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Three mechanisms affecting the mechanical properties of spruce wood dried at high temperatures

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Abstract Wood drying experiments are conducted in which the temperature and the drying rate are controlled independently. In relationship to drying processes, at least three mechanisms are believed to contribute to the properties of dried wood. However, only two of these are found to affect the properties of macroscopic specimens, the third mechanism being observable in microtomed earlywood sections, and possibly in specimens loaded in the radial direction. Degradation of structural components and irreversible hydrogen bonding (hornification) are found to contribute to both the hygroscopicity and the mechanical properties of macroscopic wood specimens. Mass loss from thermal degradation occurs predominantly in slow high-temperature drying processes. Irreversible hydrogen bonding takes place in high-temperature drying, in particular with high ultimate dryness. Regarding the effect on strength and stiffness, mass loss and hornification appear to compete. The third identified mechanism, microscopic cell wall damage caused by incompatible drying shrinkage of cell wall elements, does not seem to affect the mechanical properties of macroscopic wood specimens. Consequently, slow high-temperature drying processes do not provide much benefit regarding the mechanical behavior of dried wood. The reasons for this are discussed.

Key words Drying damage · Hornification · Mass loss · Mechanical properties · Wood

Introduction

Wood is a complex polymeric material used for many purposes such as construction, joinery, furniture, and conversion to pulp and paper. Most applications of wood require that the material is dried from the green state to some extent between the fiber saturation point (FSP) and the dry

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state. During drying, however, some defects may appear, thus reducing the quality of the final wood product. Typical drying defects are checks and cracks, distortions or warp, and discolorations.

The exposure of wood to elevated temperatures during drying may cause thermal degradation of its structure, often accompanied by loss of mass. The degree of thermal degradation depends on tree species, as well as on process parameters such as duration of treatment, temperature, and relative humidity. Drying may also induce irreversible hydrogen bonding between carbohydrate elements. Such a phenomenon is often denoted as hornification. 4-6

Significant microscopic damage has been reported to occur within the wood cell walls along with drying. 7-9 Such damage is believed to result from anisotropic shrinkage of the cell wall layers, inducing internal stresses large enough to damage the cell walls. 7,10,11 In fact, it has been shown that impregnation of wood with a chemical that reduces drying shrinkage may more than double the tensile strength of thin earlywood specimens. 9 Impregnation with chemicals, however, may not be the only way to reduce cell wall damage. The phenomenon of stress relaxation might also be utilized.

Wood is mostly composed of amorphous polymers, and thus it shows time-dependent mechanical behavior. Along with time, stresses within wood elements become reduced by stress relaxation. The molecular mobility of the amorphous polymers is dependent on the moisture content and on the temperature. 12-14 From the point of view of molecular reorganization, an increase in moisture content or temperature is analogous to an increase in available time. Therefore, relaxation of internal drying stresses can be favored by slow drying processes at high temperatures.

From the viewpoint of thermal degradation of wood, the combination of high temperature and moisture content may not be desirable.¹⁻³ The thermal degradation, often manifested as loss of mass, significantly impairs the mechanical properties of wood.^{2,15} The reduction in mechanical properties is in particular evident when specimens are compared at similar moisture content, instead of at similar ambient conditions,¹⁵ because mass loss often decreases hygroscopic-

ity, thus reducing the moisture content of wood at constant ambient conditions. ^{2,16} The decrease in hygroscopicity is mainly caused by the reduction of bonding sites available for water sorption that accompanies the degradation of hemicelluloses. ¹⁷

In addition to mass loss, the hygroscopicity of wood is reduced by irreversible hydrogen bonding within the cell wall, i.e., hornification. The degree of hornification appears to depend on the severity of the drying process, that is, on the drying temperature, as well as on the ultimate dryness. Hornification not only decreases hygroscopicity but may also increase stiffness and strength at constant moisture content.

The objective of this study was to investigate the effect of drying conditions on the mechanical properties of dried wood. Spruce wood specimens were subjected to drying processes in which the temperature and the drying rate were controlled independently. Mechanical properties of the specimens were then analyzed in relationship to the process parameters. Finally, mechanisms affecting the mechanical behavior of dried wood are discussed.

Experimental methods

Material and drying experiments

The wood material used was Norway spruce (*Picea abies*) felled in Joensuu (Finland). Wood specimens with dimensions $350 \times 20 \times 20$ mm (longitudinal, radial, tangential) were prepared. All specimens were clear of visible flaws. Six groups containing 8 to 12 specimens each were formed, and each group was subjected to a particular drying process. Drying experiments were conducted in a stainless steel pressure vessel designed by the authors, and equipped with a temperature gauge, a pressure gauge, and a valve for the removal of water vapor. The removed water vapor was condensed in a container placed on a weight scale, which allowed monitoring changes in the wood moisture content.

Before any drying experiment, a 30-mm end-piece was sawn from the specimens. The end-pieces were oven-dried at 103°C for 24 h to approximate the initial moisture content of the wood. Each group of specimens was then weighed and placed in the vessel. A measured amount of liquid water was added to create a saturated steam atmosphere. The temperature in the vessel was raised and the specimens were steamed for about 2 h, which may remove the effects of eventual variations in moisture content history. Once the temperature reached 128°C, the pressure valve was opened. Because the valve is in the upper part of the vessel, air exited first, hot air over 125°C being lighter than hot steam. After air removal, rapid water vapor removal followed until the moisture content of wood was about 45%. Thereafter, the drying process was started at a predetermined temperature and drying rate. The drying rate was controlled by operating the pressure valve, the change in the wood moisture content being given by Eq. 1:

$$\frac{\mathrm{d}M}{\mathrm{d}t} = kM\tag{1}$$

where M (g/g) is the wood moisture content, t (s) is the drying time, and k (s⁻¹) is the drying rate constant. After rearranging and integrating, Eq. 1 is transformed into

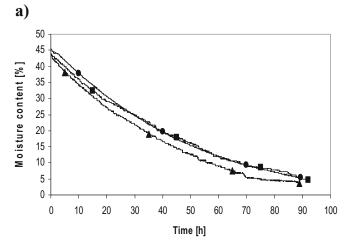
$$ln M = kt + C$$
(2)

where C is an integration constant. For t = 0, $C = \ln M_0$, where M_0 is the moisture content at the beginning of the drying process, i.e., approximately 45%. After substituting and rearranging, Eq. 2 can be rewritten as

$$M_t = M_0 e^{kt} \tag{3}$$

where M_t is the moisture content of wood at any time of the drying process, as shown in Fig. 1.

In addition, five groups of specimens were dried in a conventional laboratory oven at different temperatures (Fig. 2); the corresponding drying rates were governed by the atmospheric conditions in the oven. This type of conventional drying was chosen as a reference treatment.



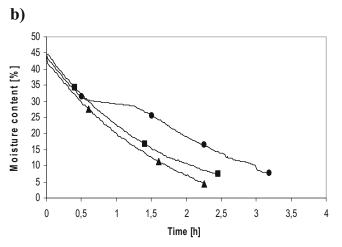


Fig. 1. Change in the wood moisture content for slow drying processes (a) and rapid drying processes (b). *Circles*, temperature 110°C; *squares*, temperature 120°C; *triangles*, temperature 130°C. Symbols are inserted to identify the curves

Table 1. Experimental parameters for each drying process

Test	Temperature (°C)	Drying rate $(-10^{-6} \mathrm{s}^{-1})^{\mathrm{b}}$	Final moisture content (%)°	Mass loss (%)	EMC (%)
A ^a	80	57.3	6.9 (1.9)	_	10.2 (0.6)
\mathbf{B}^{a}	100	93.7	5.8 (1.6)	0.2 (0.2)	9.6 (0.4)
C^{a}	110	115.4	5.3 (0.8)		8.9 (0.2)
\mathbf{D}^{a}	120	205.7	5.3 (2.0)	_	9.0 (0.4)
$\mathbf{E}^{\mathbf{a}}$	130	282.0	5.4 (1.9)	0.5 (0.5)	9.4 (0.5)
F	110	151.5	7.8 (2.7)	- ` ′	9.7 (0.7)
G	120	204.2	7.4 (2.3)	0.2 (0.5)	9.7 (0.6)
H	130	282.0	4.5 (1.5)	0.2 (0.7)	8.3 (0.4)
I	110	6.7	5.4 (1.0)	1.4 (0.9)	8.1 (0.3)
J	120	6.9	4.7 (0.9)	3.4 (0.8)	7.6 (0.4)
K	130	7.8	3.8 (0.3)	5.3 (0.4)	6.8 (0.2)

Standard deviation in parentheses

EMC, equilibrium moisture content

^cFinal moisture content of wood taking into account the mass loss

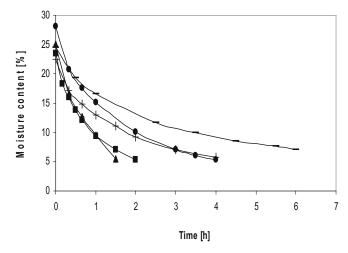


Fig. 2. Change in the wood moisture content for drying processes conducted in a conventional oven. *Lines*, temperature 80°C; *circses*, temperature 100°C; *circles*, temperature 110°C; *squares*, temperature 120°C; *triangles*, temperature 130°C. Symbols inserted on the curves correspond to experimental observations

Before oven-drying, each group of specimens was steamed in the pressure vessel following the procedure described above. However, after reaching a moisture content of 45%, the vessel was allowed to cool to about 100°C and the specimens were transferred to the oven. The experimental parameters for each drying process are shown in Table 1.

Mechanical properties

All dried specimens were placed in a climate-controlled room at 19°C and 65% relative humidity to attain equilibrium moisture content (EMC). Mechanical properties were then determined by conducting three-point bending tests, with a span length of 300 mm, in an electromechanical testing device (Matertest 100 kN). The displacement rate of the cross-head was 0.2 mm per second, and the force was applied to the face of the specimen nearest the pith. The

modulus of rupture (MOR) and modulus of elasticity (MOE) were determined according to the following equations:

$$MOR = \frac{3P_{\text{max}}L}{2bh^2} \tag{4}$$

$$MOE = \frac{PL^3}{4bh^3\delta} \tag{5}$$

where $P_{\rm max}$ (N) is the load at failure, P (N) and δ (mm) are any load and its corresponding displacement below their proportionality limit, L is the span length (mm), and h (mm) and b (mm) are the height and the width of the specimen, respectively.

The strain at failure, or ductility, was approximated according to Eq. 6:

$$\varepsilon_{\text{max}} = \frac{6\delta_{\text{max}}h}{L^2} \tag{6}$$

where $\varepsilon_{\rm max}$ (mm/mm) and $\delta_{\rm max}$ (mm) are the maximum strain and displacement at failure, respectively. The failure strain was then divided into its elastic and inelastic components. The elastic failure strain ($\varepsilon_{\rm e}$) was computed as

$$\varepsilon_{\rm e} = \frac{MOR}{MOE} \tag{7}$$

and the inelastic failure strain was computed by subtracting the elastic failure strain from the total failure strain. The mechanical properties of the dried wood are shown in Table 2.

Results

Strength and stiffness of any porous material depend on its porosity. In the particular material of this study, the density of the specimens ranged from 325 to 460 kg/m³, and thus the mechanical properties varied accordingly (Fig. 3). As our main interest lies in studying the effect of drying conditions on the mechanical properties of wood, from here on we discuss the specific strength and the specific stiffness,

^a Drying processes conducted in a conventional laboratory oven

^bDrying rates computed according to Eq. 3 for the total drying time

Table 2. Mean values for the mechanical properties and the density of dried wood

Test	MOR (MPa)	MOE (GPa)	Elastic ductility (10 ⁻³ mm/mm)	Inelastic ductility (10 ⁻³ mm/mm)	Density (kg/m³) ^b
A^a	79.3 (7.2)	14.4 (2.3)	5.6 (0.5)	7.9 (1.7)	398.7 (35.5)
\mathbf{B}^{a}	77.4 (10.4)	13.2 (1.8)	5.9 (0.6)	6.6 (1.6)	375.9 (34.7)
C^{a}	78.6 (6.6)	13.2 (1.7)	6.0 (0.4)	7.4 (2.0)	378.5 (31.8)
\mathbf{D}^{a}	85.5 (7.0)	15.4 (1.6)	5.6 (0.3)	8.3 (0.6)	394.3 (16.8)
E^{a}	86.9 (9.5)	14.7 (2.2)	6.0 (0.5)	7.9 (1.7)	395.2 (29.4)
F	75.9 (8.3)	13.2 (1.4)	5.8 (0.3)	7.5 (3.0)	364.9 (29.2)
G	86.3 (10.5)	15.8 (2.2)	5.5 (0.3)	7.5 (1.9)	400.5 (36.2)
H	83.0 (9.7)	15.1 (2.6)	5.6 (0.4)	5.9 (1.5)	399.0 (48.0)
I	92.1 (8.4)	17.1 (1.8)	5.4 (0.4)	6.5 (1.9)	408.2 (25.2)
J	73.8 (6.2)	13.2 (2.1)	5.7 (0.6)	5.2 (2.2)	366.6 (30.0)
K	83.7 (5.5)	15.8 (1.3)	5.3 (0.4)	5.7 (1.1)	386.0 (15.2)

Standard deviation in parentheses

MOR, modulus of rupture; MOE, modulus of elasticity

^bDensity is the ratio of dry mass to volume at constant ambient conditions of 19°C temperature and 65% relative humidity

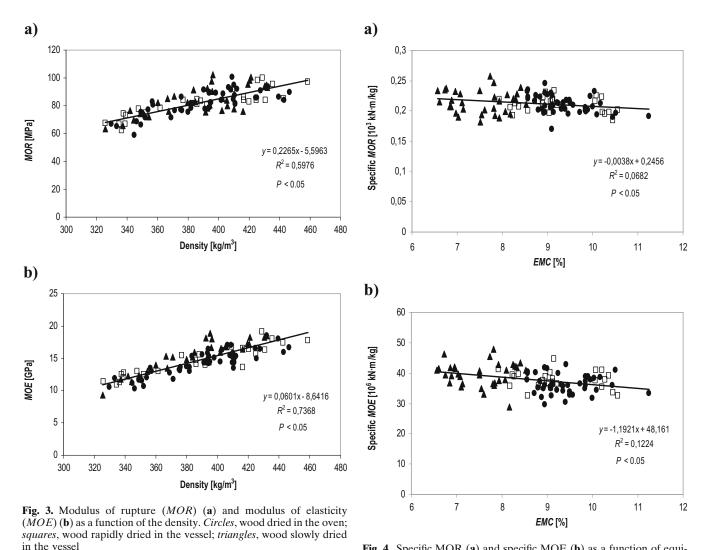


Fig. 4. Specific MOR (a) and specific MOE (b) as a function of equilibrium moisture content (*EMC*). Symbols as in Fig. 3

i.e., the MOR and the MOE divided by the density, respectively.

Let us then start analyzing the results by plotting the effect of EMC on the specific strength and stiffness (Fig. 4). It is found that both the specific MOR and MOE decrease

with increasing the EMC. Wood specimens that were slowly dried attained the lowest EMC (see Table 1), and, correspondingly, their specific strength and stiffness appear to be the highest. Nonetheless, even if the moisture content

^aDrying processes conducted in a conventional laboratory oven

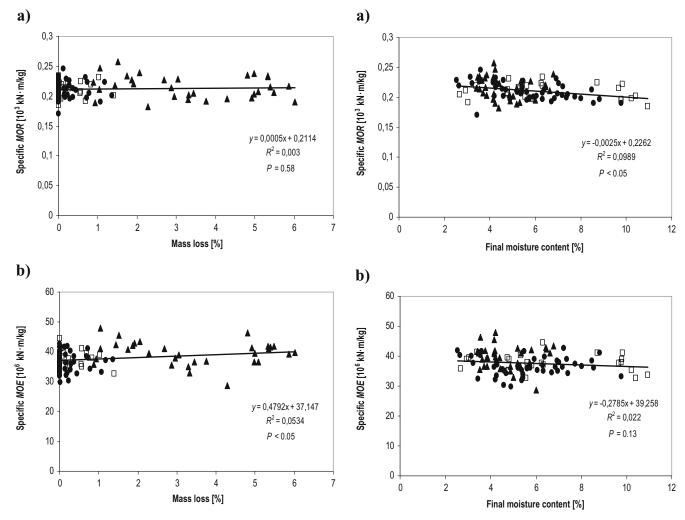


Fig. 5. Specific MOR (a) and specific MOE (b) as a function of mass loss. Symbols as in Fig. 3 $\,$

Fig. 6. Specific MOR (a) and specific MOE (b) as a function of the final moisture content reached during drying. Symbols as in Fig. 3

explains some of the variation in the specific MOR and MOE, a significant variation remains.

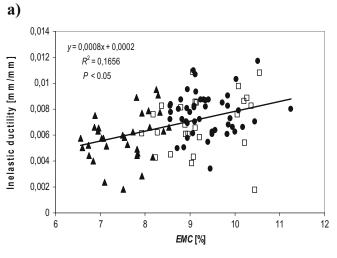
The specific strength and stiffness are plotted as a function of mass loss in Fig. 5. Surprisingly, the specific MOR remains unaltered even for mass losses up to 6%, whereas the specific MOE seems to increase, even if slightly, with increasing mass loss. This behavior might be partly caused by the correlation between the mass loss and the EMC, in particular for those specimens that were slowly dried (see Table 1). However, other mechanisms contributing to the strength and stiffness of dried wood may exist, beyond the reduced hygroscopicity. This possibility is further supported by Fig. 6, in which the specific MOR, and to a lesser extent the specific MOE, appear to decrease along with increasing the final moisture content reached during drying.

Inelastic ductility increases with the EMC and with the final moisture content reached during drying, but decreases with increasing mass loss (Figs. 7a, 8a, 9a). On the other hand, the drying process parameters seem to have little effect on elastic ductility (Figs. 7b, 8b, 9b). Therefore, the variation in ductility of wood resulting from different drying conditions is dominated by its inelastic component.

Discussion

In relationship to drying processes, at least three mechanisms contribute to the mechanical properties of dried wood. First, drying processes inducing significant mass loss result in wood products with reduced strength, stiffness, and inelastic ductility.^{2,15} The mass loss also reduces the hygroscopicity of wood.^{2,16} Second, irreversible hydrogen bonding between carbohydrate elements, i.e., hornification, reduces at least the hygroscopicity of wood.⁴⁻⁶ Hornification may also increase stiffness and strength at constant moisture content.⁶ Third, it appears that irrecoverable microscopic damage occurs within the wood cell walls during drying, thus reducing the load-bearing capability of the material.⁷⁻⁹

Let us now analyze what the present results indicate regarding the eventual role of any of the three mechanisms on the mechanical properties of dried wood. As already mentioned, both mass loss and hornification contribute to a decrement in hygroscopicity. The stiffness and strength of wood increase with decreasing moisture content, below the fiber saturation point.¹⁸ Indeed, it is found in Fig. 4 that



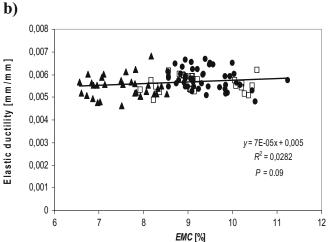
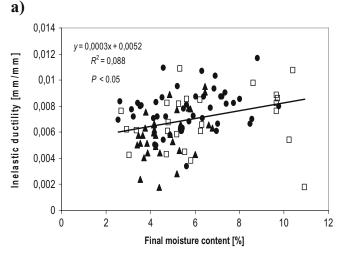


Fig. 7. Inelastic ductility (**a**) and elastic ductility (**b**) as a function of EMC. Symbols as in Fig. 3

specific strength and stiffness appear to decrease with an increase in EMC. However, the moisture content explains only partially the variation in mechanical properties.

One might wonder to which degree the EMC is here affected by the mass loss and to which degree by hornification. Figure 10 shows EMC as a function of the final moisture content on one hand and as a function of mass loss on the other hand. The EMC decreases with increasing final dryness (Fig. 10a), which is in agreement with previous observations reporting that the mechanism of hornification depends on the severity of the drying process.^{4,5} Further, the data clearly show two different groupings, slow drying processes forming a group of their own. Slow drying processes induced higher mass losses, and thus some of the effect on the EMC is obviously the result of mass loss (Fig. 10b). Nonetheless, additional hornification can be expected to occur in a drying process extended in time. The proposed mechanism for the occurrence of such an additional hornification is as follows.

Let us consider a water droplet located in a small pore within the wood cell wall. During the drying process, the droplet will exit the pore, and the surface tension of the



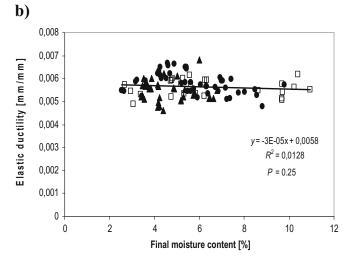


Fig. 8. Inelastic ductility (a) and elastic ductility (b) as a function of the final moisture content reached during drying. Symbols as in Fig. 3

diminishing water droplet will bring the pore walls closer to each other, attempting to close the cavity. The pore closure, however, deforms the cell wall structure, thus inducing elastic stresses within the cell wall material that counteract the pore closure. Wood polymers show time-dependent mechanical behavior, manifested either as creep or as stress relaxation. With time, the elastic stresses in the cell wall relax, and the cell wall material creeps. Thus, the pore closure is likely to be more complete in a slow drying process, in comparison with a rapid process, because it is aided by stress relaxation and creep. This is the reason why greater hydrogen bonding between wood elements can be expected in slow drying processes.

Reduced hygroscopicity is not the only effect of either of the two mechanisms, i.e., mass loss and hornification. Mass loss reduces the amount of load-bearing elements within the cell wall whereas hornification creates new connections between such elements. It has been previously shown that the strength and stiffness of wood are significantly reduced when the mass loss exceeds 2%–3%. ^{2,15} In

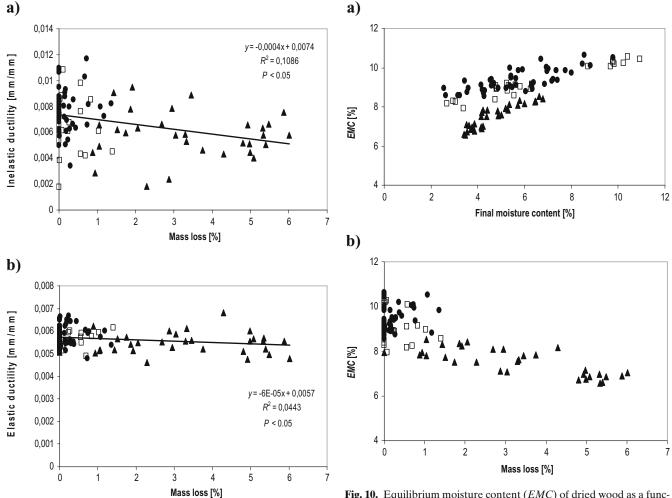


Fig. 9. Inelastic ductility (a) and elastic ductility (b) as a function of the mass loss. Symbols as in Fig. 3

Fig. 10. Equilibrium moisture content (*EMC*) of dried wood as a function of the final moisture content reached during drying (**a**) and the mass loss (**b**). Symbols as in Fig. 3

Fig. 5, however, the specific strength, and in particular the specific stiffness, increase slightly as a function of mass loss. Moreover, the specific strength and stiffness increase when the final dryness is increased (Fig. 6). Thus, the mechanism of hornification appears to dominate somewhat over mass loss. According to these results, no particular benefit is achieved by using slow drying processes. Further increment in mechanical properties might be gained by favoring hornification while avoiding mass losses greater than 2%–3%; for example, applying rapid high-temperature drying processes up to high dryness.

According to Fig. 7, ductility of wood increases with moisture content, which is in agreement with previous observations. ^{15,19,20} On the other hand, ductility is negatively affected by hornification and mass loss (Figs. 8, 9). It has been shown that ductility is reduced along with increasing mass loss, the inelastic component being more sensitive than the elastic one. ¹⁵ However, at constant mass loss, the inelastic ductility of heat-treated wood has been reported to be the highest for those specimens with the lowest hygroscopicity, this effect being related to hornification. ¹⁵ This apparent contradiction with the results shown in

Fig. 8 may be caused by different experimental arrangements. In the aforementioned study, the specimens were subjected to a heat-bath that included wetting, high-temperature drying, and rewetting along with decreasing temperature. In the present study, high-temperature drying was not followed by rewetting, which might have recovered inelastic ductility.

Finally, let us discuss the effect of drying damage, due to incompatibility of drying shrinkage, on the mechanical properties of dried wood. Such an effect is not very clear in the present data, whereas a remarkable increase in strength has been found in earlywood specimens in which drying shrinkage had been prevented. The incompatibility of drying shrinkage is pronounced when the microfibril angle varies within or between cell wall layers. In wood fibers, the S1 and S3 layers have large and widely varying microfibril angles, whereas in the S2 layer the microfibril angle is rather uniform. Thus, the drying damage is most pronounced in the case of tracheids with a small proportion of the S2 layer, i.e., earlywood tracheids.

In longitudinal tension, latewood fibers carry a significant portion of the total load, not only because of the greater density of latewood but also because it contains a higher proportion of the S2 layer, with a relatively small microfibril angle. Therefore, the drying damage is likely to be most pronounced in those sections of the microscopic structure of wood that actually carry a relatively small part of the total load. Consequently, drying damage may drastically reduce the strength of thin earlywood specimens, but it may barely affect the strength of the entire wood structure loaded in the longitudinal direction. In the case of loading in the radial direction, the influence of the S1 and S3 layers on the mechanical properties of wood is significant, and thus drying damage might possibly be observed not only in microtome-cut sections but also in macroscopic wood specimens.

Conclusions

Two mechanisms, such as degradation of structural components and formation of irreversible hydrogen bonds, are found to contribute to both the hygroscopicity and the mechanical properties of dried wood. Significant mass loss, caused by thermal degradation of cell wall components, occurs in slow high-temperature drying processes. Irreversible hydrogen bonding between wood elements (hornification) appears to be favored by high-temperature drying, as well as high ultimate dryness. Furthermore, some additional hornification is expected because of the slowness of any drying process. Regarding the specific strength and stiffness, the mechanism of hornification appears to dominate over the mass loss. Ductility, however, is negatively affected by both the mass loss and the hornification, the inelastic ductility being more sensitive than the elastic one. The effect of a single drying treatment on inelastic ductility may differ from a drying and rewetting cycle.

A third suspected mechanism, microscopic cell wall damage caused by incompatibility of drying shrinkage, does not appear to affect the mechanical properties of macroscopic wood specimens, at least not in bending experiments. Consequently, application of slow high-temperature drying processes may not provide much of benefit regarding the mechanical behavior of dried wood. An increment in the mechanical properties might be rather gained by favoring hornification while avoiding mass losses greater than 2%–3%; for instance, applying rapid high-temperature drying processes up to high dryness.

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