with parameters after Simpson (1973).

quent moisture content calculations. The oven-dry mass after desorption was also compared to the final measured mass in the sorption vessel at atmospheric pressure to compensate for drift of the load beam over time.

Chemical analyses were carried out on three replicate specimens taken from mature wood specimens of all three species both before and after desorption. A standard HPLC carbohydrate analysis with Klason lignin was performed as per the technique of Kaar et al. (1991) on the treated and untreated samples. Ash content was also determined by thermogravimetric analysis (TGA) (700°C, 10°C/min.).

RESULTS AND DISCUSSION

Figure 4 shows desorption isotherms for yellow-poplar, aspen, and loblolly pine at 50°C. The experimental isotherms exhibit the sigmoid shape commonly reported for wood

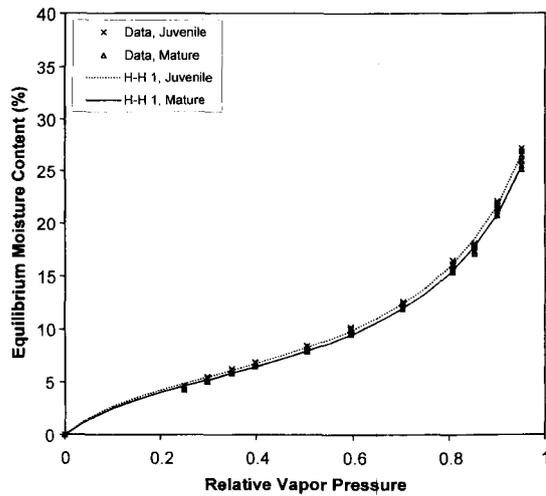


FIG. 5. Desorption isotherm data for juvenile and mature aspen wood at 50°C. Lines represent a nonlinear fit to the Hailwood-Horrobin 1-hydrate model.

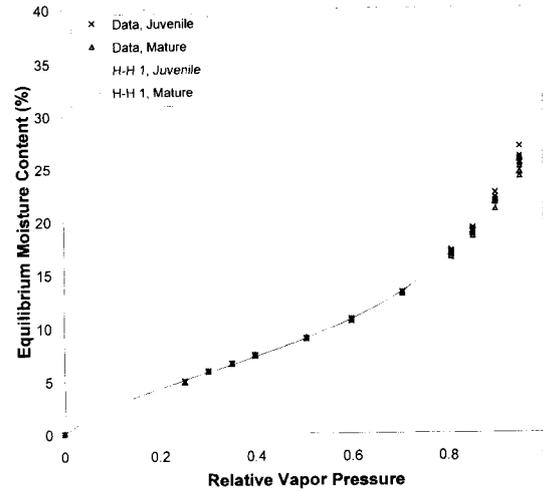


FIG. 6. Desorption isotherm data for juvenile and mature loblolly pine wood at 50°C. Lines represent a nonlinear fit to the Hailwood-Horrobin 1-hydrate model.

(Simpson 1980) and are free from any suspicious anomalies. Each data point represents an average of eleven specimens: four each of juvenile and mature wood, and three of intermediate wood. The solid line indicates predicted EMC using the Hailwood-Horrobin 1-hydrate model,

$$EMC = \frac{1.80}{W} \left[\frac{K_1 h}{1 - K_1 h} + \frac{K_1 K h}{1 + K_1 K h} \right] \quad (1)$$

where W , K , and K_1 are temperature-dependent parameters and h is the relative vapor pressure.

The Hailwood-Horrobin 1-hydrate model fit to the FPL Sitka spruce (*Picea sitchensis*) data, over the range of 0 to 100°C, give the following polynomial equations for the temperature-dependent parameters (Siau 1995).

$$K = 4.73 + 0.048(T) - 0.0005(T^2)$$

$$K_1 = 0.706 + 0.0017(T) - 0.000006(T^2)$$

$$W = 0.223 + 0.007(T) + 0.000019(T^2)$$

The experimental and predicted values agree only up to a relative vapor pressure of about 0.4, after which the data points gradually diverge upwards from the predicted curve, reaching a maximum deviation of 4% EMC at

a relative vapor pressure of 0.95. Considering that the predictions are based on data from Sitka spruce, this difference is not remarkable.

Figures 5, 6, and 7 illustrate the EMC and relative vapor pressure relationships of mature and juvenile wood for aspen, loblolly pine, and yellow-poplar, respectively. Nonlinear regression analysis was used to fit the Hailwood-

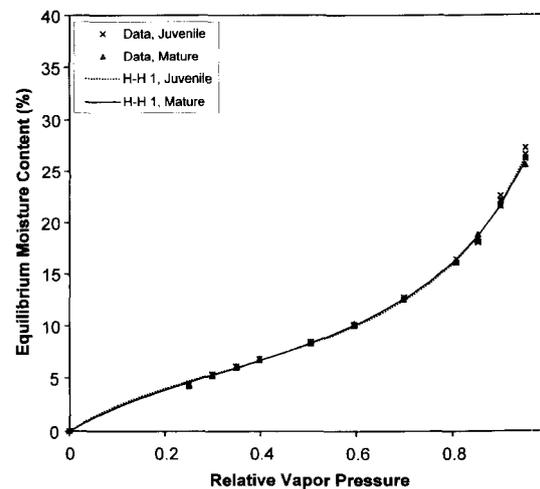


FIG. 7. Desorption isotherm data for juvenile and mature yellow-poplar wood at 50°C. Lines represent a nonlinear fit to the Hailwood-Horrobin 1-hydrate model.

TABLE 2. Parameters of the Hailwood-Horrobin 1-hydrate model evaluated at 50°C. Sitka spruce parameters from Simpson (1973).

Species	Parameters		
	W	K	K ₁
Sitka spruce	0.306	5.88	0.776
Aspen:			
Juvenile	0.330	8.010	0.841
Mature	0.340	7.334	0.839
Loblolly pine:			
Juvenile	0.278	5.837	0.801
Mature	0.263	5.171	0.777
Yellow-poplar:			
Juvenile	0.315	6.178	0.832
Mature	0.295	4.836	0.816

Horrobin 1-hydrate model to the data from this study. The parameters of the model evaluated at 50°C are given in Table 2. At a given humidity level, EMC for juvenile wood was generally higher than that for mature wood. The largest difference occurred at the highest relative humidity, with a maximum deviation of 1.3% MC for loblolly pine at 95% relative humidity. Applying a test for independence between regression lines (Neter and Wasserman 1974) revealed that the 50°C isotherms for juvenile and mature wood are statistically

independent at a significance level of α equal to 0.025 for all three species.

Experiments conducted to evaluate degradation of wood indicated that at 160°C, mass loss due to thermal degradation was much more severe at high relative humidity than at low relative humidity (Fig. 8). This behavior can be explained by the drastic increase in thermal degradation of wood that occurs with increasing moisture content. Skaar (1976) reported reaction rates for thermal degradation in wet wood to be as high as ten times those for dry wood. Hillis (1984) also reviewed several studies that corroborate these findings.

To account for wood material lost to thermal degradation, a relationship to distribute the mass loss among the stages of the desorption process was developed. This function preferentially attributed the mass loss to the higher moisture content portion of the desorption experiments and corrected the observed EMC by adjusting the oven-dry weight to reflect mass lost at each step. The mass-loss correction function applied to the wood degradation data is shown in Fig. 8. Both actual and predicted mass loss were normalized and displayed as a cumulative distribution function. Note that the loss of wood material is dependent on temperature, moisture content, and

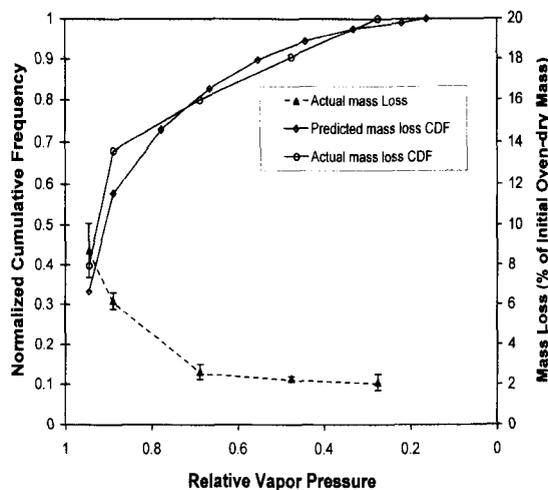


FIG. 8. Mass loss due to wood degradation as a function of relative vapor pressure, for desorption of mature yellow-poplar at 160°C. Lines represent normalized mass loss in the form of a cumulative distribution function (CDF).

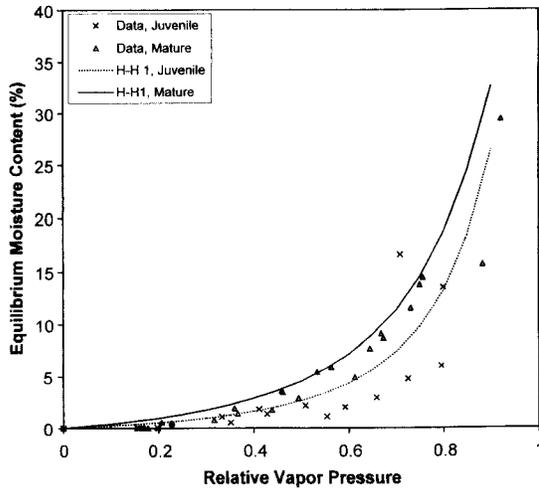


FIG. 9. Desorption isotherm data for juvenile and mature aspen wood at 160°C. Lines represent a nonlinear fit to the Hailwood-Horrobin 1-hydrate model.

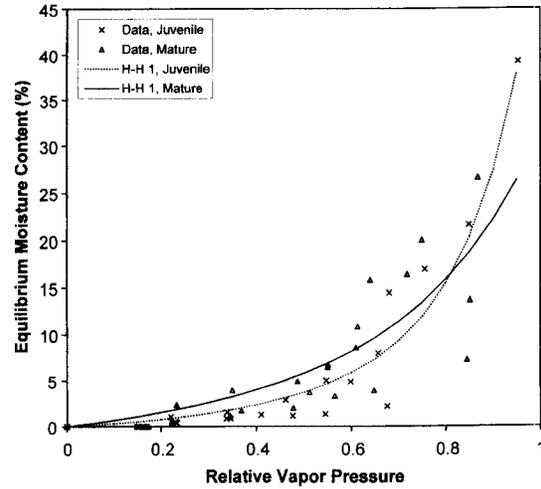


FIG. 11. Desorption isotherm data for juvenile and mature yellow-poplar wood at 160°C. Lines represent a nonlinear fit to the Hailwood-Horrobin 1-hydrate model.

time. The results in Fig. 8 are based on the time needed to achieve equilibrium at each relative humidity step of the desorption experiment.

Relationships for EMC and relative vapor pressure at 160°C for the mature and juvenile wood of aspen, loblolly pine, and yellow-poplar, respectively, are shown in Figs. 9, 10, and

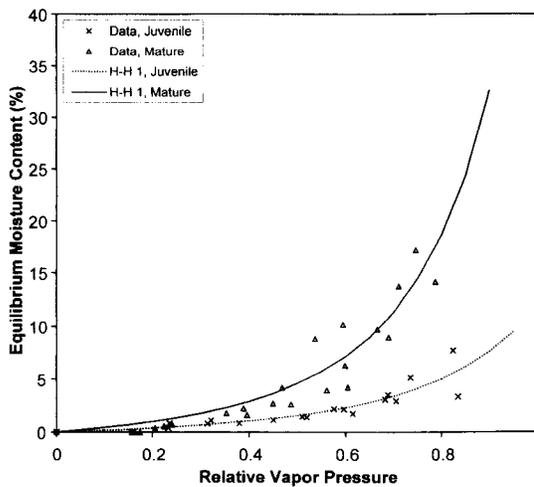


FIG. 10. Desorption isotherm data for juvenile and mature loblolly pine wood at 160°C. Lines represent a nonlinear fit to the Hailwood-Horrobin 1-hydrate model.

11. Nonlinear regression analysis was again used to fit the Hailwood-Horrobin 1-hydrate model to the data. The parameters of the model, evaluated at 160°C are given in Table 3. The high-temperature desorption isotherms were distinctly lower than their 50°C counterparts, an expected result of the reduced activity of water with increasing temperature. The initial linear portion and the first inflection point (near 0.1 relative vapor pressure) characteristic of the traditional, sigmoid-shaped, isotherm are absent in these results. This behavior is consistent with the results of Strick-

TABLE 3. Parameters of the Hailwood-Horrobin 1-hydrate model evaluated at 160°C.

Species	Parameters		
	W	K	K ₁
Aspen:			
Juvenile	-0.010	-1.012	0.830
Mature	0.004	-0.990	0.771
Loblolly pine:			
Juvenile	0.016	-0.981	0.664
Mature	0.004	-0.990	0.771
Yellow-poplar:			
Juvenile	0.002	-0.996	0.776
Mature	0.005	-0.971	0.572

ler (1968), Engelhardt (1979), and Resch et al. (1988). EMC initially increased rather slowly with relative vapor pressure, in a more or less linear fashion, up to a level of around 0.5. At this point, an inflection occurred and EMC increased at an accelerating rate as saturation was approached. This behavior suggested that some type of change in the material occurred around 50% relative humidity, affecting the sorptive behavior of the wood. Strickler (1968) recorded similar results, including a reversal effect at high relative vapor pressures, where the EMC increased with increasing temperature, causing the sorption isotherms to cross over those at lower temperatures. He suggested a revised theory of sorption for high temperatures, postulating that this inflection was due to a combination of multilayer sorption and inordinate swelling of the wood structure due to softening of the glassy wood polymers.

The temperature and humidity conditions at which the EMC increases rapidly are above the reported range for lignin to exceed its glass transition temperature (Kelly et al. 1987). When a material goes through a glass transition, it is theorized that a corresponding increase in free volume occurs. Free volume is defined as the intermolecular space within a network of polymer molecules (Aklonis and MacKnight 1983). Above the glass transition, the number of accessible primary sorption sites available in wood increases rapidly, yielding in effect an expanding monolayer of sorbed water molecules. The data of Hann (1966) and Engelhardt (1979) also support the idea that plasticization of wood influences the inflection of the EMC and relative humidity relationship. In addition, the results of Strickler (1968) indicate that as the isotherm temperature is increased from 100 to 170°C, the inflection in the EMC curve moves toward lower relative vapor pressures. This appears to reflect the coupling effect between temperature and moisture on the glass transition of polymers. As temperature increases, the moisture level necessary for the onset of the glass transition is reduced (Aklonis and MacKnight

1983). Researchers have also reported this phenomenon on sorbents other than wood. Urquhart and Williams (1924) observed a similar reversal effect at high relative humidity, which they attributed to "swelling of the material and consequent exposure of new surface." Jeffries (1960) also noted similar behavior with synthetic polymers at 120 and 150°C.

At 160°C, all three species illustrate an apparent difference in EMC behavior between juvenile and mature wood. This difference was the most significant ($\alpha < 0.025$) for loblolly pine, and less statistically robust for aspen ($\alpha = 0.15$) and yellow-poplar ($\alpha < 0.24$). Curiously, EMCs for juvenile wood were lower than those for mature wood at 160°C, whereas this hierarchy was reversed at 50°C. This was likely a result of the chemical differences between the two wood types, which was in some way manifested as a difference in sorptive behavior at high temperatures.

Table 4 lists the results of the chemical analyses performed on mature wood samples before and after desorption experiments. However, the observed changes in chemical composition are not significant in light of the accuracy of the technique. In general, mature wood samples tended to lose 1.5 to 4% (based on total mass) of their hemicelluloses during desorption, with loblolly pine recording the highest losses and yellow-poplar the least. As a result of these hemicellulose losses, there was a relative increase in the lignin and cellulose content during desorption. Loblolly pine exhibited the largest increase in total lignin, and the smallest increase in cellulose. Because juvenile wood specimens were not analyzed, these results do little to explain the differences in sorptive behavior between mature and juvenile specimens.

When compared to mature wood, juvenile wood has a higher concentration of hemicelluloses and lignin, at the expense of cellulose (Panshin and deZeeuw 1980). Concerning the three primary components of wood, hemicelluloses are both the most hygroscopic and the most susceptible to thermal degradation. Lignin is both the least hygroscopic and least sus-

TABLE 4. Chemical composition of mature (M) wood samples of southern pine (SP), aspen (AS), and yellow-poplar (YP) before (u) and after (t) desorption. Total composition of a sample is not 100% due to omission of uronic acids and acetyl content.

Sample	Sugar Composition, %						Lignin, %			Ash, % Content
	Cellulose Glucan	Non-Cellulose				Total Sugar	Acid ^a Insoluble	Acid Soluble	Total Lignin	
		Xylan	Galactan	Arabinan	Mannan					
SPMu	43.94	6.97	2.22	0.00	15.17	68.29	25.55	0.29	25.84	0.03
SPMt	44.43	5.63	1.54	0.00	13.12	64.71	29.41	0.58	29.99	0.58
ASMu	42.95	17.04	0.00	0.00	1.85	61.85	19.24	3.07	22.31	0.43
ASMt	44.40	14.80	0.00	0.00	1.66	60.86	21.59	2.97	24.56	0.13
YPMu	43.91	13.76	0.00	0.00	1.51	59.18	22.06	3.91	25.97	0.41
YPMt	47.58	12.26	0.00	0.00	1.68	61.52	25.35	3.18	28.53	0.02
Sigma Oat Spelts Xylan (control)	11.19	66.66	0.00	6.49	0.00	84.34	2.20	0.57	2.77	3.22

^a (Klason), also includes extractives in this analysis.

ceptible to wood degradation (Fengel and Wegener 1989; Christensen and Kelsey 1959). At low temperatures, juvenile wood should thus be slightly more hygroscopic than mature wood, explaining the higher EMC exhibited by juvenile wood at 50°C. Juvenile wood at 160°C would have, due to the relative thermal instability of the hemicellulose, lost its most sorptive component and become less hygroscopic than mature wood.

Based on the sorptive nature of the different

wood components, a difference in the amount of wood material lost due to thermal degradation would have helped to explain the temperature effect on the differences in EMC between mature and juvenile wood. However, all specimens exhibited similar mass losses, ranging from 8 to 11% of their oven-dry mass. Hsu et al. (1988) treated wood with saturated steam at approximately 200°C for 4 minutes and found an increase of water soluble compounds of 15% for aspen and 10% for lodgepole pine. There was essentially no change in the cellulose and lignin content.

The measured mass losses due to the desorption experiments may not reflect the complete nature of the wood degradation process. It is quite probable that some of the hemicelluloses could have been degraded such that their hygroscopicity was reduced; yet the degradation products still remained within the wood structure. Thus, many of the degradation products would contribute to the nonsorptive mass of the specimen, but not actively participate in adsorbing or desorbing water.

Figures 12 and 13 display the 160°C isotherm data from this study along with data collected by others. These comparisons illustrate that thermal degradation, species, and juvenile-mature wood difference can lead to quite different observed sorption behavior. The

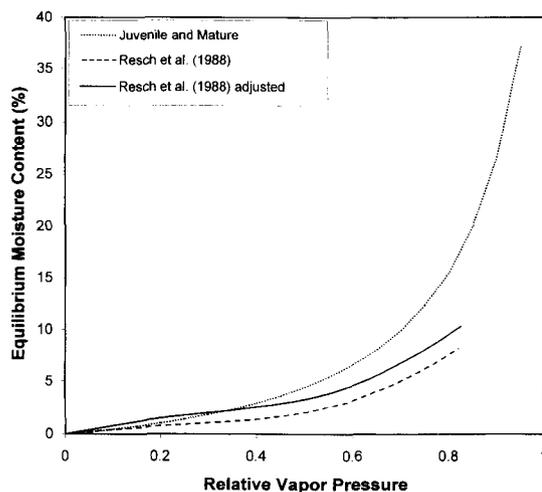


FIG. 12. EMC for yellow-poplar wood at 160°C. Data from combined mature and juvenile wood from this study in addition to data from Resch et al. (1988).

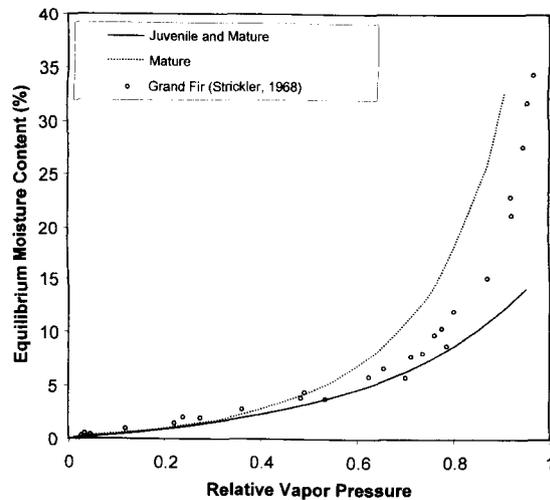


Fig. 13. EMC for mature and combined mature and juvenile loblolly pine wood at 160°C, in addition to data from Strickler (1968) for adsorption of grand fir at 150 and 170°C.

technique used by Resch et al. (1988) to collect data for the EMC of yellow-poplar at 160°C (Fig. 12) was the most similar. Resch et al. only considered time when correcting for mass lost due to wood degradation. EMC was calculated at each step based on the initial oven-dry weight of the specimens, whereas EMC values in this study were calculated based on the (corrected) oven-dry weight at each step. When corrected as per the method used herein, the data of Resch et al. appear closer to the EMC results generated in this study for yellow-poplar (juvenile and mature). Results of Hann (1966) for yellow-poplar tend to agree more closely with the data presented here, while the data of Engelhardt (1979) for beech tend to fall in between. The EMC data of Strickler (1968) for adsorption of grand fir at 150° and 170°C are slightly elevated in comparison to the desorption EMC data for loblolly pine (combined mature and juvenile wood) presented in this paper (Fig. 13). Larger deviations occur above 0.8 relative vapor pressure. The temperature-dependent parameters of the Hailwood-Horrobin 1-hydrate model presented by Siau (1995) failed to adequately describe EMC behavior at 160°C exhibited

here and in the literature, and predicted non-sensical values of EMC at temperatures above 155°C.

CONCLUSIONS

Equilibrium moisture content data in the form of desorption isotherms were collected for mature and juvenile wood of aspen, loblolly pine, and yellow-poplar. These data pertain to the understanding of material behavior during the manufacture of wood-based composites.

A high-temperature sorption apparatus was designed to generate the environments necessary to achieve saturation conditions at 160°C. This apparatus had the instrumentation and control features necessary to create a constant temperature, water vapor environment, and to reduce the humidity level of that environment in a stepwise fashion from saturation to atmospheric conditions. A gravimetric method of MC determination was performed continuously inside the apparatus.

At 50°C, EMC behavior of the wood species studied deviated only slightly (and only at high humidity) from predictions based on the data derived from Sitka spruce. EMC behavior at 160°C, however, was distinctly different from the 50°C data, or any extrapolation from existing low-temperature data. Isotherms generated at 160°C initially exhibited low EMCs before turning sharply upwards above 50% relative humidity. This behavior suggested that a change in the sorptive properties of the wood occurred as temperature and moisture conditions exceeded the glass transition temperature for lignin.

Considerable differences existed between the EMC relationships of juvenile and mature wood. At 50°C, juvenile wood tended to equilibrate at higher EMCs than mature wood. At 160°C, however, juvenile wood exhibited markedly lower EMCs than mature wood. Thermal degradation of wood was detected during desorption experiments. This degradation was manifested as mass loss and was much more severe at high humidity levels. Re-

duction in the sorptive behavior as a result of thermal degradation was proposed as a possible explanation for differences in EMC behavior for juvenile and mature wood at 160°C.

ACKNOWLEDGMENTS

Funding for this project was provided by the Cooperative State Research, Education, and Extension Service, U.S. Department of Agriculture, under Agreement No. 95-37103-2103.

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