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Mechanical properties of wood in an unstable state due to temperature changes, and analysis of the relevant mechanism IV: effect of chemical components on destabilization of wood

Abstract In order to investigate the effects of chemical components and matrix structure on the destabilization of quenched wood, we examined the physical and mechanical properties of steam-treated wood, hemicellulose-extracted wood, and delignified wood, which were treated at different levels. For steam-treated and hemicellulose-extracted wood, the relative relaxation modulus of the quenched sample was lower than that of the respective control sample. For delignified wood, the relative relaxation modulus fell with weight loss and reached a minimum value at a certain weight loss, and subsequently increased significantly. The hygroscopicity of all treated samples changed slightly by steaming, whereas increased with removing the component. Moreover, the average volumetric swelling per 1% MC at 100% relative humidity (RH) was less than at 75% RH and 93% RH for component-removed wood. It was clear that a void structure existed. As a result, the destabilization evaluated by the fluidity $(1 - E_t/E_0)$ of steam-treated wood was influenced by the amount of adsorbed water. For component-removed wood, destabilization increased temporarily at lower weight loss because of nonuniform cohesive structure. At high weight loss, destabilization will decreased because capillary-condensed water gathered in the voids and obstructed the motion of adsorbed water. However, the destabilization of all treated wood changed less than that of chemically modified wood.

Key words Quenching · Chemical components · Stress relaxation · Destabilization

Introduction

When wood undergoes rapid environment changes in temperature or relative humidity (RH), the wood must go through a transition from the initial stable state to a new stable state. However, wood has a complex structure, and, even if it equilibrates superficially to ambient temperature or relative humidity, the microstructure cannot reach the new stable state immediately and so passes through an unstable state during transition.[1–3] We called such a process "destabilization."

In previous reports, we identified the characteristics of destabilization using stress relaxation after the water-swollen wood was quenched from 30°–180°C to 20°C. The relative fluidity varied slightly at 30°–40°C and increased sharply at 40°–90°C. From 100° to 140°C, it held a constant value and above 140°C the relative fluidity increased again. It is considered that the redistribution of water molecules in the cell wall and the diversity of thermal expansion between components will cause destabilization of wood if the preheating temperature is under 100°C.[4] To clarify the function of these influencing factors, especially the redistribution of water molecules, we examined the destabilization of chemically modified wood, of which the adsorption sites were controlled. It is clear that destabilization of wood is closely linked to adsorbed water and the redistribution of water molecules significantly affects the destabilization of wood.[5]

However, if the preheating temperature is over 100°C, the components will thermally decompose[6] and subsequently recombine,[7] and the hygroscopicity changes.[8,9] The values of moisture content in the lower temperature and pressure were low while those under high temperature and pressure conditions were high. This tendency was thought to be caused by an increase in internal surface area and capillary condensed water.[9] Hemicellulose was estimated to leach markedly during steaming at 180°C, which was interpreted as the results of release of internal stress and formation of bonds of a hydrophobic nature.[6] Moreover, the stress relaxation curves of compressive deformation of sugi wood by steaming above 100°C showed a rapid decrease with increasing temperature, which were quite different from those below 100°C.[10]

Under the stable state, the relaxation modulus of delignified wood falls with the decrease in lignin content.[11] The

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effect of delignification on the mechano-sorptive creep of radial specimen was quantitatively remarkable, but was only slight in qualitative terms.[12] It is expected that these changes during the process of preheating affect the destabilization of quenched wood.

This study used steam-treated wood, hemicellulose-extracted wood, and delignified wood to investigate the effect of matrix components and structural change wood on destabilization. We examined the stress relaxation of unquenched and quenched wood to evaluate the destabilization of wood. Through the assessment of hygroscopicity, water cluster, volumetric swelling, and component contents, we discuss the relationship between chemical components, matrix structure, and the destabilization of quenched wood.

Materials and methods

Materials

The wood samples were cut from the heartwood of hinoki (*Chamaecyparis obtusa* Endl.). The sample size was 15 (T) $\times$ 4 (L) $\times$ 120 mm (R) for stress relaxation measurement and 20 (T) $\times$ 4 (L) $\times$ 40 mm (R) for the adsorption test.

Although extraction does not deeply affect the mechanical properties of wood, if the various contents of extraction remain in the cell wall due to different treatment methods,[6] it does not favor the analysis of chemical components. To know the function of chemical components clearly, all of the samples were extracted with acetone for 8 h and successively with ethanol for 8 h.

Steaming, hemicellulose removal, and delignification

After acetone extraction, the samples were treated with three methods as follows. Steaming was carried out in a high-pressure installation at 120°C for 20 min, 1, 3, and 8 h. Hemicellulose removal was also carried out in a high-pressure installation with a solution of 0.1, 0.2, 0.3, or 0.4% NaOH at 120°C for 4 h. Delignification was achieved according to Klauditz's method[13] and treatment times were 3, 6, 9, 12, 18, 24, and 48 h at 45°C. The selective and uniform removal of chemical components from wood blocks was verified in a previous study.[14] The residual content of hemicellulose was analyzed by the chlorite method,[15] and lignin content was determined by the sulfuric acid method. The component contents were calculated on the basis of the dry weight of wood before treatment.

For each reaction condition, two samples for stress relaxation and one sample for the adsorption test were treated together. Weight loss (WL) was calculated on the basis of the oven-dried weight before and after the reaction. It can be considered that the removed component is proportionate to the weight loss. Prior to subsequent quenching, the treated and untreated (reaction time: 0) wood was kept under water-saturated condition at 20°C for more than 1 month.

Quenching and stress relaxation measurement

One sample of each reaction condition was preheated at 80°C in a water bath for 3 h and was quenched to another water bath at 20°C. We called this sample "quenched wood." After 3 min, stress relaxation was measured. Another sample of each reaction condition was kept at 20°C in the water bath for a long time and stress relaxation was also measured. All samples were water saturated through this process.

The concentrated load of stress relaxation (Tensilon/UTM-4L, Toyo Measuring Instruments, Japan) was at the center of sample span (80 mm) and the initial deflection was 0.5–1 mm within the preparation limit. The values of load were 50 g–2 kg depending on the condition of treated sample. Measurement time was 2 h in the radial direction.

The relaxation modulus (E_t) and relative relaxation modulus (E_t/E_0) were calculated from the stress relaxation curves. The fluidity ($1 - E_t/E_0$), increase in fluidity between unquenched and quenched wood, and relative fluidity of quenched wood to untreated wood were used to evaluate destabilization. Increased fluidity was defined as follows:

$$\text{Increase in fluidity (\%)} = [(1-E_t/E_0)_q - (1-E_t/E_0)_c] \times 100(\%) \tag{1}$$

$$\text{Relative fluidity} = \frac{(1-E_t/E_0)_q}{(1-E_t/E_0)_{c,0}} \tag{2}$$

where $(1 - E_t/E_0)_q$ and $(1 - E_t/E_0)_c$ are the fluidities of quenched and unquenched wood, respectively, treated together and hence having the same weight loss. $(1 - E_t/E_0)_{c,0}$ is the fluidity of untreated wood kept at 20°C for a long time, and the values of E_t at 120 min were adopted.

Moisture adsorption test and volumetric swelling measurement

The sample for the moisture adsorption test was equilibrated at RHs of 22.8% (CH_3COOK), 42.6% (K_2CO_3.2aq), 75.4% (NaCl), and 93.0% (KNO_3) over saturated salt solutions and 65.0% (climatic chamber) and 100% (H_2O) at 20°C. The condition of 100% RH was obtained by keeping distilled water in a desiccator. The hygroscopicity of wood, such as the number of adsorption sites and water cluster size, was analyzed using the Hailwood-Horrobin adsorption equation.[16] Volumetric swelling per 1% moisture content (MC) was calculated by:

$$\text{Volumetric swelling per 1\% (MC)(\%)} = \left(\frac{V_m - V_0}{V_0}\right) \Big/ \text{EMC} \tag{3}$$

where V_m is the volume of treated wood at each MC, V_0 is the oven-drying volume of treated wood, and EMC is the equilibrium moisture content at each relative humidity.

Results and discussion

### Effect of quenching on stress relaxation of all treated woods

Figures 1 and 2 show the relative relaxation modulus of steam-treated wood and hemicellulose-extracted wood, respectively. The open symbols present the relative relaxation modulus of the control sample, which was kept at 20°C for a long time, and the solid symbols present that of the quenched sample. According to these figures, the relative relaxation modulus of the quenched sample was lower than that of the respective control sample, that is to say, the fluidity $(1 - E_t/E_0)$ of wood became significant in the unstable state, either steam-treated or hemicellulose-extracted wood; however, changes in fluidity did not clearly depend on weight loss.

Figure 3 shows the relative relaxation modulus of control and quenched wood that had been delignified to various extents. The amount of change in the relative relaxation modulus was much the same for the control sample and the quenched sample. This behavior is different from that of steam-treated wood and hemicellulose-extracted wood. The relative relaxation modulus fell with weight loss and reached a minimum at a certain weight loss, and subsequently increased significantly. This shows that the destabilization of delignified wood had various behaviors.

Hygroscopicity of steam-treated wood, hemicellulose-extracted wood, and delignified wood

The results of the moisture adsorption test are summarized in Table 1. At high relative humidity, changes in MC of steam-treated wood were slight. For the component-removed wood, the MC obviously increased. In particular, the MC of the delignified wood at 100% RH changed abnormally. This result is similar to a previous estimate.[11] It was thought that the capillary-condensed water is present in the cell wall. Changes in MC show that the field of adsorption was changed by treatment.

The number of adsorption sites (1/W), which can represent the field of adsorption, was calculated according to the Hailwood-Horrobin adsorption equation,[16] and the result
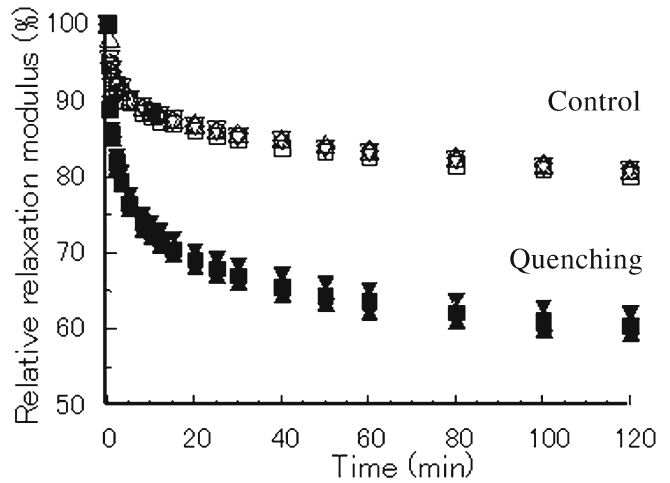

Fig. 1. Relative relaxation modulus of steam-treated wood. Weight losses (WL) are due to steaming. Control samples were kept at 20°C for a long time. Quenching samples were quenched to 20°C. *Open symbols*, controls; *filled symbols*, quenched samples; *circles*, WL 0%; *diamonds*, WL 0.74%; *triangles*, WL 1.06%, *squares*, WL 2.54%; *inverted triangles*, WL 4.96%
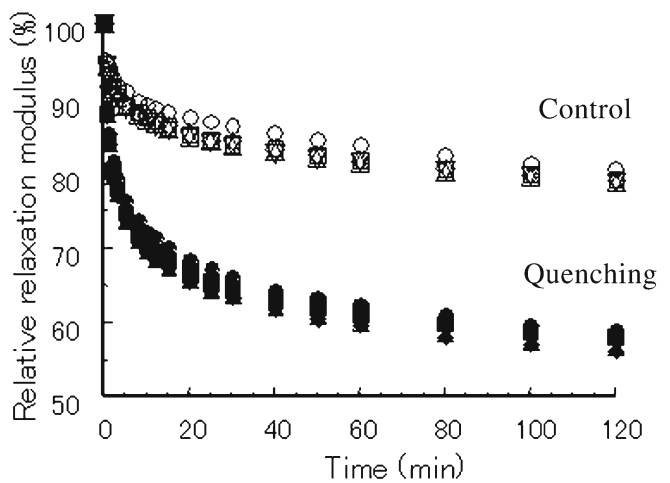
Fig. 2. Relative relaxation modulus of hemicellulose-extracted wood. Weight losses (WL) are due to hemicellulose extraction. *Open symbols*, controls; *filled symbols*, quenched samples; *circles*, WL 0%; *diamonds*, WL 2.55%; *triangles*, WL 4.43%, *squares*, WL 4.54%; *inverted triangles*, WL 9.89%


Fig. 3. Relative relaxation modulus of delignified wood. Weight losses (WL) are due to delignification. *Open symbols*, controls; *filled or bold symbols*, quenched samples; *circles*, WL 0%; *triangles*, WL 3.47%; *hashes*, WL 10.3%; *diamonds*, WL 12.9%, *squares*, WL 17.1%; *plus signs*, WL 21.3%; *inverted triangles*, WL 9.89%; *crosses*, 28.1%
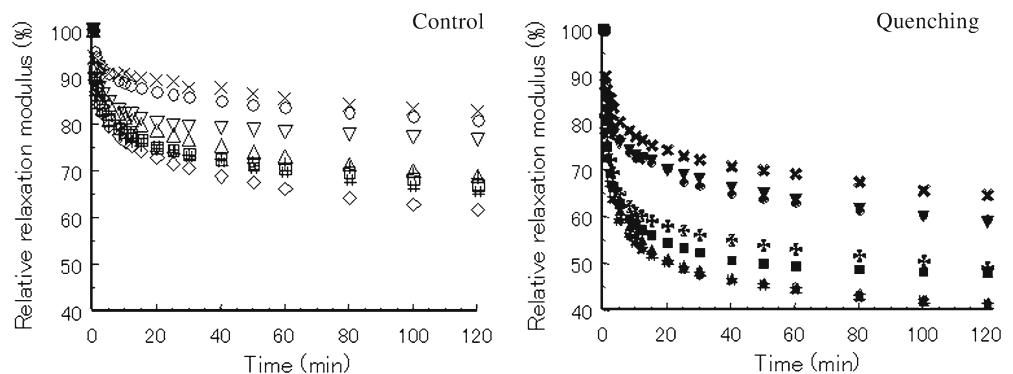
Table 1. Equilibrium moisture content of steam-treated wood, hemicellulose-extracted wood, and delignified wood at different relative humidities
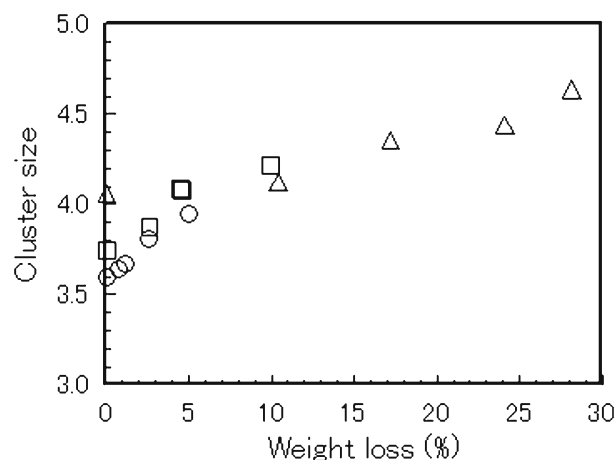
Weight loss (%)	Relative humidity at 20°C					
	22.8% (CH$_3$COOK)	42.6% (K$_2$CO$_3$·2aq)	65.0%[a]	75.4% (NaCl)	93.0% (KNO$_3$)	100% (H$_2$O)
Steam-treated wood						
0	4.44	7.43	11.0	13.8	22.2	26.7
0.74	4.64	7.68	11.2	14.1	22.8	28.3
1.06	4.60	7.59	11.3	14.1	23.0	28.7
2.54	4.52	7.46	10.8	13.6	22.4	28.7
4.96	4.35	7.09	10.4	13.2	22.0	28.2
Hemicellulose-extracted wood						
0	4.34	7.13	10.3	13.2	21.4	27.8
2.55	4.31	7.17	10.5	13.5	22.3	30.5
4.43	4.12	6.95	10.2	13.3	22.5	31.1
4.54	4.22	6.99	10.3	13.3	22.5	31.6
9.89	4.15	6.86	10.3	13.4	23.0	32.7
Delignified wood						
0	4.18	7.05	10.0	12.4	21.2	27.3
10.3	5.28	8.40	12.3	15.7	26.6	40.1
17.1	5.34	8.40	12.5	16.0	27.4	43.5
24.1	5.48	8.62	12.4	15.8	27.7	45.1
28.1	5.34	8.38	12.0	15.3	27.4	44.7

[a] Constant temperature and humidity room (20°C, 65% RH)
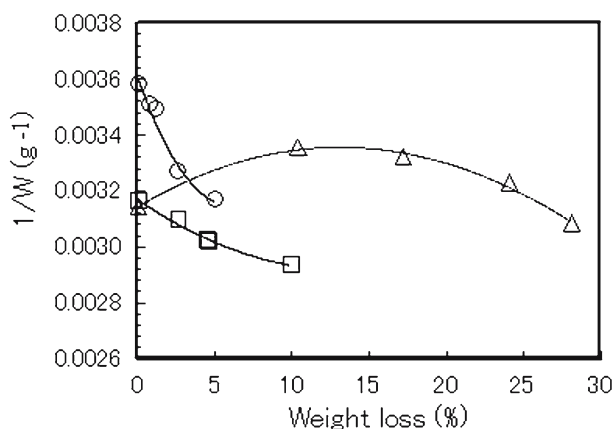
Fig. 4. Changes of adsorption site with weight loss for all treated wood. *Circles*, steam-treated wood; *squares*, hemicellulose-extracted wood; *triangles*; delignified wood

Fig. 5. Relationship between the water cluster size and weight loss for all treated wood. *Circles*, steam-treated wood; *squares*, hemicellulose-extracted wood; *triangles*; delignified wood

is shown in Fig. 4. For steam-treated and hemicellulose-extracted wood, the number of adsorption sites fell, accompanied by weight loss. Generally the amount of adsorbed water falls as adsorption sites decrease, but for steam-treated wood and hemicellulose-extracted wood with fewer adsorption sites, moisture contents at high relative humidity were more than that of untreated wood (Table 1). For delignified wood, the number of adsorption sites was more than untreated wood and the moisture content increased. These results suggest that the water state, especially dissolved water, is different in all treated woods. The water cluster size, which is closely related with dissolved water, was calculated according to water cluster theory,[17] and Fig. 5 shows the relationship between water cluster size and weight loss. The cluster size increased with increasing weight loss. With high-temperature water vapor, irregularly shaped voids remained after hemicellulose was hydrolyzed.[9] This means that the void formed easily by removing the component and there was more void in the matrix structure with weight loss; the dissolved water easily pooled at the adsorption site after hydration water was adsorbed.

Relationship between adsorbed water and destabilization of all treated wood

Figure 6 shows the relationship between increase in fluidity and equilibrium moisture content at 93% RH of treated wood. The solid symbols are the untreated sample of each treated wood group. For steam-treated wood, destabilization increased temporarily and then fell. Increase in fluidity has a positive relationship with adsorbed water in a narrow range of relative humidity, similar to acetylated wood and formaldehyde-treated wood.[5] From the results of the fiber

saturation point (FSP) in Table 1, the average value of FSP for untreated wood was 27.3%. The average FSP of steam-treated wood was about 28.5%, showing that the structure of steam-treated wood changed slightly. The destabilization due to temperature change can be explained by the redistribution of water molecules and the degree of destabilization depended on the amount of adsorbed water.[5] For hemicellulose-extracted wood and delignified wood, destabilization increased temporarily and then fell, but destabilization has no clear relationship with adsorbed water.

Figure 7 summarizes the changes in destabilization for acetylated wood, formaldehyde-treated wood, steam-treated wood, hemicellulose-extracted wood, and delignified wood at each moisture content level. The adsorbed water clearly influenced the destabilization of acetylated wood and formaldehyde-treated wood, whereas the destabilization of component-removed wood changed slightly and did not greatly depend on moisture content. Moreover, according to the data in Table 1, the FSPs of component-removed woods were higher than that of untreated wood. This suggests that an abundance of capillary-condensed water exists at high relative humidity to affect the redistribution of water molecules. Consequently, it is inferred that the cohesive structure of the matrix changed and influenced the destabilization.

Recent studies using differential scanning calorimetry (DSC) have shown that bound water on fibers and wood

can be divided into nonfreezing bound water and freezing bound water.[18–21] Nonfreezing water refers to water that is very tightly bound to the polar groups inside the cell wall. Freezing bound water is also associated with the cell wall but the interaction is not as strong. From these results, it is inferred that the effect of freezing bound water due to quenching is major.

Relationship between change of chemical components and destabilization of wood

According to the available literature for hinoki, the average volumetric swelling per 1% MC is equal to the volume of adsorbed water of 1% MC in a wide range of MC.[22] It is thought that the values of average volumetric swelling per 1% MC reflect not only changes in the cohesive structure of the matrix but also the state of the water in the cell wall. Figure 8 shows the relationship between average volumetric swelling per 1% MC and weight loss at 75%, 93%, and 100% RH. The average volumetric swelling per 1% MC of steam-treated wood hardly changed at each relative humidity level. For hemicellulose-extracted wood, the average volumetric swelling per 1% MC hardly changed with low weight loss, and subsequently increased. The average volumetric swelling per 1% MC of delignified wood increased clearly with weight loss. This confirmed that the void struc-
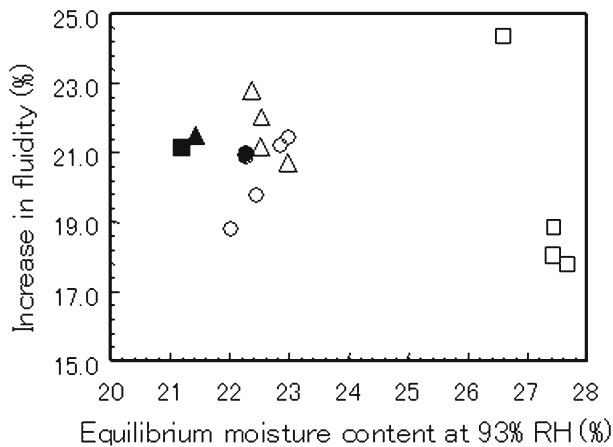

Fig. 6. Relationship between increase in fluidity and equilibrium moisture content (EMC) at 93% relative humidity (RH) for all treated wood. *Filled symbols*; controls; *open symbols*, treated samples; *circles*, steam-treated wood; *triangles*, hemicellulose-extracted wood; *squares*, delignified wood
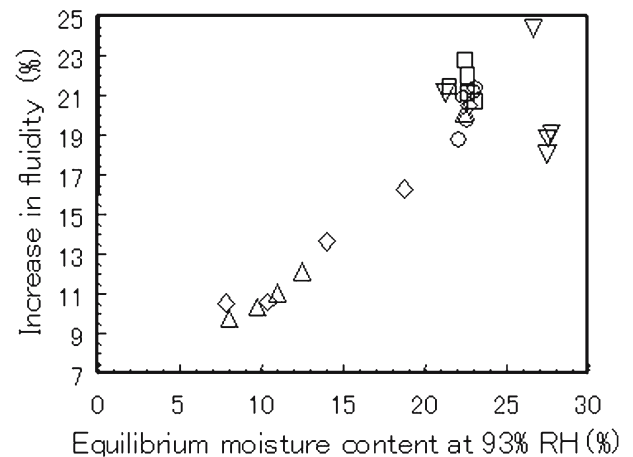
Fig. 7. Relationship between increase in fluidity and EMC at 93% RH for chemically treated wood and chemically modified wood. Note that the data for chemically modified wood is from Wang et al.[5] *Diamonds*, formaldehyde-treated wood; *triangles*, acetylated wood; *circles*, steam-treated wood; *squares*, hemicellulose-extracted wood; *inverted triangles*, delignified wood

Fig. 8. Relationship between the average volumetric swelling per 1% MC and weight loss at each high relative humidity for steam-treated wood, hemicellulose-extracted wood, and delignified wood. *Open circles*, 75.4% RH; *triangles*, 93% RH; *filled circles*, 100% RH
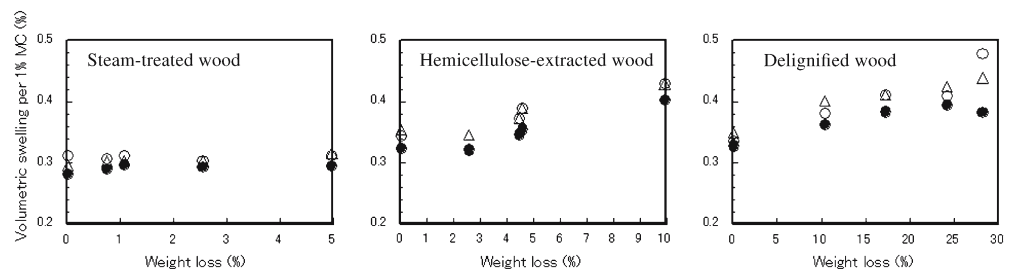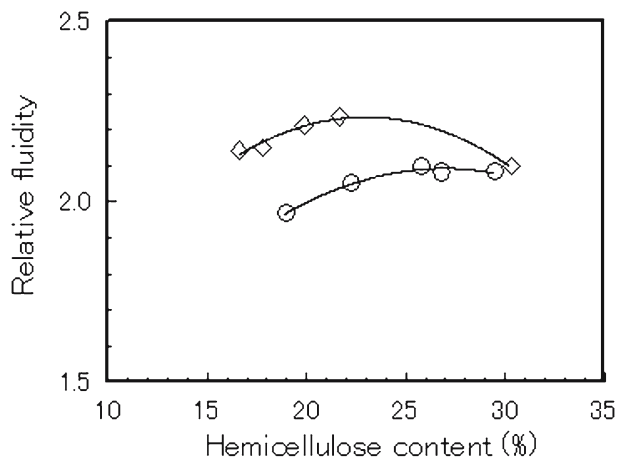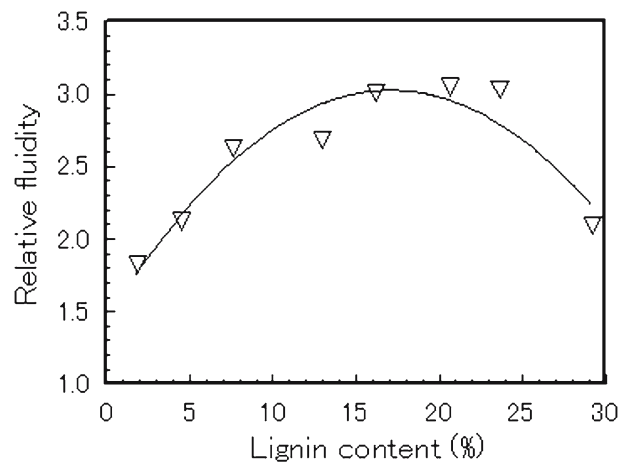
Fig. 9. Relationship between relative fluidity and hemicellulose content for steam-treated (*circles*) and hemicellulose-extracted (*diamonds*) wood

Fig. 10. Relationship between relative fluidity and lignin content for delignified wood

ture was expanded and the cohesive structure became weak for hemicellulose-extracted and delignified wood. The differences in void and cohesive structure may cause the various degrees of destabilization of wood.

The contents of hemicellulose and lignin, which are closely linked with the matrix structure, were analyzed. Lignin content shows almost no change by steaming at 180°C for 1–4h[6] and changes only slightly by NaOH solution treatment.[23] The loss of hemicellulose was controlled during the first 60% of delignification.[24] Therefore, we present data only on hemicellulose loss due to steaming and hemicellulose extraction and lignin loss due to delignification. The relationship between the residual content of chemical components and destabilization of wood is shown in Figs. 9 and 10.

Figure 9 shows the relationship between hemicellulose content and relative fluidity for steam-treated and hemicellulose-extracted wood. Although hemicellulose clearly decreased, destabilization of wood was somewhat increased temporarily and then decreased; however, the destabilization of steam-treated wood was less than that of hemicellulose-extracted wood. During the steaming process, hemicellulose was depolymerized, a portion of which remained in the cell wall because the components are difficult to leach from the surface of softwood.[7] The residual depolymerized hemicellulose, which has high hygroscopicity,[8] increased the moisture content. Destabilization increased as adsorbed water increased. When more depolymerized hemicellulose remained, hemicelluloses recombined[7] and the number of adsorption sites fell sharply, resulting in reduced destabilization. Moreover, according to the results of swelling, it can be considered that the voids in the matrix of steam-treated wood hardly affect the destabilization of wood until hemicellulose content reached 20%.

For hemicellulose-extracted wood, as hemicellulose was directly extracted from around the matrix structure by NaOH solution combined with steaming, the voids in the matrix increased and capillary-condensed water appeared, which will stop the motion of adsorbed water. The destabilization became decreased if the hemicellulose content was below 20%.

Figure 10 shows the relationship between lignin content and relative fluidity for delignified wood. Destabilization increased sharply when a small amount of lignin was removed and decreased with reduced lignin content.

When a small amount of lignin was removed, the three-dimensional network structure of lignin was partly disorganized and the interaction between hemicellulose and lignin was changed, resulting in internal strain such as with hemicellulose removal. It is considered that destabilization due to quenching increased under the function of internal strain. If significant lignin was removed, the cohesive structure was considerably disorganized. The molecular chains could move freely; more and more voids appeared between hemicellulose and lignin, and capillary-condensed water pooled in the voids. It is thought that this water state obstructs the motion of molecules or adsorbed water during quenching. As the result, destabilization will decrease with weight loss.

From these results, the destabilization of water-swollen wood, quenched from 100°–180°C to 20°C,[4] can be elucidated. Although the molecules of amorphous cellulose are relatively mobile because of disruptions to hydrogen bonding by adsorbed water,[25] the changes of hemicellulose and lignin structures were strong and various, and we focused the factor of destabilization on hemicellulose and lignin. Before quenching, hemicellulose and lignin may decompose slightly with temperature. First, hemicellulose decomposed and the depolymerized hemicelluloses recombined, reducing hygroscopicity. The degree of water molecule redistribution fell as adsorbed water decreased, and destabilization hardly changed. At a preheating temperature of over 140°C, lignin began to decompose slightly, voids formed, and the adsorbed water increased causing destabilization to increase once more.

Conclusions

For steam-treated wood, depolymerized hemicellulose could recombine and the cohesive structure of the matrix changed slightly so that destabilization of wood was influenced only by the amount of adsorbed water. For hemicellulose-extracted wood and delignified wood, the cohesive structure of the matrix became nonuniform with less weight loss, having no connection with hemicellulose or lignin removal. Consequently, the motion of adsorbed water and molecular chains showed different behavior with quenching, and, as a result, destabilization increased temporarily. With high weight loss, especially for delignified wood, the cohesive structure was disorganized and the capillary-condensed water pooled in the voids, obstructing the motion of molecules or adsorbed water during quenching. As a result, destabilization decreased based on the reaction conditions.

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