CHANGES IN SHRINKAGE AND TANGENTIAL COMPRESSION STRENGTH OF SUGAR MAPLE BELOW AND ABOVE THE FIBER SATURATION POINT

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ABSTRACT

Two experimental techniques were used to conduct moisture sorption tests in sugar maple sapwood. The first used saturated salt solutions (from 58% to 90% relative humidity) and the second used the pressure membrane method (above 96% relative humidity). These sorption tests were combined with dimensional measurements and perpendicular-to-the-grain tangential compression tests. Results indicated that at the equilibrium moisture content, radial, tangential, and volumetric shrinkage, as well as changes in transverse strength, occur above the nominal fiber saturation point. These results can be described by the effect of hysteresis at saturation on wood properties. This hysteresis implies that loss of bound water takes place in the presence of free water. The initial equilibrium moisture content at which bound water is removed from sugar maple wood was found to be 42.5%.

Keywords: Moisture sorption, desorption, fiber saturation point, wood strength, compliance coefficient, shrinkage, sugar maple.

INTRODUCTION AND BACKGROUND

The fiber saturation point (FSP) is a very important feature of wood as it governs the changes in its properties (Siau 1984; Skaar 1988). This feature has been defined from the beginning of the century by Tiemann (1906) as the moisture content (MC) at which the cell walls are saturated with bound water with no free water in the cell cavities. It is normally assumed that the FSP is the MC below which the physical and mechanical properties of wood begin to change as a function of MC (USDA 1974; Siau 1984). The FSP is therefore considered in models used to adjust the mechanical properties of wood as a function of its MC (Bodig and Jayne 1982), as well as in wood shrinkage and density adjustment models (Siau 1984; Skaar 1988).

However, some studies show that this assumption may not be realistic. Stevens (1963) indicated that shrinkage in beech wood begins taking place above the FSP. Shrinkage values reported in his study were obtained at equilibrium moisture content, in such a way that the moisture content gradient effect advanced by him for explaining this behavior appears inappropriate. Goulet and Hernández (1991) reported an appreciable hysteresis effect on the EMC and on the perpendicular-to-grain tangential tension strength of sugar maple wood at high relative humidities (RH). The difference for the tangential tension strength be-

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tween adsorption and desorption phases was 20% at 26% EMC. This effect was related to the hysteresis at saturation phenomenon, which affected the wood moisture sorption above 63% RH (Hernández 1983). This hysteresis implies that during desorption the loss of bound water begins before all free water has been removed from the wood. Although their desorption curves had 26% EMC as upper limit, Goulet and Hernández (1991) suggested that the effect of MC on the sugar maple wood properties could be extended beyond the FSP, estimated to be 31% MC. In fact, Goulet (1968) had previously shown that MC already affects the perpendicular-to-grain radial compression strength of sugar maple at 37% EMC.

The principal characteristic of these studies (Goulet 1968; Goulet and Hernández 1991) was to combine physical-mechanical tests with sorption experiments. These were conducted within the entire range of RH (from 0% to 100% RH). A study focusing on the high humidities (above 90% RH) would contribute to better understanding of the effect of hysteresis at saturation on wood properties. The purpose of this investigation was therefore to study the effect of EMC on wood properties of sugar maple below and above cell-wall saturation. Two moisture sorption techniques, namely the use of saturated salt solutions (between 58% and 90% RH) and the pressure membrane method (above 96% RH) were applied to large specimens at room temperature. These sorption tests were combined with shrinkage measurements as well as perpendicular-to-the-grain tangential compression tests.

MATERIALS AND METHODS

Experiments were carried out with sugar maple (*Acer saccharum* Marsh.) sapwood. Specimens for the pure perpendicular-to-grain tangential compression tests were cut with a cross-section of 20 (r) × 20 (l) mm and a height of 60 (t) mm. Shape and dimensions of these samples differ from those recommended by the ASTM D143. The choice of dimensions was limited by the matching techniques used and the moisture sorption tests. A length-to-width ratio of 3 was used to preclude buckling during the test (Bodig and Jayne 1982) and to limit the effect due to the growth ring curvature.

Twenty green flatsawn boards were selected and allowed to dry in two steps in a conditioning room at relative humidities of 80%, then 60%, at a temperature of 20 C. In order to avoid probable subsurface damage during planing of the wood, the fixed-knife pressure-bar method proposed by Stewart (1986) was used at an MC of approximately 14%. Tangential and radial faces of the specimens were thus obtained by orthogonal cutting instead of the conventional peripheral planing method. Each board was then cross-cut to yield 20-mm-thick specimens. In order to investigate eleven moisture conditions, the best eleven adjacent samples were selected from each board. This longitudinal matching yielded eleven comparable groups of twenty specimens each.

EXPERIMENTS

The experiments involved moisture sorption techniques combined with shrinkage and mechanical tests. Wood specimens were mechanically tested as soon as the desired EMC was reached. There were nine points in desorption and one in adsorption (Table 1). In preparation for the desorption tests, specimens were saturated with distilled water until their full moisture content was reached. At this state, their green dimensions in all principal directions were measured with a digital micrometer to the nearest 0.001 mm. The group to be conditioned in adsorption over distilled water was kept at 20 C and 60% RH prior to the adsorption test.

The sorption experiment required two experimental techniques. In the first, wood was conditioned over saturated salt solutions. The second technique involved use of a pressure membrane. The first technique was carried out between 58% and 90% RH, as well as over distilled water, using sorption vats that have been described elsewhere (Goulet 1968). These vats provided a temperature control of ±0.01 C during extended periods, thus allowing control of the relative humidity in the various

TABLE 1. *Characteristics of the moisture sorption conditions used in these experiments.*

Number of group	State of sorption	Chemical or saturated salt solution	Nominal relative humidity (%)	Water potential (Jkg^{-1})	Radius of curvature of the air-water meniscus[1] (μm)
		Equilibration under a pressure membrane at 20 C			
1	Desorption	—	99.926	−100	1.457
2	Desorption	—	99.770	−300	0.4858
3	Desorption	—	99.484	−700	0.2082
4	Desorption	—	98.532	−2,000	0.07287
5	Desorption	—	96.370	−5,000	0.02915
		Equilibration over saturated salt solutions at 21 C			
6	Desorption	$ZnSO_4$	90	−14,296	—
7	Desorption	KCl	86	−20,464	—
8	Desorption	NaCl	76	−37,237	—
9	Desorption	NaBr	58	−73,911	—
10	Adsorption	H_2O	≈100	—	—
		Full saturation under distilled water			
11	Saturation	H_2O	100	0	∞

[1] According to the capillary pressure equation (zero wetting angle). Under about 92% RH, equation is not applicable.

glass desiccators serving as small sorption chambers. For each point of sorption, one desiccator containing twenty specimens was used. In this way five sorption conditions, which lasted at least 116 days, were realized over saturated salt solutions or distilled water in a single step (Table 1). Assuming that the equilibrium process is an exponential function of time (Laforest 1981), this period was adequate to reach equilibrium. Control specimens left in adsorption over distilled water were weighed periodically, without being removed from the desiccator.

The use of a pressure membrane procedure yielded five additional points of desorption between 96% and 100% RH (Table 1). This technique is suitable for this humidity range and has been used by many workers before (Robertson 1965; Stone and Scallan 1967; Griffin 1977; Viktorin and Čermák 1977). The procedure introduces the concept of water potential (WP), which has been described by Siau (1984, 1988). This concept is derived from classical thermodynamics and is defined as the difference between the specific Gibbs free energies of water in the state under study and in a standard reference state. The reference state generally used is a hypothetical pool of pure free water at atmospheric pressure, at a given elevation and at the same temperature as that of the water in the porous material (Fortin 1979; Cloutier and Fortin 1991). The latter authors considered the water potential as the driving force for the movement of moisture within wood. This concept may be applied to the diffusion between wood and moist air, as well as to the effect of capillary forces on moisture movement (Siau 1988). The WP is normally expressed in terms of energy per unit mass in J kg^{-1}. Relationships between water potential and relative humidity are shown in Table 1.

For each point of desorption, twenty fully saturated specimens were placed into one of two apparatuses on a saturated cellulose acetate membrane. A detailed description of the apparatus is given by Cloutier and Fortin (1991). A saturated clay layer about 2 mm thick was placed on the membrane in order to ensure permanent hydraulic contact with the specimens (Fortin 1979). Pressure was then gradually applied until the required level was reached. Flow of water was collected in a burette. EMC was considered reached when outflow became negligible (no outflow during two successive days). These experiments required between seven and nineteen days of sorption, depending on the WP considered.

As soon as each sorption test was completed, the sample mass was measured to the nearest 0.001 g. Dimensions in all principal directions were taken to the nearest 0.001 mm with a micrometer. Mechanical tests were immediately carried out on a Riehle machine. Deformation in the tangential direction was measured over a span of 40 mm located in the central part of the specimen, using a two-sided clip gauge provided with a linear variable differential transformer (LVDT). Complete deformation of the specimen was also measured by the displacement of the cross-head, using another LVDT. In all cases, hygrothermal changes during the mechanical test were controlled by wrapping the specimen in cotton that had been previously conditioned above the same humidity conditions as the wood. As per Sliker (1978), the cross-head speed was set to ensure a similar strain rate for all moisture conditions. In the elastic range, this strain rate was 0.40 mm/mm/min.

In addition to the sorption experiments, a group of samples that had been fully saturated with distilled water were mechanically tested. This was done in order to compare the properties measured under this condition with those evaluated from the group conditioned over distilled water.

These tests permitted the establishment of the compliance coefficient in the tangential direction, s_{33}, of the wood. The reciprocal of this parameter is the Young's modulus. The cross-sectional area used for the calculations was the one measured during mechanical test conditions. The differences in dimensions of specimens after full moisture saturation and before the mechanical test were used to estimate the partial percent shrinkage in the tangential (β_{TH}), radial (β_{RH}) and longitudinal (β_{LH}) direction of the wood. Volumetric shrinkage was estimated to be the summation of these three directional shrinkages ($\beta_{TH} + \beta_{RH} + \beta_{LH} - \beta_{TH} \cdot \beta_{RH}$). Finally, the mass of the specimens just before the mechanical test and their mass measured after oven-drying were used to calculate the EMC, expressed as a percentage of oven-dry mass.

RESULTS AND DISCUSSION

Wood hygroscopicity

Figure 1 shows the relationship between the WP and EMC for sugar maple wood. This figure displays only the desorption points obtained using either the pressure membrane or the saturated salt solution methods. In all cases, the standard errors of the MC data do not exceed the symbol size shown. It is to be noted that this figure greatly spreads out the important region between 96% and 100% RH. An excellent continuity is apparent between the values measured by both sorption methods confirming the suitability of the pressure membrane method as demonstrated previously by Stone and Scallan (1967) for spruce, Viktorin and Čermák (1977) for beech, and Cloutier and Fortin (1991) for aspen. A comparison with previous EMCs measured above 90% RH for sugar maple has shown a good agreement (Fortin 1981). EMCs for the same wood obtained below 90% RH (Fig. 1) are also similar to previous studies (Goulet 1968; Djolani 1970; Goulet and Fortin 1975; Laforest 1981; Goulet and Hernández 1991; Hernández 1993a).

The hysteresis at saturation has been described by Goulet and Hernández (1991) as the difference between the equilibrium obtained in desorption when starting from the FSP and that reached in desorption when starting from wood containing free water. Some researchers have indicated that high EMCs in desorption are obtained using never-dried specimens (Higgins 1957; Spalt 1958). Skaar (1988) ascribed such behavior to an initial irreversible loss in hygroscopicity after the initial drying of green or water-soaked wood. However, many studies have shown that this effect is apparent during subsequent desorptions (Goulet 1968; Fortin 1979; Hart 1984; Goulet and Hernández 1991; Cloutier and Fortin 1991). Experimental data for a second desorption between 76% and 99.926% RH show an equilibrium higher than the one expected when starting from the FSP (about 31% MC). This confirms that the hysteresis at saturation is not limited to the first drying, but

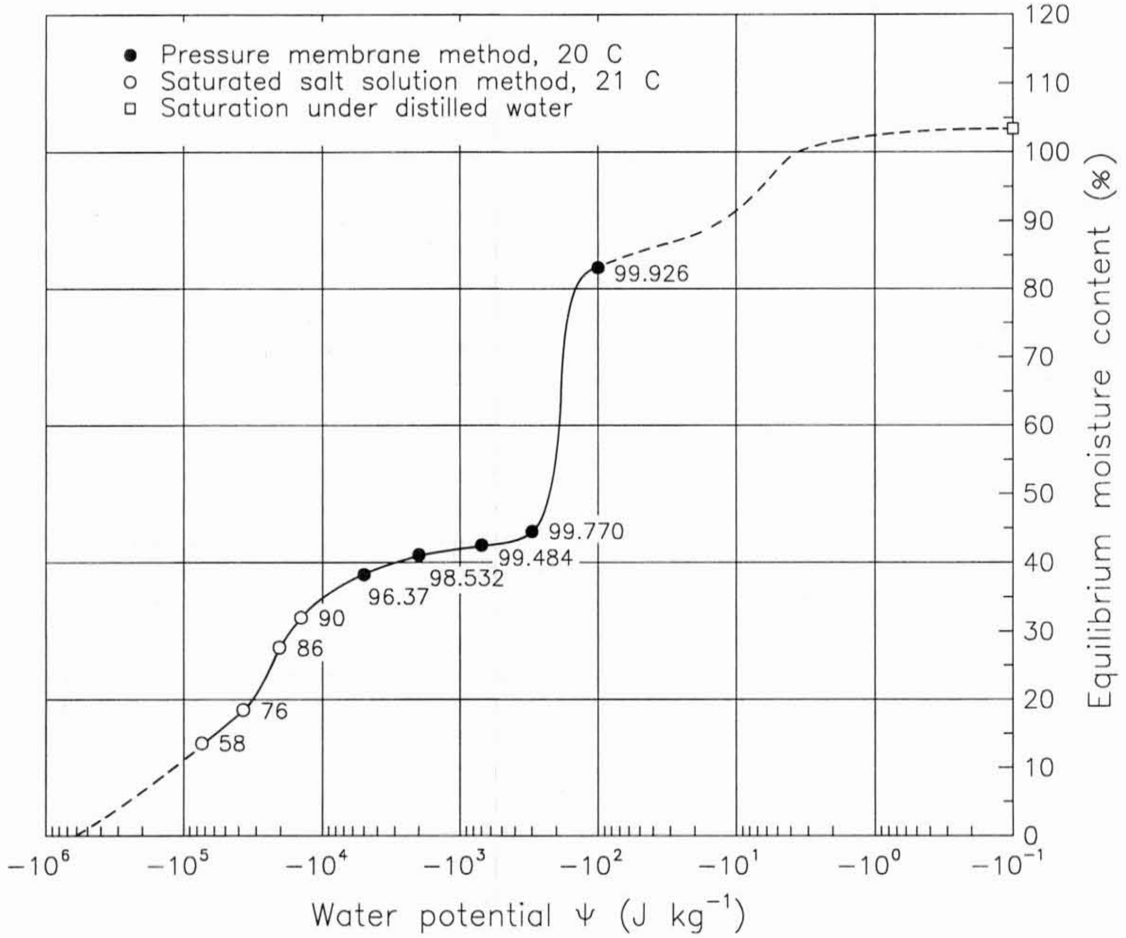

FIG. 1. Equilibrium moisture content-water potential relationship of sugar maple sapwood along the boundary desorption curve for longitudinal desorption at 20 and 21 C, where the number near each data point represents the relative humidity value.

rather to any desorption made in the presence of free water.

The importance of this hysteresis will vary according to the initial MC of the wood. In this study the desorption was carried out beginning from the fully saturated state, and the curve obtained corresponds to the maximum EMC expected for each humidity condition. The term boundary curve desorption is therefore used to describe this feature. Any desorption curve obtained from a lower initial MC would be located below this boundary desorption curve.

Although the only adsorption experiment in this work was performed over distilled water, Fortin (1979) carried out adsorption and desorption experiments using the pressure membrane methods and reported a large hysteresis for western hemlock sapwood at high humidities (above 65% RH). He suggested that the "ink-bottle effect" would be the primary cause of this hysteresis observed at high moisture contents. The capillary system of wood consists of cavities interconnected by narrow channels. The variation in dimensions between the different types of cavities connected in series suggests that the desorption tends to be governed by a lower water potential, which is determined by the narrower sections of the pores. On the other hand, adsorption conditions tend to be governed by a higher water potential, which depends on the larger sections of the pores. It follows that the desorption curve depends on the size of the channels connecting the lumina, whereas the adsorption curve depends on the size of these lumina (Fortin 1979).

Figure 1 shows that water drainage between 0 and -100 J kg^{-1} of WP was about 19% MC. From the discussion above, it appears that this water would have been removed from the larger capillaries, principally the vessel lumina. This water would occupy the 21% volume of vessels reported for sugar maple by Panshin and de Zeeuw (1980). Table 1 indicates a capillary radius of 1.457 μm at -100 J kg^{-1} WP, clearly smaller than the radius of vessel elements estimated at 35 μm for sugar maple from Sebastian and Sastry (1974). Figure 1 shows that the curve of EMC changes sharply at about -100 J kg^{-1} WP and plateaus between -300 J kg^{-1} and $-2,000$ J kg^{-1} WP. Aspen wood does not show this feature (Cloutier and Fortin 1991), which indicates, that at high humidities, the EMC-WP relationship is dependent on species. This plateau would indicate that, within these WP values, openings controlling the retention and flow of water are scarce. At these water potentials, the water remaining in the wood would be localized in capillaries having a radius equal to or smaller than about 0.073 μm (Table 1). As discussed later, the plateau shown would correspond to the transition between the drainage of the fiber cavities and the drainage of cell walls and ray cell lumina. The free water remaining below -700 J kg^{-1} WP could be entrapped in the parenchyma cells as noted by Hart (1984).

Wood shrinkage–EMC relationships

Figure 2 shows the relationships between the EMC and the radial, tangential and volumetric shrinkage for sugar maple wood. Freehand curves were drawn taking into account the sorption state, in such a way that the point obtained in adsorption over distilled water is not linked to the others. The standard errors of the shrinkage values do not exceed the size of the symbols shown. This figure indicates that the shrinkage in the two principal directions, and consequently in volume, begins at about 42.5% EMC, which corresponds to -700 J kg^{-1} WP (99.484% RH). In this study, the amount of bound water or nominal FSP for sugar maple was evaluated at 31.1% EMC (adsorption over distilled water). Since shrinkage starts at 42.5% EMC, it is clear that, even at equilibrium, there is a simultaneous presence of free and bound water in the wood. Stevens (1963) reported a similar premature shrinkage for beech wood where the expected FSP had not yet been reached. Although his results were obtained at equilibrium, he attributed this behavior to the effects of casehardening stress during desorption. Our research establishes that, even at equilibrium, loss of bound water within the cell walls provokes shrinkage of wood before all free water has evaporated.

This implies that about 11.4% MC in liquid form is still retained in the wood when shrinkage is taking place. Hernández (1983) estimated that entire loss of this liquid water will be accomplished at about 63% RH, which corresponds to nearly 14% EMC. It would be of interest to know the location of this remaining free water. Given the level of WP concerned, this water could be entrapped in wood cells interconnected by the smallest capillaries or channels. This would correspond to the openings in the membranes of the simple pit pairs located between radial parenchyma cells. The presence of this free water principally in the rays would be confirmed, given that these wood elements are considered as the least permeable flow path in hardwoods (Siau 1984). Teesdale and MacLean (1918), cited by Wheeler (1982), stated that penetration by wood preservatives took place mainly in the vessels and fibers of maple with only slight penetration of the rays. Wheeler (1982) noted that the parenchyma–parenchyma pit membranes are thicker than both the intervessel pit membranes and the fiber–fiber pit membranes, and consequently are less efficient pathways for liquid flow. The remaining free water would fill the 18% volume of rays given for sugar maple by Panshin

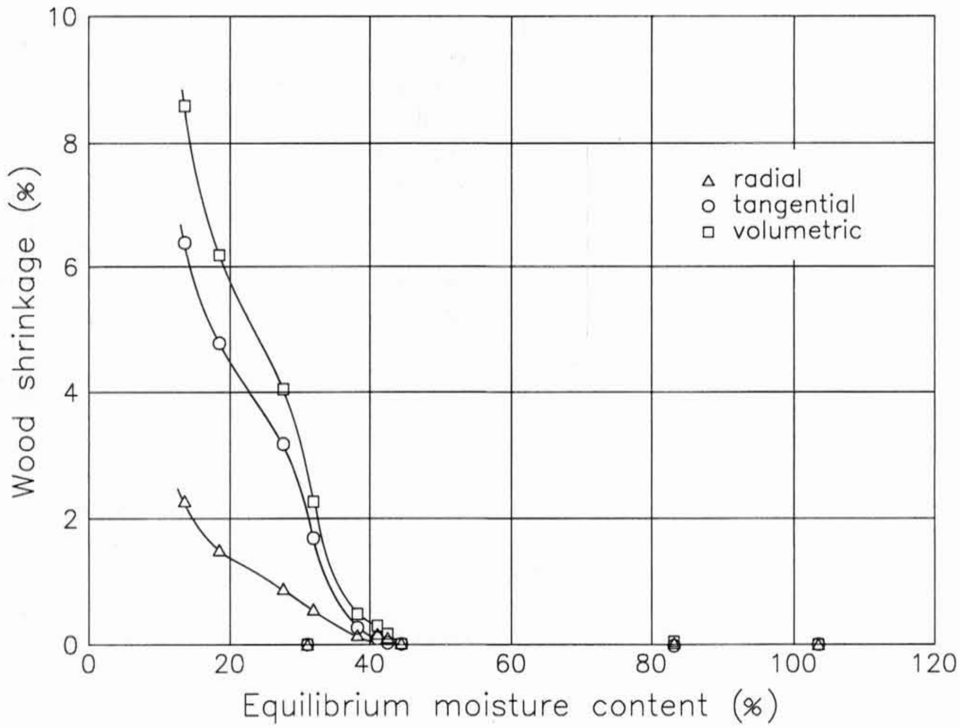

FIG. 2. Wood shrinkage of sugar maple sapwood as a function of the equilibrium moisture content.

and de Zeeuw (1980). The entrapment of free water in parenchyma cells reported by Hart (1984) for hickory and oak adds further support to the suggested hypothesis. According to Skaar (1988), the presence of water-soluble materials in the cavities of parenchyma cells may reduce the humidity at which moisture may condense. Additional work is, however, necessary before the distribution of remaining free water can be known.

In conclusion, the desorption process from the maximum MC for sugar maple wood can be divided into three parts: from the maximum MC to 42.5% EMC, there is drainage of free water; from 42.5% to 14% EMC, there is simultaneous desorption of free and bound water; and from 14% to 0% EMC, there is bound water desorption.

Tangential compression strength–EMC relationships

Relationships between EMC and the compliance coefficient s_{33} for sugar maple wood are shown in Fig. 3. This figure displays the compliance coefficient measured over the central part of the sample (40 mm), as well as the same coefficient estimated over its entire length. Freehand curves were drawn taking into account the sorption state, in such a way that the point obtained in adsorption over distilled water is not linked to the others. Standard errors of the compliance coefficients are shown only when they exceed the symbol size.

A comparison between the two curves in Fig. 3 shows that there is a heterogeneous distribution of the strain inside the wood specimen. Entire compliance coefficient s_{33} was on average 32% higher than the median compliance coefficient. This behavior is also apparent from Hernández (1993a), who used specimens having the same dimensions but tested under only three humidity conditions. This difference in strain can be partially attributed to the stress concentrations introduced by lateral restraints near the end surfaces of a sample, which are in contact with the testing machine (Bodig

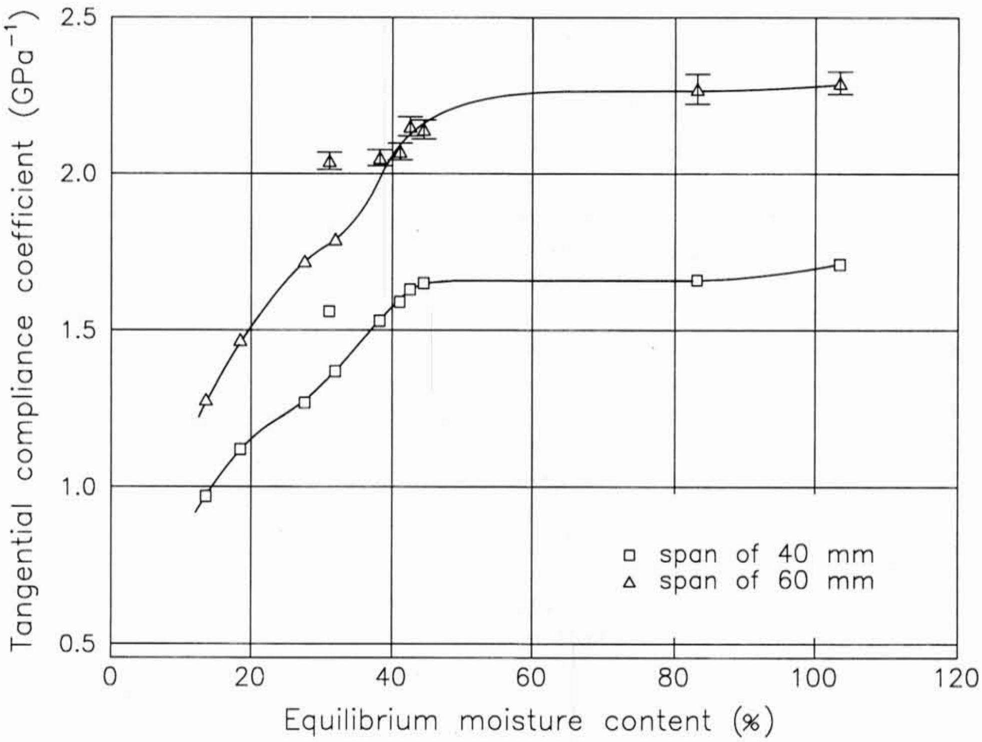

FIG. 3. Compliance coefficients s_{33} in tangential compression of sugar maple sapwood as a function of the equilibrium moisture content.

and Jayne 1982). Standard test methods specify the use of the central part of the specimen for measurement of deformation.

The presence of hysteresis at saturation is apparent when comparing the compliance coefficient obtained over distilled water (nominal FSP) with a similar EMC, but measured from the boundary desorption curve. The EMC over a saturated solution of $ZnSO_4$ was 32.0%, which approaches the nominal FSP of 31.1%. The difference between the compliance coefficients obtained under both humidity conditions was 14% for both types of strains evaluated.

On the other hand, these graphs show that there is a significant difference between the compliance coefficient obtained at the nominal FSP and the one measured at the maximum MC. This difference was 9.6% and 12.2% for the central-part-deformation and for the entire-specimen-deformation, respectively. Effects of the strain rate on strain were controlled since the mechanical tests were conducted at 0.40 mm/mm/min for all moisture conditions. The difference in strength between both moisture conditions could rather be attributed to the effect of the moisture saturation treatment before the desorption test.

The moisture saturation treatment was carried out by immersing wood specimens at an initial 14% MC in distilled water overnight. Vacuum-atmospheric pressure cycles were then done up to full saturation. This treatment could have induced some permanent creep in the cell walls, which would increase the compliance coefficient. An analysis of the shrinkage data depicted in Fig. 2 indicates a relatively high tangential shrinkage coefficient compared with previous studies (USDA 1974; Goulet and Fortin 1975; Hernández 1993b). The radial shrinkage coefficient appears unaffected. As a result, T/R shrinkage ratios for all MCs could be elevated. Consequently, this probable effect of the saturation treatment would produce an underestimation of the effect of the hysteresis at saturation on the compliance coefficient, and its overvaluation on the shrinkage behavior of wood.

On the other hand, the EMC at which changes in wood properties begin would remain unaffected by this saturation treatment. Figure 3 shows that gain in tangential strength starts at 42.5% EMC, as noted earlier from the shrinkage measurement results. This EMC value would represent the practical FSP proposed by Siau (1984) as that MC corresponding to abrupt changes in physical properties of wood. However, it is necessary to take into account that a part of this moisture content, in this case the free water, does not participate under any circumstances in these abrupt changes. Some practical consequences may be derived from these results. The terms "green" or "water-saturated" for wood having an MC above but approaching the nominal FSP, for example, should be reconsidered.

SUMMARY AND CONCLUSIONS

Moisture adsorption and desorption experiments were performed with sugar maple wood using a single step procedure at room temperature. Once equilibrium was reached, tangential compression tests and shrinkage measurements were undertaken. The results of these tests lead to the following main conclusions:

1. At equilibrium, the radial and tangential shrinkage and, consequently, the volumetric shrinkage begin significantly above the nominal fiber saturation point.
2. At equilibrium, the moisture content affects the tangential compliance coefficient beyond the nominal fiber saturation point.
3. In the desorption phase, loss of bound water takes place beginning at nearly 42.5% EMC in the presence of free water.

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