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► **To cite this version:**

Marc Borrega, Petri P. Kärenlampi. Hygroscopicity of heat-treated Norway spruce () wood. *European Journal of Wood and Wood Products*, 2009, 68 (2), pp.233-235. 10.1007/s00107-009-0371-8 . hal-00568249

HAL Id: hal-00568249

<https://hal.science/hal-00568249>

Submitted on 23 Feb 2011

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Draft Manuscript for Review

Hygroscopicity of heat-treated Norway spruce (*Picea abies*) wood

Journal:	<i>Holz als Roh- und Werkstoff</i>
Manuscript ID:	HRW-09-0083.R1
Manuscript Type:	KURZORIGINALIA / BRIEF ORIGINALS
Keywords:	drying, heat treatment, hornification, hydrogen bonding, hygroscopicity, mass loss, wood



Review

Hygroscopicity of heat-treated Norway spruce (*Picea abies*) wood

Marc Borrega* and Petri P. Kärenlampi

*Faculty of Forest Sciences, University of Joensuu, P.O. Box 111, 80101 Joensuu,
Finland*

*Corresponding author:

marc.borrega@joensuu.fi

Subject

Hygroscopicity of heat-treated spruce wood is investigated in relation to the mass loss that occurs during the thermal treatment. It is found that the reduction in hygroscopicity is not only due to mass loss, but another mechanism exists. It is hypothesized that this mechanism is related to irreversible hydrogen bonding in the course of water movements within the pore system of the cell walls. Regarding batch experiments with constant amount of water and wood, such hornification occurs during a wetting and drying cycle induced by heating to a predetermined steady-state temperature, provided an intermediate relative humidity prevails at the isothermal stage.

This paper was partly presented at the Nordic Workshop in Wood Engineering, Skellefteå, Sweden, February 21, 2007

1. Introduction

The exposure of wood to elevated temperatures causes thermal degradation of its components, which often results in loss of mass. Thermal degradation is utilized in heat treatment processes to produce wood products with reduced hygroscopicity, among other modified properties (Militz 2002, Stamm et al. 1946). The reduced hygroscopicity of heat-treated wood is often related to the mass loss that occurs during the thermal treatment. This is because the mass loss is mainly due to degradation of hemicelluloses, the most hydrophilic component in the wood cell wall (Bourgois and Guyonnet 1988, Zaman et al. 2000). Chemical changes and structural modifications in the cell wall during heating have also been reported to play a significant role in hydrophobicity of wood (Hakkou et al. 2005a, Repellin and Guyonnet 2005).

The water-swelling capability of pulp fibres is known to be reduced along with drying, even at temperatures as low as 20 °C (Matsuda et al. 1994). This phenomenon, denoted as hornification, is usually explained by the adhesion of adjacent microfibrils during water removal, following the collapse of pores in the cell wall (Crawshaw and Cameron 2000, Weise 1998). The mechanism behind hornification is thought to be the formation of irreversible hydrogen bonds between carbohydrate elements (Kato and Cameron 1999, Matsuda et al. 1994, Weise 1998). Hornification has been shown to be enhanced with additional drying cycles (Crawshaw and Cameron 2000, Weise 1998).

This study aims at investigating the mechanisms affecting the hygroscopicity of wood subjected to thermal treatments under different process conditions. The hygroscopicity of heat-treated wood is analyzed in relation to the mass loss that occurs during the thermal treatment.

2. Experimental

The wood material used was Norway spruce (*Picea abies*) felled in Joensuu, Finland. Wood specimens with dimensions of 24 x 24 mm² in cross section and 320 mm in length were prepared. All specimens were clear of visible defects. Eighteen groups containing 12 specimens each were formed, and each group was subjected to a particular heat-bath treatment. An additional group of 24 specimens, oven-dried at 85 °C for 48 hours and subsequently at 103 °C for 24 hours, was used as reference.

Heat-bath treatments were conducted in a pressure vessel equipped with a temperature gauge and a pressure gauge. Dry specimens (about 1% moisture content) were placed in the vessel, together with a measured amount of liquid water. The vessel was heated to a predetermined setup temperature, the amount of water in the vessel corresponding to a predetermined relative humidity at the setup temperature. The setup temperature was retained for an isothermal period, after

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3 which the vessel was allowed to slowly cool before removing the specimens. The
4 experimental parameters for each treatment are shown in Table 1. The mass loss
5 of any heat-treated specimen was determined on a dry mass basis after oven-
6 drying at 103 °C for 24 hours. All the specimens were then conditioned in a
7 climate-controlled room at 19 °C and 65 % relative humidity for a minimum
8 period of 3 months, during which every specimen attained equilibrium moisture
9 content (EMC).
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14 (Table 1 about here)
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17 **3. Results and discussion**

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19 Hygroscopicity of heat-treated wood, measured by the EMC, is shown as a
20 function of mass loss in Fig. 1. As compared with the reference specimens, heat-
21 treated wood clearly exhibits a reduction in hygroscopicity. At any setup
22 temperature and relative humidity, the hygroscopicity decreases with increasing
23 mass loss. Furthermore, as a function of mass loss, the data seem to form two
24 different groups, specimens treated at intermediate relative humidity showing
25 almost 2 % units lower EMC than specimens treated either in dry or water-
26 saturated conditions. Therefore, hygroscopicity of heat-treated wood cannot be
27 solely explained in terms of mass loss. Obviously, some phenomenon related to
28 the relative humidity of the heating atmosphere must be involved.
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33 (Figure 1 about here)
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37 High temperature increases the mobility of molecular chains in the wood cell wall
38 promoting structural rearrangements (Fahlén and Salmén 2003, Hakkou et al.
39 2005b). Water, as a softener, provides further flexibility to the cell wall and thus
40 structural rearrangements are increased (Matsuda et al. 1994). Along with drying,
41 this results in a tightly bonded structure in which some of the polar sites
42 previously available for water sorption may not open upon rewetting (Crawshaw
43 and Cameron 2000). Thus, some of the hydrogen bonds formed within the
44 hemicelluloses and the amorphous regions of cellulose may be irreversible (Kato
45 and Cameron 1999, Matsuda et al. 1994, Weise 1998). The consequences of the
46 irreversible hydrogen bonding might well be denoted as hornification.
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51 Hornification is generally associated with changes in moisture content being most
52 pronounced at high temperatures (Crawshaw and Cameron 2000; Weise 1998).
53 Intermediate relative humidity at the setup temperature was produced by placing
54 dry specimens in the pressure vessel along with a predetermined amount of liquid
55 water. Then, the water penetrated the specimens entering more readily the large
56 pores and cavities, but also entering smaller pores along with time. Further on,
57 along with increased temperature, the relative humidity within the vessel became
58 reduced. Thus, some of the water that had entered the wood vaporized inducing a
59 drying sequence in the disordered pore system of the cell walls.
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3 High-temperature drying obviously did not happen in the case of specimens
4 heated in water-saturated conditions. On the other hand, specimens treated in a
5 dry atmosphere were not wetted during the experiment. Only those specimens
6 treated at intermediate relative humidity experienced a wetting and high-
7 temperature drying cycle during the experiment with the consequent formation of
8 irreversible hydrogen bonds within the wood cell walls. This is likely to explain
9 the lower hygroscopicity of such specimens, as shown in Fig. 1.
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14 The experiments reported here were batch-experiments. In other words, all the
15 constituents involved in the heat-bath treatment, *i.e.*, dry wood and water, were
16 added at the beginning of the experiment and extracted at the end of the
17 experiment. This induced a sequence of wetting and high-temperature drying in
18 the case of the specimens treated at intermediate relative humidity. The authors
19 hypothesize that intermediate relative humidity during a heat treatment probably
20 would not result in reduced hygroscopicity if the relative humidity remained
21 constant during the heat treatment. Such an experiment would require steaming of
22 the specimens in the course of the temperature increment.
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30 Acknowledgements

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33 Marc Borrega gratefully acknowledges funding provided by the North Karelia Regional Fund of
34 the Finnish Cultural Foundation.
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40 References

- 41
42 Bourgois J, Guyonnet R (1988) Characterization and analysis of torrefied wood. Wood Sci
43 Technol 22:143-155
44
45 Crawshaw J, Cameron RE (2000) A small angle X-ray scattering study of pore structure in
46 Tencel[®] cellulose fibres and the effects of physical treatments. Polymer 41:4691-4698
47
48 Fahlén J, Salmén L (2003) Cross-sectional structure of the secondary wall of wood fibers as
49 affected by processing. J Mater Sci 38:119-126
50
51 Hakkou M, Pétrissans M, El Bakali I, Gérardin P, Zoulalian A (2005a) Wettability changes and
52 mass loss during heat treatments of wood. Holzforschung, 59:35-37
53
54 Hakkou M, Pétrissans M, Zoulalian A, Gérardin P (2005b) Investigation of wood wettability
55 changes during heat treatment on the basis of chemical analysis. Polym Degrad Stabil 89:1-5
56
57 Kato KL, Cameron RE (1999) Structure-property relationships in thermally aged cellulose fibers
58 and paper. J Appl Polym Sci 74:1465-1477
59
60 Matsuda Y, Isogai A, Onabe F (1994) Effects of thermal and hydrothermal treatments on the
reswelling capabilities of pulps and papersheets. J Pulp Pap Sci 20:323-327

- 1
2
3 Miltz H (2002) Heat treatment technologies in Europe: scientific background and technological
4 state-of-art. In: Enhancing the durability of lumber and engineered wood products. Forest Products
5 Society, Kissimmee, Orlando, USA, February 11-13
6
7 Repellin V, Guyonnet R (2005) Evaluation of heat-treated wood swelling by differential scanning
8 calorimetry in relation to chemical composition. *Holzforschung* 59:28-34
9
10 Stamm AJ, Burr HK, Kline AA (1946) Staybwood...Heat-stabilized wood. *Ind Eng Chem* 38:630-
11 634
12
13 Weise U (1998) Hornification: mechanisms and terminology. *Pap Puu- Pap Tim* 80:110-115
14
15 Zaman A, Alén R, Kotilainen R (2000) Thermal behavior of Scots pine (*Pinus sylvestris*) and
16 silver birch (*Betula pendula*) at 200-230°C. *Wood Fiber Sci* 32:138-143
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Table 1. Process parameters for each heat-bath treatment. Standard deviation in parentheses

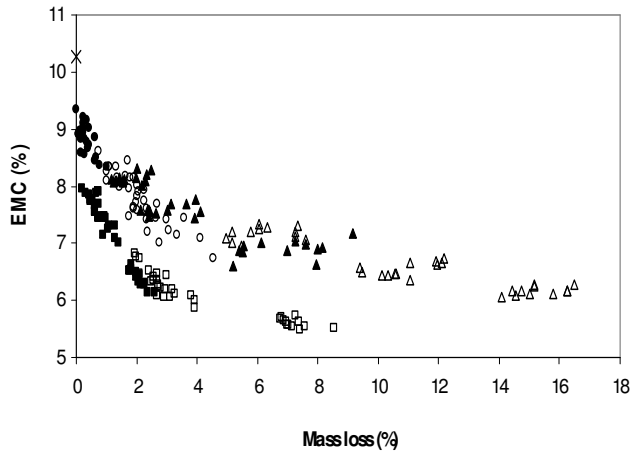
Tabelle 1 Versuchsparameter für verschiedene Wärmebadbehandlungen. Standardabweichung in Klammern

Setup temperature [°C]	*Relative humidity [%]	Isothermal treatment [hours]	Loss of mass [%]	EMC [%]
150	8	0	-	9.3 (0.3)
	8	2	0.2 (0.3)	9.0 (0.3)
	8	8	0.4 (0.3)	8.7 (0.3)
	60	0	0.6 (0.2)	7.8 (0.2)
	51	2	1.0 (0.2)	7.3 (0.2)
	57	8	2.1 (0.3)	6.4 (0.2)
	100	0	1.8 (0.5)	8.1 (0.1)
	100	2	3.0 (0.7)	7.6 (0.1)
	100	8	6.9 (1.3)	6.9 (0.2)
	170	10	0	1.5 (0.5)
15		2	2.1 (0.7)	7.8 (0.3)
15		8	2.8 (0.9)	7.3 (0.3)
55		0	2.4 (0.4)	6.5 (0.2)
50		2	3.2 (0.5)	6.2 (0.2)
61		8	7.2 (0.5)	5.6 (0.1)
100		0	6.2 (0.9)	7.2 (0.1)
100		2	10.9 (1.0)	6.6 (0.1)
100		6	15.3 (0.8)	6.2 (0.1)

* Relative humidity at the beginning of the isothermal treatment

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4 **Figure 1.** Equilibrium moisture content (EMC) of heat-treated wood at 19 °C and 65 % relative
5 humidity, plotted as a function of the mass loss for each heat-bath treatment. *Filled circles*, dry
6 conditions at 150 °C; *open circles*, dry conditions at 170 °C; *filled squares*, intermediate relative
7 humidity at 150 °C; *open squares*, intermediate relative humidity at 170 °C; *filled triangles*, water-
8 saturated conditions at 150 °C; *open triangles*, water-saturated conditions at 170 °C; marker on the
9 y-axis: mean value for reference wood
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14 Abb. 1 Gleichgewichtsfeuchte (EMC) von wärmebehandeltem Holz bei 19°C und
15 65% relativer Feuchte in Abhängigkeit des Masseverlustes für jede
16 Wärmebadbehandlung. *Ausgefüllte Kreise*: trockenes Klima, 150°C, *leere Kreise*:
17 trockenes Klima, 170°C, *ausgefülltes Rechteck*: mittlere relative Luftfeuchte,
18 150°C, *leere Rechtecke*: mittlere relative Luftfeuchte, 170°C, *ausgefüllte*
19 *Dreiecke*: wassergesättigt, 150°C, *offene Dreiecke*: wassergesättigt, 170°C.
20 Markierung auf der y-Achse: Mittelwert für Kontrollprobe
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