J Wood Sci (2002) 48:81–85

NOTE

Ikuho Iida · Sulaeman Yusuf · Ugai Watanabe
Yuji Imamura

Liquid penetration of precompressed wood VII: combined treatment of precompression and extraction in hot water on the liquid penetration of wood

Received: June 21, 2000 / Accepted: January 29, 2001

Abstract The object of this study was to determine the cause of differences in the improvement in liquid penetration of precompressed wood species. The maximum amount of water uptake by the capillary rise method and changes in the aspirated pits seen with scanning electron microscopy before and after of preextraction and precompression were investigated using heartwood samples of four softwoods. The height of penetration and the weight by the capillary rise method for preextractive wood powders are discussed. Three wood species and *Larix leptolepis* showed marked increases in the amount of solution uptake after precompressed treatment only. *Larix leptolepis* wood required compression after extraction by boiling in water. These differences among wood species were caused by the accumulation of extractive material. It was also recognized that the accumulative material in *Larix* wood has plasticity and that in *Pseudotsuga* is brittle. Based on these results it was found that it is difficult to destroy aspirated pits in the former and easy in the latter. On the other hand, the difference in penetration of each wood species was caused by the quantity and quality of the extraction material in addition to the extent of the wettability of the surface of the cell cavity as well as aspirated pit.

Key words Refractory wood · Large deformation · Liquid penetration · Precompressed wood · Aspirated pit

Introduction

In previous papers[1–4] we proposed a method for applying a large deformation perpendicular to the grain before liquid impregnation of a wood sample to accelerate wood permeability. In those studies we investigated various factors affecting the penetration level (i.e., the ratio of compression, moisture content, and temperature in which the specimens were compressed; the length and thickness of the specimens; preservative solutions; and the wood species).

This compression method is intended to separate and destroy aspirated pits in the wood during the compressive deformation process, leading to increased liquid uptake during the recovery process of the deformation owing to volumetric pressure. The results indicated that the precompression treatment method increased the penetration level with a small decrease in the mechanical properties of the treated wood. The acceleration of penetration properties differed among wood species. In addition, the most suitable press condition for each species was different and depended on the moisture content and temperature of its compressed wood. This condition suggests that each wood species differs in terms of the quantity and qualitative properties of the aspirated pits, thereby causing different magnitudes of wettability within the lumen surface.

The objectives of the present study were to investigate the penetration process, determine the maximum liquid uptake, and explore the changes in aspirated pits of wood treated by a combination of compression and hot-water extraction. Powders of the treated wood and an untreated control were used to measure the height of penetration by the capillary attraction method.

I. Iida (✉)
Faculty of Agriculture, Kyoto Prefectural University, Shimogamo Nakaragi-cho, Sakyo-ku, Kyoto 606-8522, Japan
Tel. +81-75-703-5639; Fax +81-75-703-5639
e-mail: i_iida@kpu.ac.jp

S. Yusuf
R & D Center for Applied Physics, Indonesian Institute of Sciences, Serpong, Indonesia

U. Watanabe · Y. Imamura
Wood Research Institute, Kyoto University, Gokasho, Uji, Kyoto 611, Japan

Part of this report was presented at the 46th annual meeting of the Japan Wood Research Society, Kumamoto, April 1996

Materials and methods

Materials

The heartwoods of four coniferous species (*Cryptomeria japonica* D. Don., *Picer sitchensis Carr.*, *Larix leptolepis* Gordon, *Pseudotsuga menziesii* Franco) were used in this experiment. The size of specimens subjected to compression and extraction treatment was 10mm (radial direction) × 10mm (tangential direction) × 120mm (longitudinal direction).

Extraction and compression treatments

Prior to compression treatment, hot-water extraction was performed on all samples daily: 10 hours in boiling water and 14 hours of conditioning at room temperature. Specimens of *Cryptomeria japonica* D. Don., *Picer sitchensis* Carr., and *Pseudotsuga menziesii* Franco were then soaked in cold water for 1–3 days followed by reextraction with boiling water for 5 days. For the *Larix leptolepis* Gordon sample, five levels of advanced treatment were applied: cold-water extraction and hot-water extraction for 1, 3, 5, and 10 days.

The air-dried and water-saturated samples resulting from the extraction treatments were compressed to deformation ratios of 0%, 10%, 20%, and 50% for *Larix leptolepis* Gordon and to deformation ratios of 0% and 50% for the other three species. A material testing machine of 10 tons capacity was used for the compression treatment, and the test machine was operated with a deformable speed of 1.5mm/min. The amount of deformation was measured with a dial-gauge for deformable measurement. The water-saturated sample was then dried to the air-dried state under restraint of the deformation, and preset-fixed wood was made.

Measuring the amount of penetration

The specimens mentioned above were used to measure permeability at 360min by the capillary attraction method. The test sample was placed in a standing position on one side of the cross section at the surface of the dyeing solution.

The recovery dimension of preset-fixed wood was calculated according to the following formula:

$$R_S = (L_R - L_C)/L_C \times 100\ (\%)$$

where R_S is the ratio of dimensional recovery after solution uptake; L_R is the dimension of the radial direction after solution uptake; and L_C is the dimension of the radial direction before solution uptake.

SEM observation

Scanning electron microscopy (SEM) was used to observe the cell wall and aspirated pit characteristics in precompressed and recovered wood before and after the extraction treatment. Samples of the radial-longitudinal surfaces were prepared by splitting, mounting, and coating them with gold prior to observation using a Hitachi S-500 SEM.

Measuring liquid uptake in wood powder before and after extraction

Part of each extracted and compressed specimen was prepared in the form of powder (60–80 mesh). A 1-g powder sample was packed into a 2-ml pipet and subjected to wetting treatment. The solution uptake was measured by the height of penetration and the increase in weight.

Results and discussion

Solution uptake of karamatyu wood (*Larix leptolepis* Gordon) after extraction and precompression

Figure 1 shows the solution uptake by *Larix leptolepis* Gordon treated with the various extraction steps without precompressive deformation. It shows that the control sample (nonextracted samples) took up solution with a maximum value of only 0.05g/cm^2, whereas the wood samples that underwent extraction treatment showed a marked increase in uptake with longer times. Maximum uptake values of about 0.32–0.34g/cm^2 were seen in the samples extracted for 3–10 days, with no difference in uptake among the samples treated for 3–10 days. This result may indicate that extraction treatment is the limiting factor in improving permeability.

Figure 2 shows the maximum solution uptake of the wood samples treated by a combination of preextraction in hot water and precompressive deformation treatments. The four bar graphs of each group show the difference in the degree of extraction conditions, and each value for a particular group shows the amount of solution uptake by

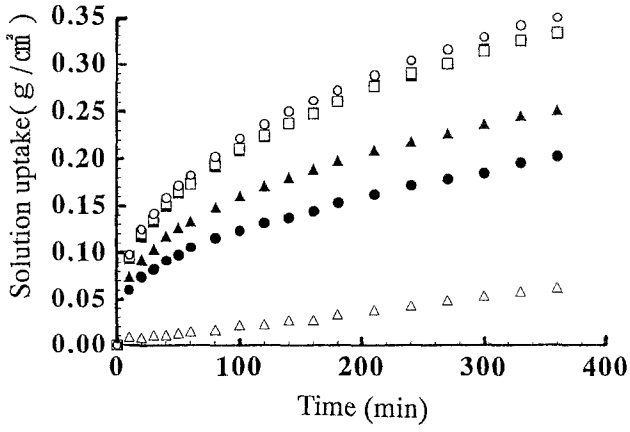

Fig. 1. Results of solution uptake for *Larix leptolepis* Gordon wood treated with hot-water extraction. *Open triangles*, unextracted wood in air dry; *filled circles*, treated with extraction in cold water; *filled triangles*, treated with 1 day of hot-water extraction; *open squares*, treated with 3 days of hot-water extraction; *open circles*, treated with 5 days of hot-water extraction; *filled squares*, treated with 10 days of hot-water extraction

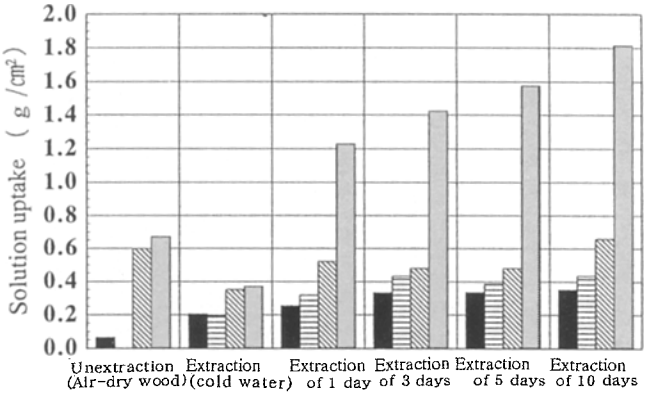

Fig. 2. Maximum amount of solution uptake of wood after the combined treatment of precompression and hot-water extraction for *Larix leptolepis* Gordon. *Solid bars*, without compression; *horizontally cross-hatched bars*, compression of 10%; *diagonally cross-hatched bars*, compression of 20%; *box-hatched bars*, compression of 50%

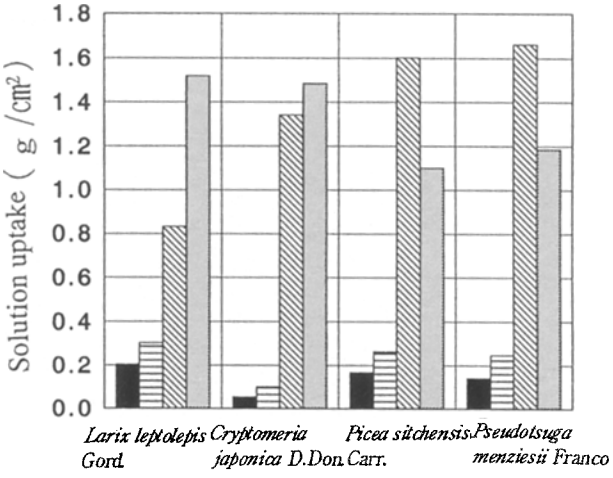

Fig. 3. Maximum amount of solution uptake of each species after the combined treatment of precompression and hot-water extraction. *Solid bars*, wet and without compression; *horizontally hatched bars*, extraction for 5 days, without compression; *diagonally hatched bars*, wet and compression of 50%; *box-hatched bars*, extraction for 5 days and compression of 50%

samples treated with compression sets of 0%, 10%, 20%, and 50%. Figure 2 shows a marked increase in uptake after precompression and extraction treatment. For example, with extraction treatment for 10 days and precompression of 50%, the maximum value of the solution uptake reached 1.8 g/cm^2, which is about 36 times the value of the control samples. Moreover, the maximum amount is about 5.3 times the maximum value of the uptake of samples that were only preheated in water for 10 days.

Based on the above facts, it is clear that penetration was markedly improved by the combined treatment in *Larix leptolepis* Gordon, a refractory wood. However, from the results obtained for *Larix leptolepis* Gordon only, we did not know whether this means of improving penetration was sufficient. Therefore, the mechanism of accelerated penetration could not yet be determined. The study then involved testing the species further.

Solution uptake for treated samples of four wood species

Figure 3 shows the values of the maximum solution uptake for the treated samples of each species tested. By comparing the species it was demonstrated that the amount of penetration markedly increased after precompression treatment. In the case of *Larix leptolepis* Gordon the solution uptake of the extracted sample with 50% compression was usually about seven to eight times greater than that of the control. For *Crytomeria japonica* D. Don, these values rose to about 1.3–1.4 g/cm^2, about 13–14 times that obtained in the control (0.1 g/cm^2). The amount increased when wood was extracted in hot water. When the increases in the solution uptake of each species under the four conditions indicated in Fig. 3 were compared, *Larix leptolepis* Gordon showed a marked increase in uptake in the extracted samples without compression. The values of the extracted samples were about seven to eight times larger than those of the control samples.

The results are obviously different in *Larix leptolepis* Gordon and *Crytomeria japonica* D. Don. Moreover, when *Picer sitchensis* Carr. was tested, the maximum values were larger in the extracted sample than in the untreated sample, and the precompressed samples without extraction treatment had the highest values under the four tested conditions. The values for samples compressed after extraction were smaller than those of the compressed samples without preextraction. The same result was seen after treatment of *Pseudotsuga menziesii* Franco. Because the latter two species had an evident crack along the tangential plane and the difference in the recoverable ratio under both conditions decreased about 2% in *Picer sitchensis* Carr about 5% in *Pseudotsuga menziesii* Franco when the large deformation of 50% was applied, it is important to fix the extent of solution uptake. From the results obtained, it can be noted that each species exhibited a characteristic behavior. The maximum values for solution uptake with 50% compression and without extraction in hot water of each species were almost identical.

Larix leptolepis Gordon and *Pseudotsuga menziesii* Franco, which are known to be refractory woods, are difficult to inject with solution via the Bethell method. However, it is becoming possible to impregnate those species to the same extent as sugi woods using the precompression and extraction method.

The difference among the wood species with respect to solution uptake after treatment, as mentioned above, correlates with whether the accumulation of extractive material on the aspirated pits can be destroyed under the large compressive deformation in the transverse direction. In this manner, extractives of *Larix leptolepis* Gordon consequently accumulated in the pit positions, preventing effective passage. In contrast, sugi wood consists of a thin film of extractives on pit aspiration, making it breakable under large compressive deformation in the transverse direction.

The factors for permeable obstruction in *Larix leptolepis* Gordon cannot be determined by experiments with solution

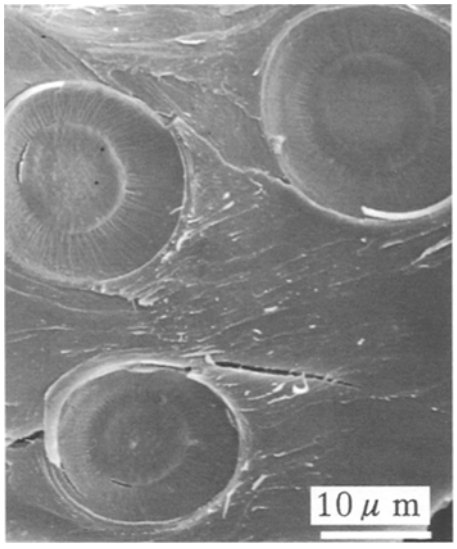

Fig. 4. Scanning electron microscopy (SEM) scan of pit aspiration of a wood control sample

uptake alone. In particular, it is necessary to determine the extended position of the extractive materials, including their type and properties, as well as the wettability of the surface on the cell cavity. For these reasons, changes in pit structures due to the applied treatments should be investigated. In this study we observed the pit membranes by SEM and the wettability of wood powders by the capillary attraction method. The results are discussed in the next section.

Observation of pit membrane by SEM

Figure 4 shows the results of the SEM observations of the pit aspiration of wood control samples, which were prepared without extraction treatment and compression in the transverse direction. In addition, Fig. 5 shows SEM micrographs of samples extracted for 3 days in hot water without compression treatment in the transverse direction. In Fig. 4 it is evident that microfibrils in the pit membranes were not clearly observed in all the extractive material, although they were seen in certain regions. They may be observed in the sample shown in Fig. 5, which had been extracted with boiling water.

Figure 6 shows the state of aspirated pit changes in samples previously compressed in the radial direction without extraction treatment. Figure 7 shows a sample that had been extracted for 3 days and compressed in the radial direction. In the absence of extraction treatment, deformation of pit aspiration was caused by compression and showed no evidence that would indicate a process of separation and breaking of the torus and margo. This condition is supposed to have more deformation ability, approaching plastic properties. However, it cannot be improved by the formation of effective passages to accelerate liquid movement. On the contrary, when compression was applied after the extraction treatment, the marked increase in penetration was due to the loss of extractives during hot-water

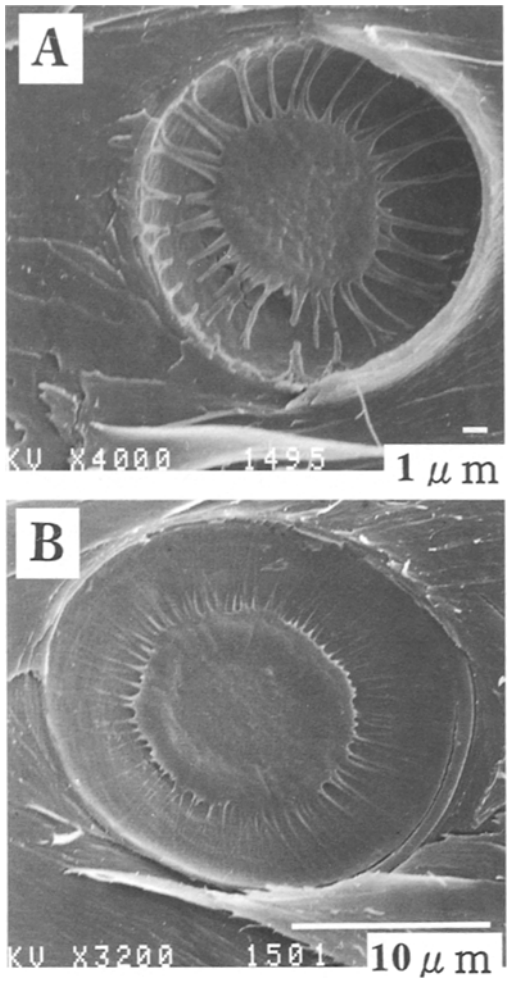

Fig. 5. Microfibrils of a pit membrane after extraction treatment with boiling water (*Larix leptolepis* Gordon). **A** Extreme sample. **B** Standard sample

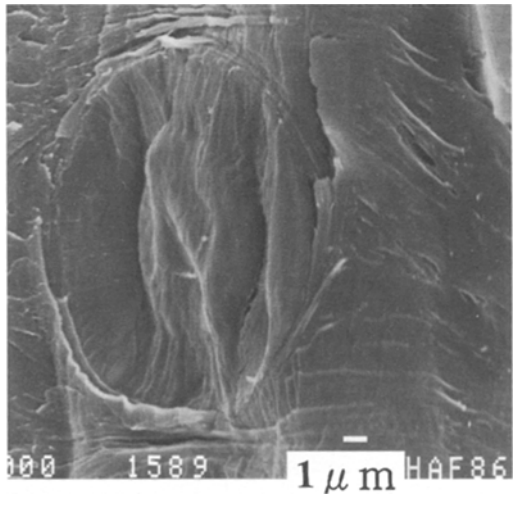

Fig. 6. Deformation of pit membrane caused by compression of *Larix leptolepis* Gordon in the radial direction. Compression treatment did not produce the fracture, although it did cause deformation

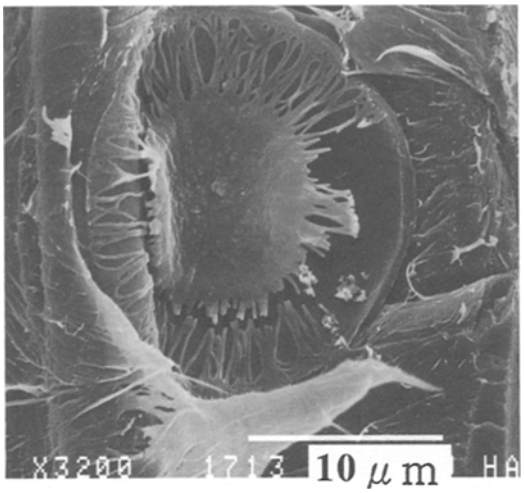

Fig. 7. Fracture of pit membrane caused by compression alone in the radial direction (*Larix leptolepis* Gordon) without extraction treatment

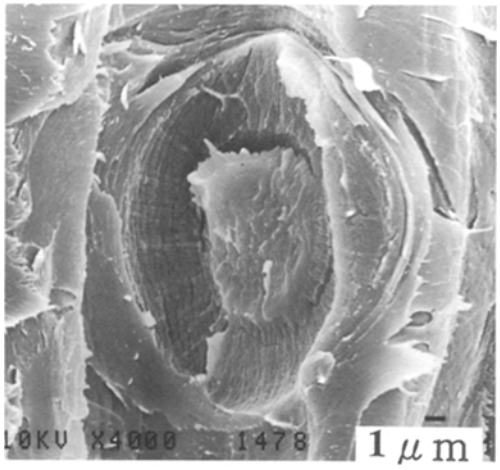

Fig. 8. Fracture of pit membrane caused by hot-water extraction and compression in the radial direction (*Pseudotsuga menziesii*)

treatment; and when greater compression is applied to such a sample, the pit aspiration is easily broken.

Figure 8 shows an SEM scan of a sample of *Pseudotsuga menziesii* Franco that had been compressed but not subjected to extraction. Similar to *Larix leptolepis* Gordon (although *Pseudotsuga menziesii* Franco has a considerable amount of accumulated extractives), it is estimated that the accumulated material is brittle and easily fractured when the pit membrane is subjected to compressive deformation. *Pseudotsuga menziesii* Franco showed marked penetration with precompression without the extraction treatment. These results differ among species in terms of the closed state of the pit membrane. For that reason, it is necessary to discuss the properties and qualities of the closed pits.

Table 1. Solution uptake of extracted wood powders by the capillary rise method

Sample	Extracted samples		Unextracted samples	
	Weight (g)	Height (cm)	Weight (g)	Height (cm)
1	0.663	12.6	0.236	2.8
2	0.534	8.5	0.273	4.2
3	0.630	10.5	0.276	3.6
4	0.427	6.8	0.176	2.0
5	–	11.4	–	3.2
6	–	11.5	–	3.8
Average value	0.564	10.2	0.24	3.2

Height of the capillary rise in the wood powder of karamatyu wood

It is difficult to measure directly the wettability of the surface of the cell lumen. Therefore, the tested samples were prepared in the form of wood powder, and the height of capillary penetration and the increase of sample weight were measured by the capillary attraction method. The results were used to estimate the wettability of the surface of the cell cavity.

Table 1 shows the height of capillary penetration and the increase in weight: The increases for the extracted wood powder were higher than the increases for the unextracted powder. In fact, the values for the extracted samples are approximately three times as high as those for the unextracted samples. These results suggest that solution uptake is affected by the poor wettability of the surface of the cell lumen.

References

1. Iida I, Takayama C, Miyagawa O, Imamura Y (1992) Liquid penetration of precompressed wood. I. Effect of compressive deformation and recovery upon liquid uptake. Mokuzai Gakkaishi 38: 233–240
2. Iida I, Ikeuchi A, Imamura Y (1995) Liquid penetration of precompressed wood. III. Effects of moisture content of specimens and ambient temperature while compression on liquid uptake of softwoods and hardwoods. Mokuzai Gakkaishi 41:811–819
3. Iida I, Mori S, Nakamura Y, Sakai H, Imamura Y (1996) Liquid penetration of precompressed wood. V. Effects of cyclic loading: type of impregnated chemicals and annual ring angles on the uptake of water or oily solvents. Mokuzai Gakkaishi 42:581–588
4. Watanabe U, Imamura Y, Iida I (1998) Liquid penetration of precompressed wood. VI. Anatomical characterization of pit fractures. J Wood Sci 44:158–162