WEATHERING BEHAVIOR OF DIMENSIONALLY STABILIZED WOOD TREATED BY HEATING UNDER PRESSURE OF NITROGEN GAS

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ABSTRACT

With the goal of improving the weathering behavior of wood by reducing its hygroscopicity and accompanying dimensional changes, samples of spruce and beech were heat-treated under nitrogen pressure at 175 to 195 C and subjected to natural and artificial weathering. Beech had a significantly lower hygroscopicity and improved dimensional stability after heat treatment and was more resistant to weathering than the unheated control. Although the hygroscopicity of spruce was also significantly reduced by heat treatment, weathering resistance was diminished. Heat treatment of either species had small, but measurable effects on the performance and durability of semitransparent and film-forming stains applied to the samples.

Keywords: Heat treatment, weathering, accelerated weathering, dimensional stability, erosion, softwoods, hardwoods.

INTRODUCTION

Photochemical degradation of the near-surface wood substance and dimensional changes of wood members due to climate variations are important factors affecting the durability of outdoor wood finishes. Dimensional changes cause serious stresses in the wood surface region and in turn stresses in any coating film. This results in deformation, fatigue, and cracking of the film. Eventually, photochemical degradation and this stressing destroy the whole film.

Swelling and shrinking of wood also influences the reliability of those wood constructions that must have a high dimensional stability for good performance. This is, in particular, true for wood millwork (joinery) such as window frames. Thus, the dimensional stabilization of wood in these situations would improve their long-term durability in outdoor exposures. According to many earlier studies, heat treatment is one method of dimensionally stabilizing wood. The following report describes the benefits and disadvantages of heat treatment of wood with regard to weather resistance.

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PHYSICAL AND CHEMICAL CHANGES DUE TO HEAT TREATMENT

The physical property changes caused by the heat treatment of wood and the chemical and physical mechanisms of these changes have been studied by numerous researchers for a long time. It is not possible to give a comprehensive overview, in this report, of the previous work. Three major reviews have been published (Hillis 1984; Shafizadeh 1984; Stamm 1964).

The heat treatment of wood at high temperatures (100 to 200 C) produces an increasing dimensional stabilization effect as well as a significant loss of wood toughness and strength properties. This change of properties is mainly due to a thermal degradation of the hemicellulose. Cellulose and particularly lignin will be degraded slower and at higher temperatures than hemicellulose. Volatile decomposition products have less free polar adsorption places and are less hygroscopic, respectively, than the initial hemicellulose. The pentosans of hardwoods are more susceptible to degradation than the hexosans of the softwoods.

As a component of the wood polymer, hemicelluloses have the main function to connect cellulose and lignin components within the cell-wall substance, and also may have "gluing" effects between adjacent cell walls. Thus, changes in, or loss of, hemicellulose due to thermal degradation show some important wood property effects:

1. The effects of heat treatment on different wood species differ depending on the type and amount of hemicellulose. A hardwood like beech, for instance, shows more intense reactions than pine, and pine more than spruce. Thus, the optimal ratio of weight loss/dimensional stabilization is somewhat higher for beech than for pine and spruce (Giebeler 1983; Kollmann and Fengel 1965; Stamm 1964).
2. The resistance against decay by microorganisms is improved by heat treatment. Stamm (1956) and Stamm and Baechler (1960) explained that this resistance is caused mainly by the loss of constitutional water, by the reduced availability of hydroxyl groups, and by the replacing of these groups with nondecay-susceptible groups. Thus, an enzymatic attack cannot take place as fast as usual.
3. The observed reduction of toughness and strength across the wood fiber after heat treatment is greater for tension and shear loading than for compression. Also, internal strains can create some unusual checks. This loss of strength properties could reduce the possibilities of commercial use of the heat treatment (Seborg et al. 1953; Stamm 1964).
4. The stabilization process can be improved, and the loss of strength can be reduced using a closed system (autoclave) with an inert gas like nitrogen instead of air (Seborg et al. 1953).

In spite of Stamm's (1956) earlier skeptical judgments about the practical usefulness of heat treatment, Burmester (1973) tried to optimize the heat stabilization process by treating moist wood specimens under pressure in an autoclave. He called this process, "*F*euchte/*W*aerme/*D*ruck-Behandlung" (FWD); translated in English, *M*oisture/*H*eat/*P*ressure-Treatment (MHP). Burmester achieved stabilization effects up to 50% and found only small losses of wood strength. In 1983, Giebeler described a technical process that he introduced using an autoclave with a volume of 1.8 m^3. He calculated that a dimensional stabilization of about 50% could be achieved and would be economical for applications such as window millwork where a high dimensional stability is required and a 10% loss of strength could be tolerated.

TABLE 1. *Heat-treatment conditions in a nitrogen atmosphere.*

Treatment number	Intended stabilization level of D[b]	Treatment conditions[a]				
		Temperature		Pressure		Duration
	(%)	(C)	(F)	(bar)	(psi)	(h)
0	0 (for comparison)	—	—	—	—	—
1	25	175	350	10	145	2
2	50	185[c]	365	10	145	3

[a] According to data received from Ruetgerswerbre, West Germany.

[b] Calculated as the relation of the reduced swelling of the treated wood g_1 to the swelling of the normal wood g_2:

$$D(\%) = \left(1 - \frac{g_1}{g_2}\right) \cdot 100 = \text{dimensional stabilization.}$$

[c] Beech was treated at 195 C (385 F).

In 1984, the Wood Section of the Swiss Federal Laboratory of Materials Testing (EMPA) started an investigation on the properties and practical suitability of MHP-treated wood in cooperation with Giebeler (1983). A portion of the project is concerned with the weathering behavior of MHP-treated wood with and without surface protection with stains or paints. Because of the chemical modification of the wood due to heat treatment (reduction or loss of hydroxyl groups, low thermal dissociation) and physical changes (reduction of internal bond strength of the fibers), it was expected that weathering behavior would also be affected (Feist and Hon 1984).

MATERIALS AND METHODS

Heat treatment

Boards of European spruce (*Picea abies*) and beech (*Fagus sylvatica*) were treated using the procedure described by Giebeler (1983) (Table 1). Unlike the earlier recommendations of Burmester (1973), wood moisture content (MC) was kept low (8 to 12%) to avoid drying damage (Giebeler 1983). After treatment, the MC was between 4 and 6%.

The average wood density (oven-dry weight and volume) of the untreated and heat-treated wood was:

	Treatment number		
	Untreated, 0	MHP 1	MHP 2
Spruce, g/cm^3	0.41	0.51	0.44
Beech, g/cm^3	0.74	0.69	0.72

The loss of weight due to the treatment was approximately 10% (treatment MHP 1) and 15% (treatment MHP 2) for beech and 5 and 10% for spruce, respectively.

Measurement of dimensional stabilization

Three small boards of each wood species and treatment level were stored stepwise in conditioning rooms after treatment until equilibrium moisture content (EMC) was achieved. The climates were 27 C, 30% relative humidity (RH); 27 C, 65% RH; 27 C, 80% RH; and 21 C, about 100% RH (over water). Finally, the samples were oven-dried at 105 C for 24 hours. The size of the samples was 100 mm by 75 mm (longitudinal by across the fiber) with a thickness of about 6 mm.

TABLE 2. *Reduction of equilibrium moisture content, and reduction of swelling for heat-treated spruce and beech compared with untreated wood.*

Wood species, treatment	Reduction[a] of Equilibrium moisture	Reduction[a] of Swelling
	%	%
	27 C, 30% relative humidity	
Spruce, growth rings 45°		
MHP 1	30.3 ± 3.1	25.0 ± 15.0
MHP 2	43.1 ± 2.2	41.7 ± 3.3
Beech, radial		
MHP 1	27.2 ± 2.0	14.3 ± 8.6
MHP 2	59.1 ± 0.9	43.0 ± 5.7
Beech, tangential		
MHP 1	—	63.2 ± 15.8
MHP 2	—	76.0 ± 20.9
	27 C, 65% relative humidity	
Spruce, growth rings 45°		
MHP 1	30.0 ± 1.1	17.9 ± 7.5
MHP 2	44.7 ± 2.3	35.7 ± 2.9
Beech, radial		
MHP 1	30.4 ± 0.7	25.0 ± 4.5
MHP 2	55.9 ± 0.4	55.0 ± 4.0
Beech, tangential		
MHP 1	—	43.9 ± 8.0
MHP 2	—	63.4 ± 5.5
	27 C, 80% relative humidity	
Spruce, growth rings 45°		
MHP 1	28.2 ± 1.8	15.7 ± 7.7
MHP 2	44.9 ± 1.9	30.8 ± 2.7
Beech, radial		
MHP 1	28.2 ± 0.3	23.3 ± 4.7
MHP 2	56.3 ± 0.5	53.3 ± 4.3
Beech, tangential		
MHP 1	—	29.9 ± 13.0
MHP 2	—	63.1 ± 8.8
	21 C, 100% relative humidity	
Spruce, growth rings 45°		
MHP 1	27.8 ± 1.4	0.0 ± 17.7
MHP 2	41.6 ± 1.3	25.0 ± 5.9
Beech, radial		
MHP 1	18.0 ± 1.7	17.0 ± 6.4
MHP 2	52.9 ± 1.7	54.2 ± 5.6
Beech, tangential		
MHP 1	—	27.3 ± 15.0
MHP 2	—	65.5 ± 4.3

[a] Average value ± standard deviation.

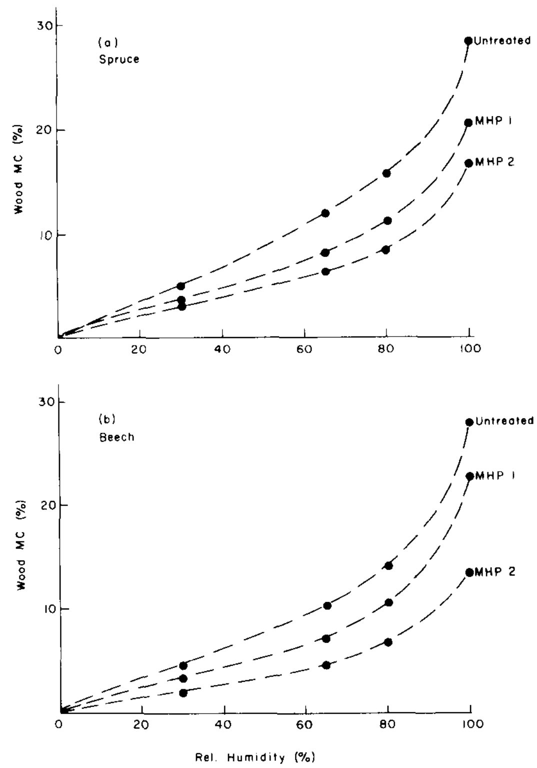

FIG. 1. Sorption isotherms of untreated and heat-treated spruce and beech.

Growth ring position was 90° to the sample surface for beech (plain sawed) and about 45° for spruce (bastard sawn). Samples with identical angles of the growth rings were not available. Therefore, only the relative results of dimensional stabilization of spruce and beech samples are comparable.

At the end of each climate step, sample weights and dimensions were measured in the radial and tangential direction (beech) and the 45° direction (spruce).

Artificial weathering

Three samples of each wood species (same dimensions as mentioned above) and the same treatment levels were left unfinished, whereas three samples each were finished by brushing with one coat of a laboratory-prepared semitransparent penetrating oil-based stain (Black et al. 1979), or with a film-forming semitransparent stain (commercial European alkyd resin product). The consumption of the liquid products was about 95 g/m^2 (penetrating stain) and 90 g/m^2 (film-forming stain) for beech, and 105 g/m^2 or 95 g/m^2 for spruce. Fifty-four samples were artificially weathered using a commercial accelerated-weathering chamber. Exposure consisted of a 24-hour-per-day light exposure (high intensity xenon arc) including a 4-hour period of distilled water spray while the light was on. Samples were fixed in stainless steel holders, which covered the upper part of the sample surface so that an unweathered reference area could be saved.

After exposure intervals of 600 hours of light, the samples were removed from

TABLE 3. *Erosion of untreated and heat-treated spruce and beech (radial surfaces) after exposure to artificial weathering.*

Wood species, treatment	Wood density (ovendry volume and weight)[a]	Depth of erosion after four intervals (h) of exposure (average ± standard deviation) 650	1,200	1,800	2,400	Average change of erosion from untreated samples
	(g/cm³)		*(μm)*[b]			*(%)*
Spruce, earlywood						
Untreated, 0	0.41 (0.3)	145 ± 30	235 ± 50	295 ± 60	435 ± 60	—
MHP 1	0.51 (0.3)	175 ± 20	350 ± 40	420 ± 40	580 ± 40	+33
MHP 2	0.44 (0.3)	95 ± 30	195 ± 50	235 ± 40	330 ± 80	−24
Spruce, latewood						
Untreated, 0	0.41 (0.95)	50 ± 30	65 ± 30	75 ± 30	100 ± 30	—
MHP 1	0.54 (0.95)	40 ± 10	60 ± 20	70 ± 30	95 ± 20	−5
MHP 2	0.44 (0.95)	25 ± 10	40 ± 10	55 ± 10	75 ± 30	−25
Beech, average of earlywood and latewood						
Untreated, 0	0.74	65 ± 20	100 ± 40	125 ± 40	160 ± 40	—
MHP 1	0.69	45 ± 20	85 ± 20	95 ± 30	115 ± 40	−28
MHP 2	0.72	30 ± 10	45 ± 20	50 ± 20	70 ± 30	−56

[a] Values in parentheses are estimates, other values are measured averages of earlywood and latewood combined.
[b] Nine single measurements.

the weathering chamber and kept under normal room climate conditions. Then the erosion depth of the surface of the unfinished samples was measured microscopically as described earlier by Black and Mraz (1974). The finished samples were judged by the eye concerning cracks, changes of the finish appearance, etc.

Natural weathering

Same size samples of each wood species and heat treatment were weathered naturally on the roof of a building at Madison, WI. Exposure conditions were 45° inclination towards the south. Both stain types were applied on one-half of each of the finished samples.

RESULTS AND DISCUSSION

Dimensional stabilization and other properties

As found earlier by others (Kollmann and Fengel 1965; Kollmann and Schneider 1963; Seborg et al. 1953; Stamm 1956), the main effect of heat treatment was a reduction of wood hygroscopicity (Table 2, Fig. 1). For spruce, this reduction of the wood EMC ranged between 30% (at 30 and 65% RH) and 28% (at 100% RH) following the lower temperature treatment MHP 1, and between 45% (at 65 and 80% RH) and 42% (at 100% RH) for treatment MHP 2. The equivalent EMC reduction values for beech were 30 to 18% (MHP 1) and 59 to 53% (MHP 2).

At higher MCs and near the fiber saturation point in particular, the reduction of the hygroscopicity decreased significantly. This could be explained by the fact that the number of hydrophilic groups of the wood substance was reduced by the heat treatment (as reported earlier). The cell-wall capillary system, on the other hand, might not be reduced or even expanded. Thus, the portion of the capillary condensation that takes place at higher levels of water sorption is not reduced to a large extent (Kollmann and Schneider 1963).

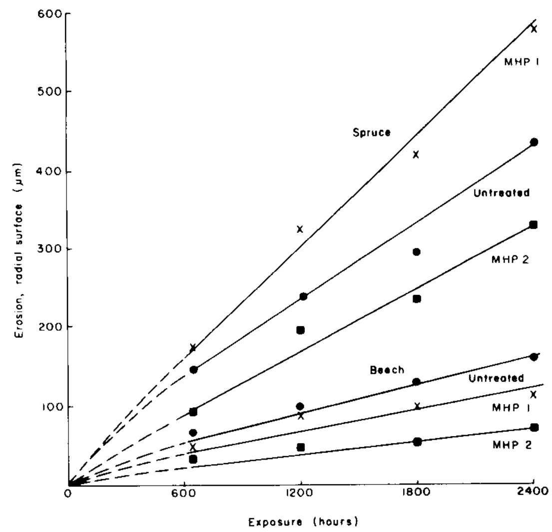

FIG. 2. Erosion depth for untreated and heat-treated spruce earlywood and beech (average of earlywood and latewood) as a function of the exposure time to artificial weathering. (Standard deviations shown in Table 3.)

Generally, the dimensional stabilization achieved the intended values of about 25% at treatment level MHP 1 and 50% for MHP 2 (Table 2). Spruce showed, however, a significant trend of decrease in the stabilization with increasing wood MC at both heat treatment levels. Beech did not exhibit this trend significantly.

It is obvious that the swelling of spruce per unit MC was extended by the treatment at both heat levels, in particular at higher MC levels. Beech, on the other hand, showed only a small effect and the swelling at higher MC levels was not increased, as Giebeler (1983) also showed.

The unsatisfactory result of the heat treatment on spruce can also be explained by changes of the internal wood cell-wall structure due to the treatment. However, it still needs to be explained why a hardwood like beech with a particularly high content of heat-degradable hemicellulose does not show this effect. According to Kollmann and Fengel (1965), one reason could be that the softwood cellulose has a lower thermal stability than the hardwood cellulose and will be degraded above temperatures of about 150 C. The degradation products might have a lower dimensional stability than the original cellulose.

Another effect of the heat treatment was, as expected, the increased brittleness of both spruce and beech, and reduction of strength properties across the fiber. A tendency for splintering was observed, particularly for those samples treated at the higher heat level. This splintering tendency also interferes with the workability of heat-treated wood.

Artificial weathering

The erosion rates (Table 3) of untreated spruce earlywood and latewood, and the average erosion rate of untreated beech (measured on radial surfaces), corresponded very well with the erosion of other softwood and hardwood species of similar wood densities (Sell and Feist 1986). The heat treatment significantly

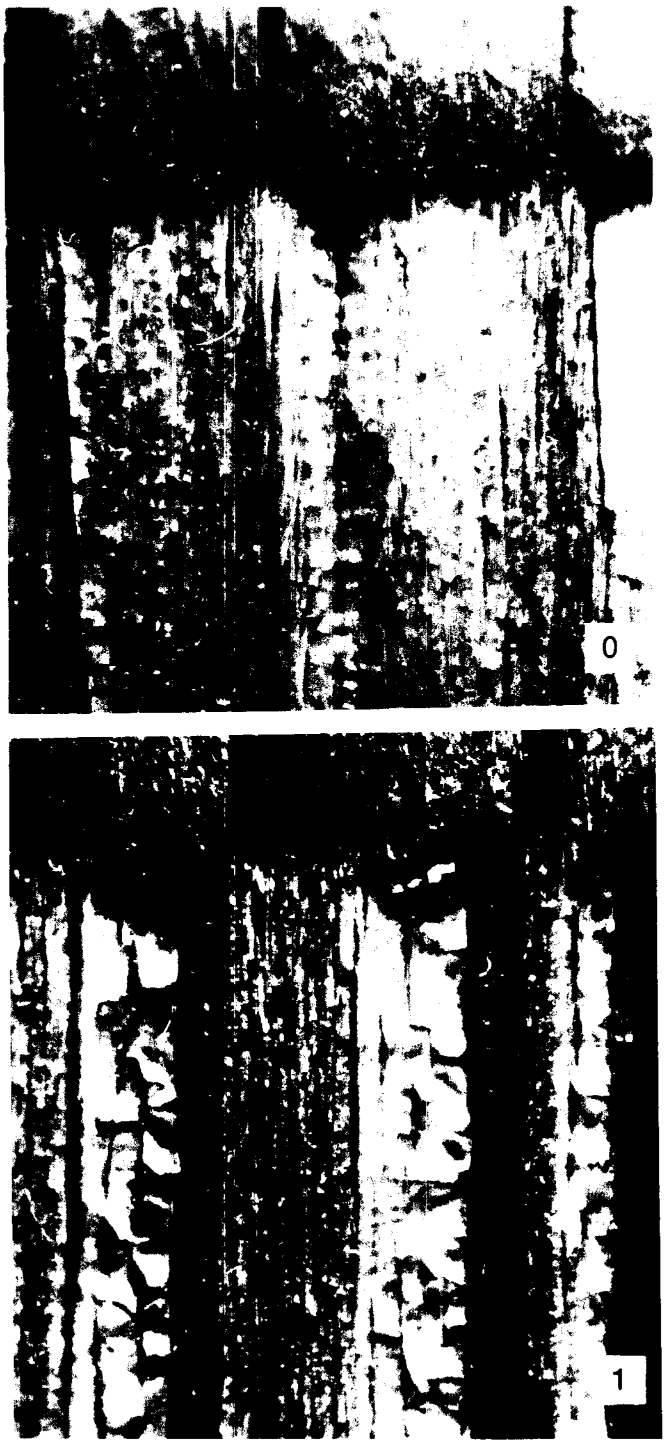

FIG. 3. Erosion patterns of untreated spruce (o) and heat-treated spruce (treatment level (1)) after 2,400 hours' exposure to artificial weathering. The earlywood of the heat-treated wood shows numerous cracks across the fiber; the untreated wood, only a few. The upper part of the samples was protected by a metal foil. Magnification: 20×.

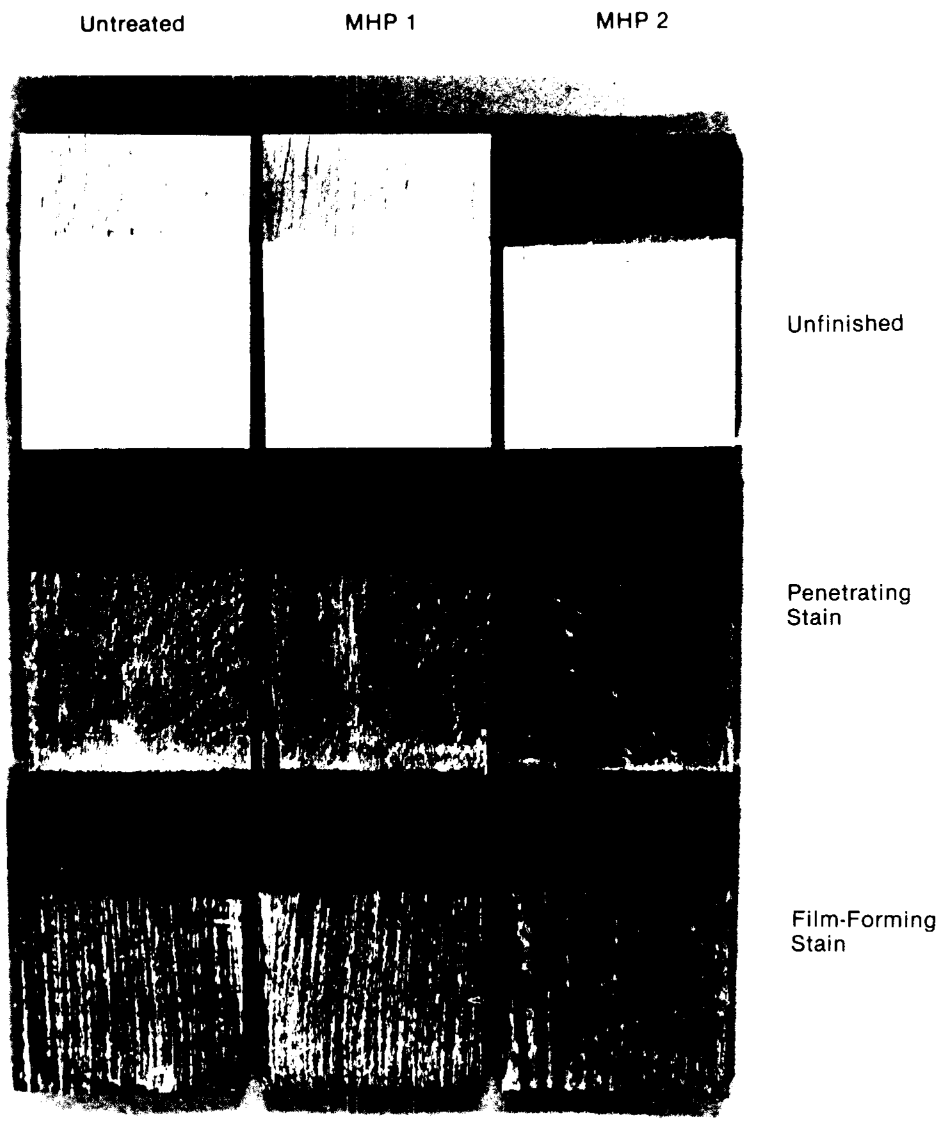

FIG. 4. Beech samples after 2,400 hours' exposure to artificial weathering. The degradations of the different treated samples show no significant differences between untreated and heat-treated (MHP 1, MHP 2) wood.

reduced the erosion rates of both spruce and beech with the exception of spruce at the lower treatment level (MHP 1). In this case, the erosion of the treated wood was significantly greater (+33%) than the erosion of the untreated wood (Table 3, Fig. 2).

With respect to the loss of internal bond strength and abrasion resistance of wood caused by the heat treatment (Stamm 1964), the reduced rates of erosion seem to be surprising, especially since the wood cell-wall density is reduced by the thermal degradation of hemicellulose. The opposite could be expected because

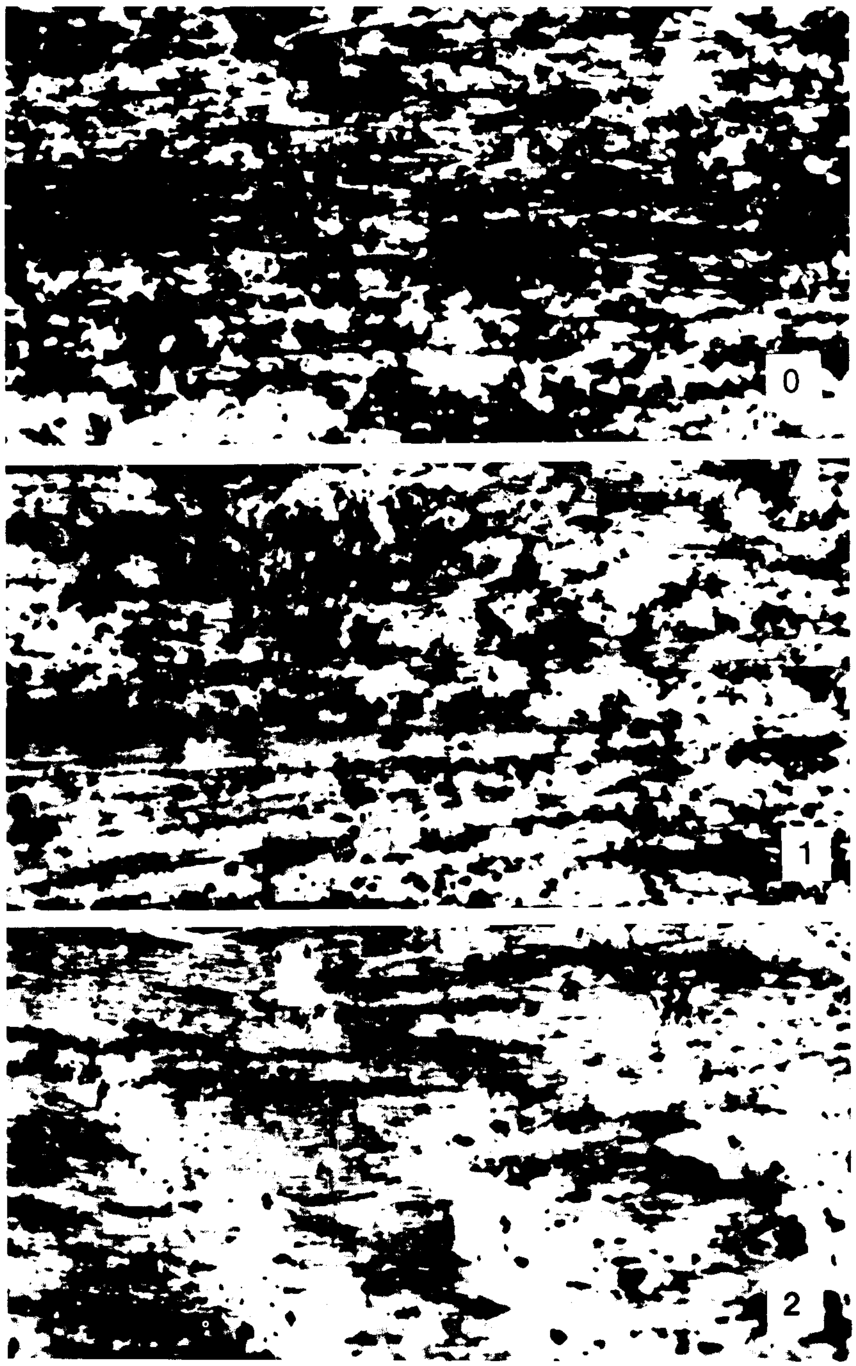

FIG. 5. Intensity of dark-colored mold fungi on untreated beech (o) compared to heat-treated beech with two different treatment levels (1, 2) after 9 months' natural weathering. Magnification: 45×.

TABLE 4. *Finish performance of stains on heat-treated wood after 14 months' outdoor exposure at 45° south in Madison, WI.*

Wood species, treatment	Finish performance[a]	
	Semitransparent stain	Film-forming stain
Spruce		
Untreated, 0	9.0	8.0
MHP 1	7.7	5.0
MHP 2	8.3	6.0
Beech		
Untreated, 0	5.3	3.7
MHP 1	6.3	2.3
MHP 2	7.7	3.3

[a] Finish performance values are on a 10 (no failure) to 1 (total failure) scale. The semitransparent stain was evaluated for erosion and the film-forming stain for flaking. Values are averages of three replicate samples.

of the dominant influence of wood density on the rate of erosion (Sell and Feist 1986). Besides chemical modification of the heat-treated wood, one important reason for this effect might be that photochemical degradation of wood is less after the heat treatment because of a reduction of the wood MC. The wood MC strongly influences photochemical degradation (Hon 1981). The unusual behavior of the spruce samples heat treated at the lower level is still not explained. These samples, however, also showed the lowest reduction of the dimensional changes (see above).

The different rates of erosion during artificial weathering were accompanied by different changes in surface texture. Generally the heat-treated beech samples had smoother surfaces with fewer checks and fissures than the untreated samples. Spruce, on the other hand, did not show such an effect of improved or smoother surfaces. Again the spruce samples heat treated at the lower level exhibited even rougher and more checked surfaces. Contrary to the untreated samples, numerous checks ran perpendicular to the fibers indicating that the cell-wall substance had been degraded to a large extent (Fig. 3).

The durability of both the semitransparent and film-forming stains was not significantly influenced by heat treatment. The penetrating stain was continuously weathered, and degraded after 2,400 hours of exposure on both heated and unheated samples. The film-forming stain, on the other hand, tended to fail in a more peeling-like behavior and showed major degradation after 1,200 hours (Fig. 4). There was no effect observed for heat treatment, and both heated and unheated samples performed similarly.

Natural weathering

After only 8 months' outdoor exposure, unfinished beech showed a significant influence of heat treatment on the intensity of mildew growth on the wood surface. Surface colonization by dark-colored hyphae (probably *Aureobasidium pullulans* (Feist and Hon 1984; Sell and Waechli 1969)) occurred much slower with increasing level of heat treatment (Fig. 5). This expected effect was explained by Stamm and Baechler (1960) as a result of a replacement of readily available hydroxyl groups by nondecay-susceptible groups. This effect continued even after 14 months' exposure, and mildew growth on heat-treated samples was less than on unheated ones, and fungal hyphae colonies were smaller. All samples were covered with mildew growth, however, and were gray.

After 14 months' exposure, unfinished samples had surfaces similar to those found during artificial weathering. There was considerably more grain-raising and cracking on heat-treated samples of spruce than on unheated ones, and surfaces were noticeably rougher. In contrast, heat-treated samples of beech were smoother than untreated ones with little noticeable differences in cracking.

The samples exposed outdoors with stain finishes showed measurable differences in performance after 14 months' exposure (Table 4). Both semitransparent penetrating and film-forming stains performed worse on the heat-treated spruce samples than on the untreated control. The effect was more pronounced for the film-forming stain. For beech, the semitransparent stain performed somewhat better on heat-treated samples than on unheated controls, while the film-forming stain performed very poorly on both heated and unheated samples (a value of 3 for finish performance means only 30% of the original finish is still on the wood surface).

CONCLUSIONS

By selectively degrading wood hemicelluloses, heat treatment reduces the quantity of free polar adsorption places available for water (particularly the hydroxyl groups). This is supposed to lead to a reduction of the wood hygroscopicity and thus reduces dimensional changes. The two wood species studied here (beech and spruce) showed, however, major differences in their response to heat treatment under nitrogen gas.

The moisture-related properties of the hardwood (beech) were significantly improved by both heat treatment levels. Also, a reduction of photochemical degradation and erosion during artificial weathering was observed. Heat-treated samples had improved natural weathering properties over those of unheated samples. Finally, the heat-treated beech samples showed an improvement of the resistance against discoloring mold fungi (mildew) during natural weathering. It is to be expected that the resistance of beech against wood-destroying fungi also is improved by the heat treatment.

Conversely, spruce exhibited only a minor part of these improvements, although the hygroscopicity was also significantly reduced by the heat treatment. The durability of penetrating and film-forming stains during artificial weathering was not significantly influenced by the heat treatments on spruce. Greater differences in durability were observed after natural weathering, but the results were inconsistent.

The improvement effects of hardwoods with a low-dimensional stability, like beech, that can be achieved by the MHP treatment within a nitrogen atmosphere are interesting enough to study further under practical conditions.

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