J Wood Sci (2001) 47:185–191

ORIGINAL ARTICLE

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Physical and mechanical properties of wood after moisture conditioning

Received: March 22, 2000 / Accepted: May 22, 2000

Abstract Some properties of wood (hinoki: *Chamaecyparis obtusa*) moisture-conditioned by an adsorption process from a dry state and by two desorption processes (from a water-saturated state and from a state with a moisture content slightly below the fiber saturation point) were investigated. The moisture contents of wood conditioned by the adsorption process and by the desorption process continued to approach to one another for the moisture-conditioning period of over 50 weeks. Accordingly, sorption hysteresis should be regarded as a transitional phenomenon that occurs during the process of approaching the true equilibrium, which requires a long time. The wood conditioned by the desorption process beginning from a water-saturated state showed slightly smaller dimensions than those conditioned by the adsorption process with the same moisture content; however, the wood conditioned by the desorption process from a moisture content below the fiber saturation point showed slightly larger dimensions than those conditioned by the adsorption process. The wood conditioned by the adsorption process from a dry state showed a higher modulus of elasticity and modulus of rupture than did the wood conditioned from a water-saturated state with the same moisture content. The mechanical properties of the wood also varied based on the states at which the desorption process was started. This is a notable characteristic of the relation between the drying condition and the mechanical properties of wood.

Key words Moisture-conditioned wood · Sorption hysteresis · Dimensional change · Modulus of elasticity · Modulus of rupture

Introduction

Regarding the sorption hysteresis of wood and cellulosic materials, that the amount of moisture held by these materials depends not only on the equilibrium of the relative humidity but on the direction from which the equilibrium is approached has been well known for many years. Summarizing the explanation by Urquhart[1] followed by those of Smith[2] and White and Eyring,[3] the sorption hysteresis of water by cellulosic materials can be attributed to irreversible uncoupling and recoupling of hydroxyl groups in the cellulose structure; that is, the uncoupling occurs during adsorption, and the recoupling occurs at relatively delayed stages in the desorption process. These delayed stages are due to the rigid structure of the cellulose chains in which a hydroxyl group hardly recouples when a water molecule is desorbed. Consequently, the available sites for the adsorption of water are larger during the desorption process than during the adsorption process. In other words, water molecules bound directly to sites in the cellulose chain are larger during the desorption process than during the adsorption process.

Stamm noted that hysteresis is not due to lack of attaining the true sorption equilibrium because greatly extending the sorption time shows no tendency to eliminate hysteresis.[4] However, we have not found a study in which hysteresis was traced for more than several weeks. The above-mentioned hysteresis theory implies the existence of a time lag for the response of cellulose chains during the desorption process. Furthermore, it has been observed that hysteresis becomes obscure with elevating temperatures. These findings suggest that hysteresis is not a phenomenon that occurs in an equilibrium state. Therefore, the possibility that sorption hysteresis is a transitional state until true equilibrium is reached remains, even if a long time is necessary to attain true equilibrium.

In any event, the existence of sorption hysteresis suggests that wood moisture-conditioned during the adsorption process and that during the desorption process differ in terms of their existing *states* of moisture at the same mois-

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Table 1. Saturated salt solutions used to regulate relative humidities

Salt	Relative humidity[5] (%)	Usage for each set of moisture conditioning					
		A	B	C	D	E	F
LiBr·2aq	6.3	+	+		+		
LiCl·aq	11.1				+		
CH_3COOH	22.8	+	+	+	+		+
MgCl·6aq	33.1	+	+		+	+	
K_2CO_3·2aq	42.6	+	+		+		+
$Mg(NO_3)_2$·6aq	54.3	+	+	+	+	+	
NH_4NO_3	64.9	+	+				+
$(CH_3COO)_2Mg$·4aq	74.0				+	+	
NaCl	75.4	+	+	+			
$(NH_4)SO_4$	80.1				+		
KCl	84.9	+	+	+	+		+
Na_2CO_3·10aq	92.0			+			
KNO_3	93.8	+	+		+	+	
K_2SO_4	96.5	+	+		+		
$CaSO_4$·2aq	98.0	+		+	+		

A and B: long-term examinations of the moisture contents and the dimensions in the adsorpton process beginning from a dry state and in the desorption process beginning from a water-saturated state, respectively. C and D: examinations of static bending properties and the moisture contents and the dimensions in the adsorption and desorption processes from the water-saturated state, respectively. E and G: examinations of mechanical properties in bending and the moisture contents and dimensions in the adsorption and desorption processes beginning from a moisture content slightly below the fiber saturation point, respectively
aq, water

ture *content.* It is interesting how such a difference in the existing state of moisture affects various properties of wood. Hence the sorption hysteresis of wood was traced for 50 weeks, and differences in dimension and certain mechanical properties of wood moisture-conditioned during the adsorption process and two different desorption processes were investigated.

Materials and methods

The present study consisted of six sets of moisture-conditioned wood, as shown in Table 1: A and B in Table 1 are long-term measurements of the moisture content and the dimensions of the wood samples conditioned during the adsorption process from a dry state and during the desorption process from the water-saturated state, respectively; C and D are evaluations of some mechanical properties in static bending and the moisture contents and dimensions of the samples conditioned from a dry state and from a water-saturated state, respectively; and E and F are evaluations of the static bending properties and the moisture contents and dimensions of the samples conditioned from a dry state and during the desorption process from a moisture content of about 25%, which is slightly below the fiber saturation point (FSP), respectively.

Wood samples

Wood samples were obtained from the outer region of heartwood with a straight grain in a log of Japanese cypress (hinoki: *Chamaecyparis obtusa*). For measuring moisture contents and dimensions, about 70 cross-sectional wood pieces [40 radial (R) × 20 tangential (T) × 4 longitudinal (L) mm] were successively cut from three wood sticks having cross sections of 40 (R) × 20 (T) mm. Their dimensions were measured in the tangential and radial directions with a precision of 0.01 mm using a screw micrometer after swelling in water (1 week at 30°C). Their weights and dimensions (dry weights and dimensions) were measured after vacuum drying (more than 50 h at room temperature). After oven-drying for 15 h at 105°C, their dimensions (oven-dried dimensions) were measured again; then the samples that appeared to have dried inadequately or abnormally were discarded, judging from the dry and oven-dried dimensions.

For examinations A and B, 105 wood pieces obtained from two wood sticks were divided into 21 groups of five pieces each by selecting five pieces, one by one, for 21 times. Each group was moisture-conditioned using 21 different methods. For examinations E and G, the wood pieces obtained from a single stick were divided into eight groups of five each in a manner similar to that described above. Each group was moisture-conditioned using eight different methods.

For the static bending tests, cross-sectional wood pieces [100 (R) × 15 (T) × 4 (L) mm] were successively cut from three wood sticks having cross sections of 100 (R) × 15 (T) mm. Each wood stick was marked along an annual ring near the central part of the radial direction, and the loading point for the static bending test was at the marked position. The wood pieces were grouped in a manner similar to that described above.

All wood samples were kept in a desiccator containing dry calcium chloride until the next treatment stages.

Moisture conditioning

For moisture-conditioning during the adsorption process from a dry state, each sample to be measured for moisture content and dimension (examinations A and E) was placed in a separate weighing bottle and dried at 105°C for 15h in an oven to ensure that it was moisture-free. Then each group of five wood pieces was separately placed in different desiccators containing a saturated aqueous solution of the various salts listed in Table 1. The desiccators were placed in a room regulated at 20°C for 50 weeks for examination A and for 9 weeks for examination E. Changes in the weight and the tangential and radial dimensions of the samples with conditioning time were traced. For the static bending test, each group of samples was placed separately into different desiccators containing a saturated aqueous solution of the various salts listed in Table 1 (examinations C and E) immediately after drying at 105°C for 15h. The moisture-conditioning periods were 4 weeks for examination C and 9 weeks for examination E.

For moisture-conditioning during the desorption process from a water-saturated state (examinations B and D), the wood samples were soaked in water under vacuum for a period of less than 10min, and water was wiped off the surface of the samples with a paper towel. Then each group of five samples to be measured for the moisture content and dimension (examination B) and for measurement of the static bending properties (examination D) was placed separately into different desiccators containing a saturated aqueous solution of the various salts listed in Table 1 in a manner similar to that described above. The moisture-conditioning periods were 50 weeks for examination B and 4 weeks for examination D.

For moisture-conditioning during the desorption process from slightly below the FSP (examination F), all the samples were placed in a desiccator containing a saturated aqueous solution of $CaSO_4 \cdot 2aq$ ($2H_2O$) until the moisture content of the samples reached about 25%. The samples were then moisture-conditioned for 4 weeks in a manner similar to that described above.

Determination of moisture content and swelling

For moisture-conditioning during the adsorption processes and the desorption process from below the FSP, the moisture contents and swelling of the conditioned samples were determined using the dry weights and tangential and radial dimensions of the samples and those of moisture-conditioned samples. In the case of desorption from the water-saturated state, some extractives were leached from the samples, and they possibly affected the weights and dimensions of the samples. Therefore, the samples were dried at 105°C for more than 24h. Their dry weights and dimensions were then measured again. These dry weights and dimensions were used to determine the moisture content and swelling of the samples. The degree of swelling was evaluated from the cross-sectional swelling relative to that in water to eliminate variations among wood samples.

Static bending test

Dry and water-saturated samples for examination C and dry samples for examination D were prepared in addition to the moisture-conditioned samples for the static bending test. The prepared test samples were sealed using a polyethylene film prior to the test to avoid changes in moisture content. A testing machine (Tensilon UTM-4L; Toyo Sokki, Japan) was used for the static bending test in the radial direction under the following conditions: span 80mm of central loading, down-speed of a cross-head 4mm/min. The tests were discontinued when the samples had broken down completely. Immediately after the test, the wood samples were unsealed, and their widths and thicknesses were measured. The modulus of elasticity (MOE) and modulus of rupture (MOR) were calculated using the conventional method employing the obtained stress-strain diagrams and the dimensional data.

Results and discussion

Change in the sorption hysteresis with time

Figure 1 shows the sorption hysteresis for the wood samples moisture-conditioned for 50 weeks by the adsorption process and for those by the desorption process. These were no apparent differences from previous results.[4,6,7]

Figure 2 shows the ratios of the moisture content of wood samples conditioned for 3, 10, and 50 weeks at 20°C by the adsorption process and those conditioned by the desorption process (adsorption/desorption ratios) as a function of relative humidity (RH). The ratios of all the conditioning periods increased with increases in RH except the values of the samples conditioned for 3 weeks at high RH. It can also seen that the ratios increased with increases in the conditioning period over the entire RH range. This means that the width of the hysteresis loop narrowed with increases during the conditioning period.

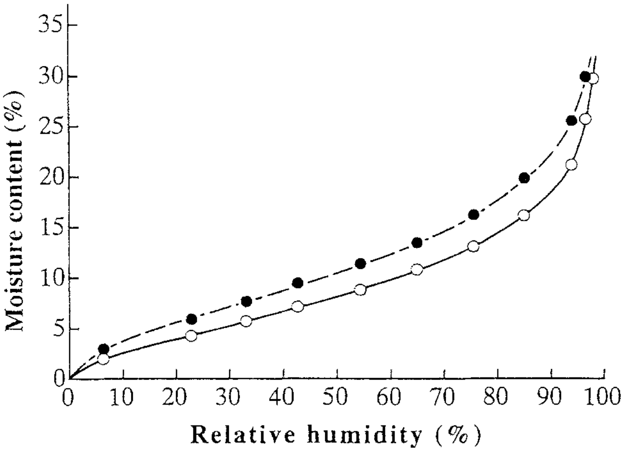

Fig. 1. Adsorption (*open symbols*) and desorption (*solid symbols*) isotherms of wood moisture-conditioned for a period of 50 weeks

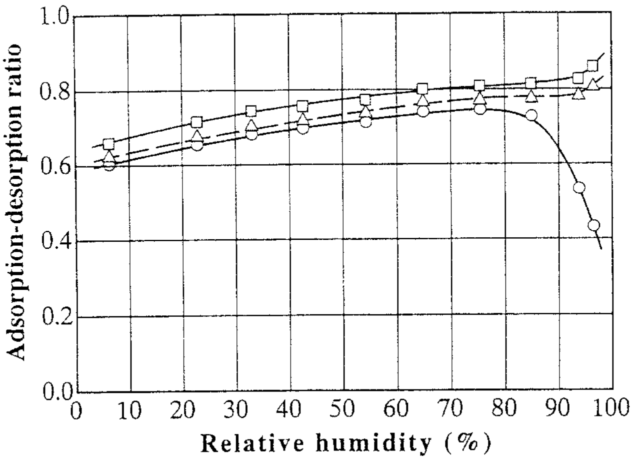

Fig. 2. Adsorption/desorption ratios for wood moisture-conditioned for 3 weeks (*circles*), 10 weeks (*triangles*), and 50 weeks (*squares*)

Stamm and Woodruff found that the adsorption/desorption ratios were 0.79–0.86 at 10%–90% RH for white spruce at 20°C.[6] These values are significantly higher than the present values for the samples conditioned for 50 weeks, even though their conditioning times were only 24 h or less. The ratios calculated from the data of Kelsey were 0.64–0.81 at 10%–90% RH for klinki pine at 25°C.[7] These values are comparable to the present values for the samples conditioned for 10 or 50 weeks, though Kelsey's conditioning times were described as 2 days at most. Considering the differences of the conditioning periods between present study and previous studies, the adsorption/desorption ratios in the studies referred to above are thought to be somewhat higher than those in the present study.

These differences cannot be attributed to the difference in the adsorption and desorption rate caused by the difference in the sample shape or apparatus used for moisture-conditioning because the moisture-conditioning periods differed to such a degree. Another possible explanation for the difference may lie in the fluctuations of the RH or temperature (which could itself also cause fluctuations in the humidity level) in the apparatus used during moisture-conditioning periods in the previous studies. Stamm and Loughborough[8] proved that such fluctuations narrow the hysteresis loop. In light of this, the present study was performed by desiccators containing various saturated aqueous solutions of salts for moisture-conditioning. Moreover, the room in which the desiccators were placed was closely temperature-controlled based on the monitored record, which showed variations of temperature of less than 0.2°C over the conditioning period. Accordingly, the present result should be highly reliable. In fact, this experiment was stopped after 50 weeks because a larger temperature variation was found in the monitored record before the next measurement was planned.

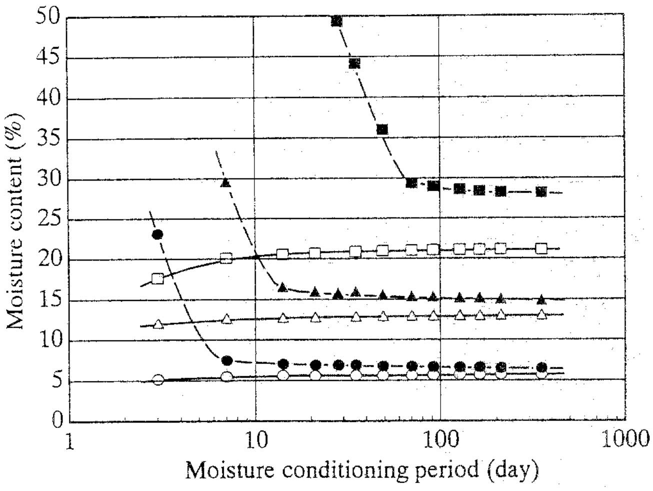

Fig. 3. Changes in moisture content during the adsorption and desorption processes with an increasing moisture-conditioning period. *Circles*, conditioned at 33% relative humidity (RH); *triangles*, 75.4% RH; *squares*, 93.8% RH; *open symbols*, adsorption process, *solid symbols*, desorption process

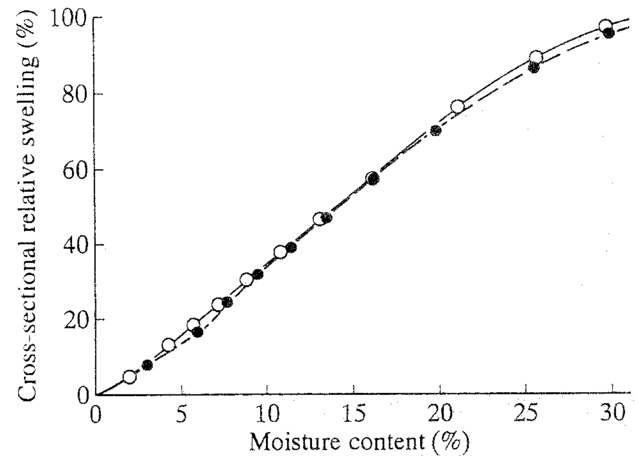

Fig. 4. Difference in cross-sectional relative swelling between wood moisture-conditioned for 50 weeks during the adsorption process (*open symbols*) and that during the desorption process from a water-saturated state (*solid symbols*)

The changes in the moisture contents of the samples conditioned during the adsorption and desorption processes at three RHs for 50 weeks are shown in Fig. 3. It was observed that the differences between the moisture contents during the adsorption process and those during the desorption process decreased with increases in the conditioning period at all the RHs examined. Thus, true adsorption and desorption equilibriums were obviously not attained even at 50 weeks. Accordingly, sorption hysteresis should be regarded as a transitional phenomenon during the process of achieving true equilibrium, for which a long time is required.

Difference in dimensions of wood after different moisture-conditioning methods

Figure 4 shows the cross-sectional relative swelling of samples conditioned during the adsorption process from the dry state and those conditioned during the desorption pro-

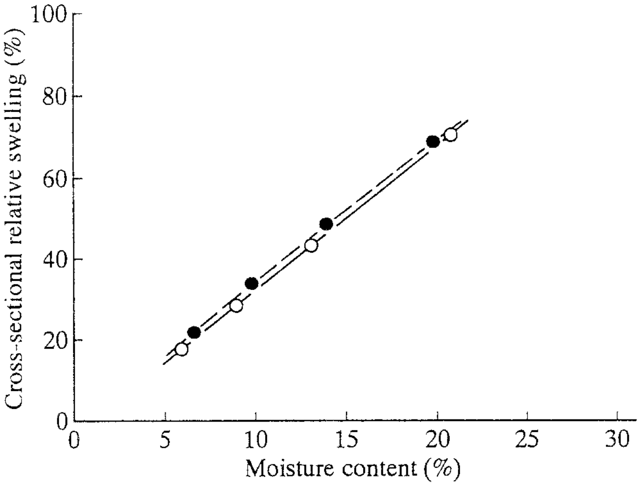

Fig. 5. Difference in cross-sectional relative swelling between wood moisture-conditioned for 9 weeks during the adsorption process (*open symbols*) and that during the desorption process from a moisture content slightly below the fiber saturation point (FSP) (*solid symbols*)

cess from the water-saturated state for 50 weeks as a function of their moisture content. Comparing the swelling produced by the adsorption process with that produced by the desorption process, the former showed slightly more swelling than the latter in a range above 25% moisture content and in a range below 10% moisture content, though the difference in swelling for both processes was not apparent in the intermediate region. Similar results were found for all conditioning periods examined. The difference in the degree of swelling in the higher moisture content range was accounted for by the shrinkage of the samples due to the decrease in moisture content near the surface of the samples below the FSP despite the inner potion of the samples remaining above the FSP.

The cross-sectional relative swelling of samples conditioned for 9 weeks during the adsorption process beginning at a dry state and those during the desorption process from about 25% moisture content (which was less than the FSP) are compared in Fig. 5. The moisture contents of the samples after the adsorption process were slightly larger than those after the desorption process over the entire moisture content region. Similar results were obtained for the shorter conditioning periods, though the difference between the moisture contents for both processes decreased with increased conditioning duration. These results are inconsistent with the results shown in Fig. 4. This discrepancy could be attributed to the difference in desorption processes in that the samples were conditioned from the water-saturated state in the case shown in Fig. 4, whereas the samples were conditioned from a moisture content below the FSP in the case shown in Fig. 5. In other words, the former samples included liquid water, whereas the latter samples did not.

Now, let us consider the difference between drying from a water-saturated state and drying from a moisture content below the FSP. The samples used in the present study had a length of 4mm, which is somewhat longer than the 3.5mm average length of tracheid cells of Japanese cypress.[9] Hence, in the case of drying from a water-saturated state, the liquid water included in some of the tracheid cells, of which both ends were closed, had to evaporate through the pit membrane pores. In this situation, the so-called liquid tension acted on the cell wall of the tracheid corresponding to the curvature of the largest meniscus in the pit membrane pores; thus, the tracheid tends to contract toward its interior.[10]

On the other hand, for samples dried from a moisture content below the FSP, only moisture included in the cell walls evaporate. Even in this situation, so-called drying stress caused by a moisture gradient between an area near the surface and the interior of the samples develops. As a result, these wood samples shrink less than those that undergo stress-free drying. During the adsorption process, the wood samples are subjected to compressive stress, in contrast to the drying process; however, the set caused by compressive stress during the wetting process should be much smaller than that caused by tensile stress during the drying process. Because wood samples used in the present study were thin cross sections with a thickness of 4mm in the longitudinal direction, the stress and set that occurred during both process we deduced to be much smaller than those of a wood sample with larger dimensions. However, the possibility that similar stress and set occurred in microportions, such as portions between the surface and inner parts of cell walls, cannot be ignored.

Based on the above description, the result shown in Fig. 5 that the samples showed more swelling during the desorption process than during the adsorption process is accounted for by the drying stress. On the other hand, the result shown in Fig. 4, in which the swelling was slightly more in the samples during the adsorption process than during the desorption process from a water-saturated state, is attributed to the fact that the liquid tension has a greater effect than the drying set.

Difference in mechanical properties of wood after different moisture-conditioning methods

Figure 6 compares the MOE and MOR of samples conditioned for 4 weeks by the adsorption process and those conditioned by the desorption process from the water-saturated state as a function of relative swelling. The MOE and MOR are shown as values relative to those in a dry sate. The MOE during the adsorption process decreased with increases in relative swelling above about 20%, until which the MOE did not change much; in contrast, the MOE during the desorption process decreased with increases in relative swelling over the entire relative swelling range. The MOR in the adsorption process reached its maximum at about 25% relative swelling and then decreased with increased swelling; the MOR, during the desorption process, maintained a plateau and then decreased as the swelling increased. As a result, the MOE and MOR during the desorption process were lower than the values for the adsorption process, especially in the area of medium swelling.

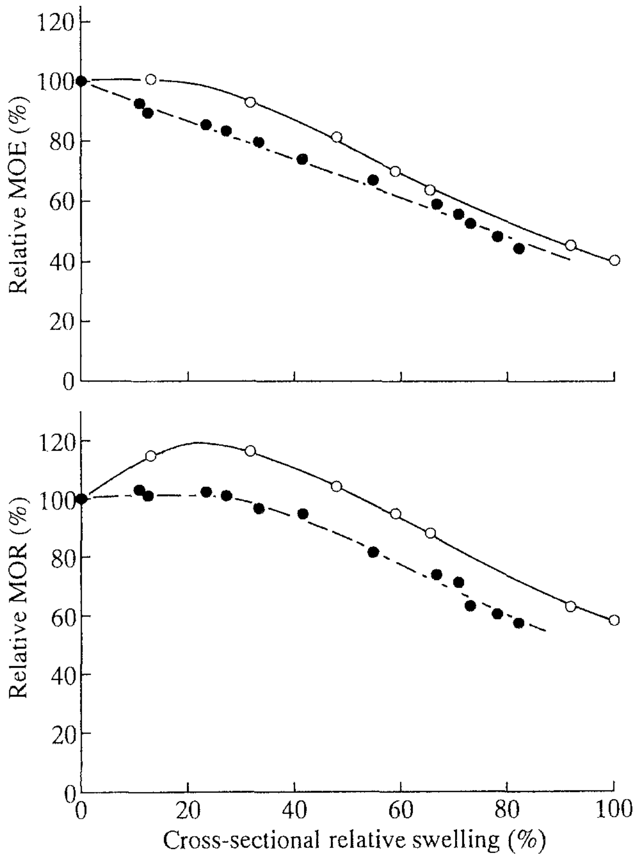

Fig. 6. Difference in modulus of elasticity (*MOE*) and modulus of rupture (*MOR*) for wood moisture-conditioned for 4 weeks by the adsorption process (*open symbols*) and by the desorption process from a water-saturated state (*solid symbols*) as a function of the cross-sectional relative swelling

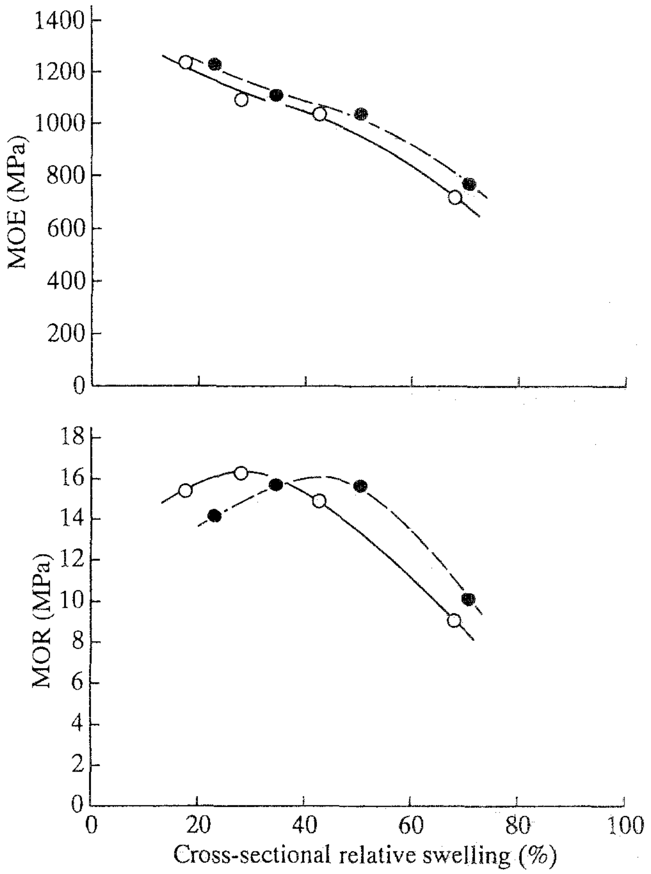

Fig. 7. Difference in MOE and MOR for wood moisture-conditioned for 4 weeks by the adsorption process (*open symbols*) and by the desorption process from a moisture content below the FSP (*solid symbols*) as a function of cross-sectional relative swelling

In Fig. 7 the MOE and MOR of samples conditioned for 4 weeks by the desorption process beginning at 25% moisture content (below the FSP) are compared with those of samples conditioned by the adsorption process. The wood samples after the desorption process showed slightly larger MOEs than those after the adsorption process. The MOR reached a maximum with both processes, but the maximum percent of cross-sectional swelling differed for the two processes.

Summarizing the results shown in Figs. 6 and 7, the MOEs and MORs of the samples conditioned by the desorption process from the water-saturated state were lower than those conditioned by the adsorption process over the whole swelling range, and the samples from the desorption process with a moisture content below the FSP showed slightly higher MOEs and MORs than those from the adsorption process, though this was not the case for MORs in the lower swelling regions. This indicates that the mechanical properties of the wood conditioned by the desorption process from a water-saturated state are lower than those conditioned from a moisture content below the FSP, except the MOR in a lower swelling region.

This discrepancy regarding the mechanical properties of the two desorption processes could be due to the difference in the desorption processes; that is, the former was the desorption process used on samples that contained liquid water, and the latter was the desorption process used on samples that did not contain liquid water. As described above, the liquid tension acted locally on the cell walls of the tracheid, which were filled with liquid water; thus, the tracheid tends to contract toward its inside. Such local stress and deformation affected other parts of the samples, producing inhomogeneous stress and strain and decreasing the wood samples' resistance to an external force. This is thought to account for the finding that the wood moisture-conditioned by the desorption process from a water-saturated state showed lower MOEs and MORs than those with a moisture content below the FSP.

As described above, the dimensions of the wood samples conditioned by different methods differed from each other at the same moisture content in terms of the liquid tension and the moisture gradient in the samples during the desorption process. Hence, in a strict sense, relative swelling is not a measure of the amount of moisture included in the cell walls of the wood. Thus the difference in MOE and MOR between the adsorption process and the desorption process beginning at a moisture content below the FSP was further elucidated using Fig. 8, in which the MOE and MOR were plotted against the moisture content.

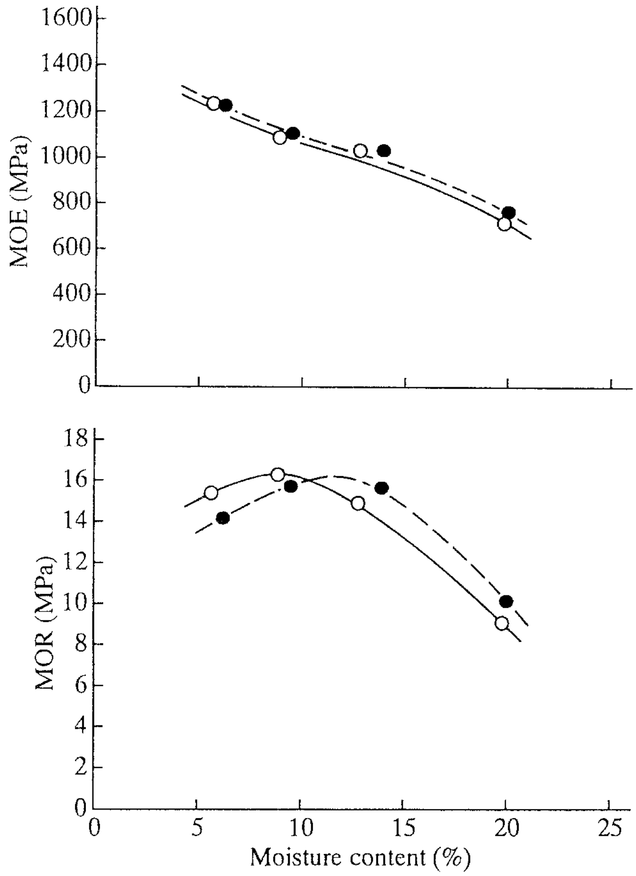

Fig. 8. Difference in MOE and MOR for wood moisture-conditioned for 4 weeks by the adsorption process (*open symbols*) and by the desorption process from a moisture content below the FSP (*solid symbols*) as a function of the moisture content

The difference in the MOEs in Fig. 8 was slight, although a difference in the moisture content at which the MOR reached a maximum was similar to that shown in Fig. 7. This suggests that the effect of reinforcing a weak point due to inclusions of a proper amount of moisture in the cell walls[11,12] is reflected more distinctly in the MOR than in the MOE, and that the existing state of moisture in the cell walls during the adsorption and desorption processes differs. Moreover, it is notable that the decrease in the MOR during the desorption process was seen at a moisture content below 10%. This suggests that generation of a weak point in the cell wall or, more concretely, an incomplete rearrangement of molecular chains of cell wall constituents caused by desorption of moisture from the cell walls occurs extensively at a moisture content below 10%.

In any event, the present results regarding these mechanical properties suggest that wood conditioned from a dry state has higher elasticity and strength than wood conditioned from a water-saturated state with common moisture contents, and that the mechanical properties of the wood vary in terms of the difference in desorption processes, that is, in regard to either a water-saturated state or a moisture content below FSP.

Conclusions

A sorption hysteresis loop of wood by moisture narrowed continuously over a moisture-conditioning period of 50 weeks. Based on this result, the sorption hysteresis should be regarded as a transitional phenomenon that occurs during the process of achieving true equilibrium, which requires a long interval. The wood moisture-conditioned by the desorption process from a water-saturated state showed slightly smaller dimensions than that conditioned by the adsorption process; however, wood conditioned by the desorption process from a moisture content below the FSP showed a slightly larger dimension than that conditioned by the adsorption process. These results are attributable to the liquid tension and drying stress during the desorption processes. Wood moisture-conditioned by the adsorption process from a dry state had higher MOEs and MORs than wood conditioned by the desorption process from a water-saturated state with the same moisture content: The mechanical properties of the wood also varied with the difference in desorption processes (i.e., the desorption processes beginning from a water-saturated state or from a moisture content below the FSP). This is notable in terms of the relation between the drying condition and the mechanical properties of wood. This relation should be investigated in more detail in the near future.

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