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Durability of heat-treated wood

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Heat-treated wood from the French process was laboratory tested for decay using agar block test and a modified soil block test. Water absorption, bending strength, lignin content and acid number were also determined to evaluate the effect of heat treatment. Heat treated samples exhibit a higher lignin content and a lower acid number compared to the untreated controls indicating the degradation of some hemicellulose and extractives compounds. The significant amount of water absorbed during water soaking or exposure to different relative humidities suggest that the heat treatment helps in releasing the stress in wood after the removal of hemicellulose and degradation of lignin rather than the reported significant cross link reaction of organic acid and the benzene ring of lignin. Cubes extracted with water or acetone or chloroform, and exposed to pure fungus cultures show a considerable weight loss, which confirms the absence of any extractable compounds toxic for decay fungi during the heat treatment. Significant weight loss was observed after 12 weeks exposure for laboratory soil block or after 6 to 8 weeks for agar block test. For the soil block test, weight loss of 11% was obtained for heat-treated samples exposed to *G. trabeum* and 46% for *P. placenta*. About 56% and 54% weight loss were obtained for southern pine control exposed to *G. trabeum* and *P. placenta*, respectively. The weight loss of the water and acetone extracted heat-treated sample exposed to *P. placenta* was 49.7% and 53.9%, respectively. Only about 11% and 14.8% weight loss was obtained for water and acetone extracted samples exposed to *G. trabeum*. The moisture content of the tested samples was about 70 ± 10% for the unheated controls and 50 ± 10% for heat-treated samples. This treatment may modify the durability from non-resistant to moderate/resistant species depending on the fungus species as defined in the ASTM 2017 standard. The data from the bending test indicate that such treatment may create a 10 to 50% reduction in MOR and deflection, which will limit the use of such wood for structural purposes.

Dauerhaftigkeit von hitzebehandeltem Holz

In Frankreich hitzebehandeltes Holz wurde auf seine Widerstandfähigkeit gegen biologischen Abbau geprüft. Dazu wurden der Agarblock-Test und ein modifizierter Bodentest verwendet. Wasserabsorption, Ligningehalt und Säurezahl wurden ebenfalls bestimmt, um den Einfluß der Hitzebehandlung abzuschätzen. Hitzebehandelte Proben haben einen höheren Ligningehalt und niedrige Säurezahl im Vergleich zu den Kontrollen, was auf den Abbau von Hemicellulosen und Extraktstoffen zurückzuführen ist. Beträchtliche Mengen an absorbiertem Wasser während des Wässerns oder bei verschiedenen Luftfeuchtigkeiten deuten darauf hin, daß die Hitzebehandlung Spannungen im Holz abbaut nach dem Entfernen von Hemicelluosen und Ligninabbau. Dies ist wahrscheinlicher als die früher angenommene Quervernetzung zwischen organischen Säuren und den aromatischen Ringen des Lignins. Holzwürfel, die vor der Hitzebehandlung mit Wasser, Aceton oder Chloroform extrahiert waren und reinen Pilzkulturen ausgesetzt wurden, zeigten einen beträchtlichen Gewichtsverlust, d.h. daß keine toxischen Inhaltsstoffe durch die Hitzebehandlung freigesetzt wurden. Beträchtliche Gewichtsverluste ergaben sich auch nach dem 12-wöchigen Bodentest oder nach 6 bis 8 Wochen für den Agartest. Beim Bodentest erreichte der Gewichtsverlust durch *G. trabeum* 11%, durch *P. placenta* 46%. Bei Kiefernproben waren es sogar 56% bzw. 54%. Die Gewichtsverluste von mit Wasser bzw. Aceton extrahierten und dann hitzebehandelten Proben betrugen gegenüber *P. placenta* 49,7 bzw. 53,9%. Die Werte gegenüber *G. trabeum* lagen dagegen nur bei 11% und 14,8%. Die Feuchte der geprüften hitzebehandelten Proben betrug 70 ± 10%, die der nicht erhitzten Proben 50 ± 10%. Die Hitzebehandlung könnte also die Dauerhaftigkeit von Holzproben von einer nicht widerstandfähigen zu einer mäßig widerstandsfähigen modifizieren, abhängig von der Pilzart gemäß ASTM 2017. Die Prüfung der Biegefestigkeiten ergab, daß die Hitzebehandlung einen Rückgang des MOR und der Durchbiegung um 10–50% verursachen kann, wodurch die Verwendung solcher Proben als Bauholz eingeschränkt ist.

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1
Introduction

Recent work on the heat treatment processes of timber currently called torrefied wood in France, plato wood in the Netherlands and retified wood in the USA has suggested that such types of processes can improve the performance of timber in several aspects (Ruyter 1989; van Zuylen 1995; Holz-Zentralblatt 1996). The foremost advantages reported of wood treated in this manner are the remarkable resistance to fungal decay attack (Ruyter 1989; van Zuylen 1995) without the need to use wood preservatives and with alleged consequent environmental advantages, the noticeably improved water repellancy, and the improved dimensional stability in relation to moisture variations. A one-step process has been reported by several french patents (Armines 1981a, 1981b, 1985) and two-step heat-treatment has been reported for Dutch processes (Ruyter 1989; van Zuylen 1995).

The maximum temperature during the heat treatment is varied from 180 °C to 280 °C and from 15 minutes to 24 hours depending on the process, wood species, sample size, moisture content of the wood, the desirable mechanical properties, resistance to biological attack and the dimensional stability of the final product. The presence of air or oxidant during the heat treatment may accelerate the degradation of wood components. Inert or reducing atmosphere is reported to facilitate the heat-treatment. A nitrogen atmosphere is normally used. The rate of thermal degradation of wood polyose in air is greater than in an inert environment. The chemical degradation of wood occurs in the order of hemicellulose, cellulose and lignin. A limited decomposition of lignin is observed at a temperature as low as 220 °C with the presence of phenolic substances such as vanillin, coniferaldehyde and syringyl aldehyde (Sandermann and Augustin 1964).

Improved dimensional stability of heat treated wood is reported to be due to the loss of constitutional water in wood (Seborg et al. 1953). Several authors (Guyonnet 1998; Tjierdsma et al. 2000) suggested that the heat treatment enhances cross-linking reactions of formaldehyde generated during the decomposition of wood organic acids and the phenol units of wood lignin. This theory may partially explain the dimensional stability of heat treated wood.

The chemistry of the modification of the polymeric wood constituents induced by heat treatments and leading to all such improvements has recently been investigated by CP MAS 13C NMR, elucidated and reported (Tjierdsma et al. 2000). However, the somewhat dark appearance of the heat-treated wood, as well as the very characteristic and persistent odor indicated that by-products, such as derivatives of extractives, lignin, hemicellulose and cellulose, are generated. In a recent paper (Kamdem et al. 1999), heat treated wood was extensively sohxlet extracted for several hours using organic solvent and water and then analyzed using GC/MS. Polynuclear aromatic hydrocarbons derivatives of phenantrene and acenaphtylene as well as other classes of polyaromatics compounds were present. It is most likely that the presence of all such compounds contributes, perhaps to a non-indifferent extent, to the reported resistance of heat treated timber to fungal and other biological attack. The extent of toxic and non-toxic compounds in the heat treated wood were not quantified, but their proportion appears to be quite low. Most of the extracted compounds were alkylated phenols, alkylated guaiacyl and syringyl structures resulting from the degradation of lignin. There was insufficient evidence in support of the hypothesis that the formation of toxic compounds generated during the heat treatment contributed to the decay resistance of heat treated wood.

Stamm et al. (1956) reported that when wood is heated at elevated temperatures, the reduction in hygroscopy and in swelling and shrinkage are due to the formation of ether linkage by the splitting of two adjacent hydroxyl groups. An appreciable increase in decay resistance and significant losses in strength was obtained at a temperature above 270 °C which is close to the temperature at which exothermic decomposition of wood becomes appreciable, and where the hemicellulose and lignin are attacked and the crystallinity of the cellulose modified. The loss of strength was attributed to the embrittlement of the fiber. The decay resistance was explained by the inertness of the material which is no longer susceptible to fungus attack because sufficient water is prevented from entering the cell wall to support the decay. Similar explanations were recently proposed to explain the decay resistance of heat-treated wood (Vernois et al. 1999).

2
Experimental

Maritime pine, spruce, beech and poplar measuring 5 cm by 10 cm by 200 cm that were heat-treated at temperatures ranging from 200 to 260 °C for 1 to 24 hours were obtained from France. The exact schedule is proprietary information (Avat 1993).

2.1
Laboratory soil block and agar tests

Heat-treated samples were sterilized by ionizing radiation (AWPA-E10 1998). For each treatment and each fungus, seven replica were used. Two brown rot fungi, *Gloeophyllum trabeum* (Pers ex. Fr.) Murr (Madison 617, ATCC 11539) and *Poria placenta* (Fr.) Cooke Madison 698, ATCC 11538) and one white rot fungus, *Irpex lacteus* Fries (Madison 517, ATCC 11245) were used to evaluate the decay resistance as described in AWPA standard method E10-91 (1) with some slight modifications (Kamdem et al. 2000). Boxes were inoculated with monocultures of fungus and incubated until the aspen feeder strip was covered before cubes were introduced. After 12 weeks for the soil block and 8 weeks for the agar test, cubes were removed from the culture boxes and scraped clean to remove superficial mycelium and reconditioned until their weight stabilized. The percentage of weight loss after exposure to pure monoculture of test fungus was used as the index of decay. The moisture content of samples after exposure to the decay test was measured.

2.2
Water absorption

Samples of heat treated wood measuring 1 by 2 by 2 inch were soaked in water and in an alkali solution containing 18% sodium hydroxide for 24 hours. The water absorbed and the volumetric swelling were measured and compared to that of the unheated control.

To determine the equilibrium moisture content of heat treated wood, samples were placed in a desiccator at 23 ± 3 °C with the relative humidity of 66, 86, and 100%. The specific relative humidity was provided by a saturated salt solution at 68 F. Sodium nitrite was used for 66% RH and potassium chloride for 86% RH. For 100% RH, liquid water was placed in the bottom of the desiccator. The weight of exposed samples was recorded every 48 hours until an equilibrium moisture content was reached. After 30 days the weight of the samples was constant.

2.2
Acid number in wood using conductance titration

Solid wood was Wiley milled, and the fraction retained by 40–60 mesh size was weighed and used for the acid number determination. About 5 grams were treated with 200 ml of 0.001 molar sodium chloride and 2 ml of 0.1 molar HCl to convert all the carboxyl groups in wood to their acid form. A solution of 0.1 M NaOH was used for titration. A laboratory conductivity meter from VWR was used to measure the conductance during the titration. This yields the amount of carboxylate groups in the 5 grams of ground wood. The percentage of Klason lignin in control and heat-treated samples was determined following the TAPPI method (1990).

3
Results and discussion

3.1
Lignin content and acid number

The lignin content and the acid number of the heat-treated and untreated control are listed in Table 1. The Klason lignin content of heat-treated wood was higher than that of the untreated control and this increase in lignin content can be attributed to the loss of hemicellulose or fragile pentoses and hexoses during the heat treatment. The increase in lignin content does not imply the formation of lignin during the process but the reduction of other wood components. The percent increase in lignin content was higher for beech than for the softwood. Since the exact condition of heat treatment is unknown, the explanation for this phenomenon is purely speculative, although it is known that the pentoses of hardwood are more unstable than the hexoses of softwood.

The acid number yields information on the polyose in wood with carboxylic groups or other groups susceptible to be easily converted into carboxylic groups. The wood components rich in carboxylic acid are the hemicelluloses. The values of the acid number in Table 1 clearly indicate a decrease in acid content after the heat treatment. It was reported that hemicellulose is significantly degraded during the treatment of wood with chromated copper arsenate (Winandy 1988) and at a temperature as low as 180 °C (Seborg et al. 1953) without CCA.

Table 2 contains the percentage of wood swelling after 24 hours soaking in water and in a 18% sodium hydroxide solution. Sodium hydroxide was used to establish if there were any ether cross linking bonds during the heat treatment as often speculated in the literature (Pizzi 1998). Seborg et al. (1953) reported that if ether cross links were formed during the heating of wood, the swelling in concentrated sodium hydroxide should be significantly less than the swelling of the unheated controls as sodium hydroxide is not capable of breaking ether linkages. The data in Table 2 on the swelling of spruce and beech cross sections show that with beech, the heat treatment reduces the subsequent swelling only in water. No significant difference was noticed in alkali solution. For spruce, heat treatment reduces the swelling in water and increased the swelling in sodium hydroxide to 28%. This indicates that the resulting stability from heat treatment cannot be totally attributed to ether cross links but to some other causes that need to be investigated.

The moisture content of wood exposed to 66, 86 and 100% relative humidity after 30 days are listed in Table 3. At 66% RH, the equilibrium moisture content (EMC) was comparable between the spruce controls and the heated spruce specimens, but not for beech. However, beech, ash

Table 2. Swelling of heat-treated wood in water and in 10% sodium hydroxide solution after 24 hours soaking
Tabelle 2. Quellung von hitzebehandeltem Holz in Wasser und 10% NaOH nach 24 h Lagerung

Species	Treatment	Water liquid	18% NaOH in water
Spruce	Control	38	18
Spruce	Heat	18	28
Beech	Control	27	33
Beech	Heat	17	28

Table 1. Lignin content and acid number of heat-treated wood
Tabelle 1. Ligningehalt und Säurezahl der hitzebehandelten Proben

Species	Treatment	Lignin, %		Acid, mmol/gram wood	
		Value	Change	Value	Change
Pine	No	26.0		0.081	
Pine	Heat	28.0	+7	0.032	−61
Spruce	No	25.3		0.052	
Spruce	Heat	29.9	+15	0.024	−54
Beech	No	20.5		0.042	
Beech	Heat	26.6	+23	0.023	−45

and birch exhibit the lowest EMC.(12%) at 100% compared to the softwoods with EMC varying from 16 to 20%. The low EMC of hardwood could be due to the removal of important amount of hemicellulose as suggested by the high lignin content and low acid number of beech after the heat treatment.

3.2
Water absorption

Table 3 contains the moisture content of 0.5 inch cubes used in an agar block test. After a 6 weeks exposure of heat treated specimens and controls to different monocultures of fungi in petri dishes containing 2% of malt agar, the moisture content of the samples varies from 72% to 156%. This clearly suggests that heat-treatment does not limit the absorption of water. Although a slight difference may be noticed between the MC of heat-treated specimens and controls, the number of replica and the size of samples used in this study may not be sufficient to draw any conclusions, and further investigations are needed to confirm the practical importance of this level of water absorption in heat-treated wood.

After three months exposure of heat-treated samples to soil substrate containing about 120 ± 10% moisture content, the moisture content of heat-treated wood ranged from 30 to 70% (Table 5). The moisture content of the untreated control was about 80%. The heat treated samples absorb a significant amount of water.

The effect of heat-treatment points to a stress relaxation as one of the plausible causes rather than just a modification of the hygroscopicity of the heat-treated samples. Optimal moisture content levels for the growth of wood destroying fungi are not well known, but experience with laboratory decay tests indicates that the optimal levels range from 40 to 80%. It was reported that heat-treated wood under severe thermal treatment, such as 260 °C, results in a low EMC of 10% compared to 30% with untreated wood exposed to 96 to 100% RH and an improvement in dimensional stability of about 50% (Tjeerdsma 1998). The low water absorption as an explanation of the decay resistance of heat-treated wood was not observed in this study.

The moisture content of wood is usually explained by interaction of chemical constituents of wood and the water from the capillary nature of the amorphous zones of the microfibrils of the cell walls. Hemicellulose equilibrates at the highest EMC and the lignin at the lowest. Hemicelluloses degraded by heat-treatment at 180 °C (Kollmann and Fengel 1965) and the stresses stored in the matrix substance and the microfibrils can be released. It can be postulated that the dimensional stability of heat-treated wood may partially result from the release of the stresses stored in microfibrils and wood matrix following the degradation of hemicellulose.

3.3
Surface

The reduction of surface roughness of heat-treated wood was significant. A difference was detected just by rubbing a hand over the heat-treated and the untreated wood. The surface color of heat-treated samples was also darker

Table 3. Moisture content at different RH after 30 days
Tabelle 3. Feuchte nach 30 Tagen Lagerung bei verschiedenen Luftfeuchten

Species	Heat treatment	Initial MC, %	66%	86%	100%
Spruce	No	8 ± 1*	8 ± 1	14 ± 1	26 ± 2
Spruce	Yes	5 ± 1	7 ± 1	11 ± 1	20 ± 2
Beech	No	7 ± 1	10.0 ± 2	14.5 ± 2	21.8 ± 2
Beech	Yes	4 ± 1	5 ± 1	8 ± 1	12 ± 2
Pine	Yes	3 ± 1	6 ± 1	9.4 ± 2	16 ± 2
Ash	Yes	3 ± 1	4.4 ± 1	7.7 ± 1	12 ± 1
Birch	Yes	3 ± 1	4.9 ± 1	6.6 ± 1	12 ± 1

Table 4. Weight loss (WL, %) and moisture content (MC, %) after agar plate block test
Tabelle 4. Gewichtsverlust (WL) und Feuchte (MC) nach dem Agarblock-Test

Species and treatment	Brown rot *G. trabeum*		Brown rot *P. placenta*		White rot *I. lacteus*		soft rot *C. globosum*		control no fungus	
	WL	MC	WL	MC	WL	MC	WL	MC	WL	MC
Pine control, no heat	19(2)	156(5)	20(1)	159(3)	13(1)	129(5)	8(1)	118(11)	3(1)	121 (9)
Pine heat-treated no extraction	7(2)	120(7)	8(2)	125(15)	6(1)	120(4)	4(1)	104(17)	3(1)	72(26)
Pine heat-treated + Water extract	8(2)		14(2)		9(1)					
Spruce heat-treated + CHCL3	9(2)		8(5)		9(3)					
Spruce heat-treated no extraction	7(2)		17(3)		8(2)					
Spruce heat-treated + Water extract	10(3)		19(5)		10(2)					
Spruce heat-treated + CHCL3 extracted	12(3))		17(4)		12(3)					
Poplar heat-treated	16(5)		16(2)		13(2)					
Poplar heat-treated + Water extract	10(2)		9(2)		8(2)					
Poplar heat-treated + CHCL3 extracted	13(2)		10(3)		8(3)					
Control Southern pine	47(10)		55(10)		40(10)					

Values in parentheses are the standard deviations

Table 5. Weight loss (%) and moisture content (%) after laboratory soil block
Tabelle 5. Gewichtsverlust und Feuchte nach dem Bodentest

Species	Extraction solvent	Brown rot *G. trabeum*		Brown rot *P. placenta*		White rot *I. lacteus*		Soil MC
		WL	MC	WL	MC	WL	MC	
Pine		11(8)	63(26)	47(8)	62(13)	28(10)	29(10)	127
	Water	11(10)	54(16)	50(8)	71(13)	12(7)	35(10)	112
	Acetone	15(17)	48(10)	54(3)	66(12)	24(3)	34(12)	136
SYP	Control	57(8)	81(19)	54(3)	62(10)	35(6)	60(16)	123

Table 6. The effect of heat-treatment on the bending properties
Tabelle 6. Einfluß der Hitzebehandlung auf die Biegeeigenschaften

Species	Treatment	Density kg/m^3		Deflection, inch		MOE, 10^6 * psi		MOR, 10^3 * psi	
		Value	change	Value	Change	Value	Change	Value	Change
Spruce	Control	447(6)		0.40(0.11)		1.18(0.3)		11.3 (2)	
	Heat	381(13)	−15%	0.33(0.03)	−17%	1.07(0.1)	−11%	10.4(0.9)	−8%
Beech	Control	623(10)		0.29(0.01)		1.25(0.1)		16(1)	
	Heat	617(63)	−1%	0.14(0.04)	−52%	1.0(0.2)	−20%	9.6(2.2)	−40%
Pine	Heat	530(41)		0.28(0.08)		1.76(0.5)		6.5(0.6)	

than the untreated samples suggesting an important modification in the chemical composition of heat-treated wood.

3.4
Biological performance

Data of the laboratory agar block test using heat-treated specimens and non-heat-treated controls are listed in Table 4. After 6 weeks exposure to the agar block tests, the weight loss (WL) of untreated and heat-treated blocks was higher than the one of the controls exposed to agar without fungus inoculation. The weight loss of control samples and heat-treated samples without inoculation averaged 3%. The weight loss of control southern pine used to assess the vigor of the fungus are listed in Table 4: about 47% for *G. trabeum*, 55% with *P. placenta* and 40% with *I. Lacteus*. The level of weight loss of heat-treated samples was significantly different compared to the operational weight loss, suggesting that the level of decay resistance achieved by such heat treatment is not sufficient to justify the use of heat-treated wood in ground contact without any further protection.

The weight losses from laboratory soil block tests in Table 5 with brown and white rot fungi suggest that heat treatment does not sufficiently increase the decay resistance to use the heat-treated wood in ground contact without further protection. For above ground use, specific tests need to be carried out.

Table 6 shows that the deflection and the modulus of rupture of bending are severely affected by the heat treatment. Beech is the most affected with a reduction of 52% in deflection and 40% in MOR. The reduction in mechanical properties may be correlated with the low EMC, the darker color, the low acid number and the high lignin content after the heat treatment. It is more likely that the lowest EMC will correspond to a high decay resistance and then to a high dimensional stability that could be achieved with severe heat-treatment conditions, such as high temperature over a prolonged period.

Initially, one of the objectives was to determine whether or not during the heat treatment some extractable compounds toxic to decay fungi may be generated. Heat-treated pine, spruce and poplar extracted with either water, acetone, chloroform and exposed to fungus exhibit weight loss. Table 4 indicates that the extent of fungus attack varies with the solvent used for extraction. No significant difference at 95% confidence was observed for pine species and the three solvents used. The same conclusion held for spruce. For poplar, the WL of heat-treated but no extraction was quite high and no scientific reasons other than the variability known in wood science could be given.

4
Conclusions

This study shows that heat-treatment affects the mechanical properties of wood and that hardwood species are more susceptible than softwood. The decay resistance of heat-treatment may be achieved but at a high cost and with reduced mechanical properties. Laboratory and field tests data are needed to assess the use of heat-treated wood for above ground contact.

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