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

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Review

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

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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

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

(Table1 about here)

3. Results and discussion

Hygroscopicity of heat-treated wood, measured by the EMC, is shown as a function of mass loss in Fig. 1. As compared with the reference specimens, heat-treated wood clearly exhibits a reduction in hygroscopicity. At any setup temperature and relative humidity, the hygroscopicity decreases with increasing mass loss. Furthermore, as a function of mass loss, the data seem to form two different groups, specimens treated at intermediate relative humidity showing almost 2 % units lower EMC than specimens treated either in dry or water-saturated conditions. Therefore, hygroscopicity of heat-treated wood cannot be solely explained in terms of mass loss. Obviously, some phenomenon related to the relative humidity of the heating atmosphere must be involved.

(Figure 1 about here)

High temperature increases the mobility of molecular chains in the wood cell wall promoting structural rearrangements (Fahlén and Salmén 2003, Hakkou et al. 2005b). Water, as a softener, provides further flexibility to the cell wall and thus structural rearrangements are increased (Matsuda et al. 1994). Along with drying, this results in a tightly bonded structure in which some of the polar sites previously available for water sorption may not open upon rewetting (Crawshaw and Cameron 2000). Thus, some of the hydrogen bonds formed within the hemicelluloses and the amorphous regions of cellulose may be irreversible (Kato and Cameron 1999, Matsuda et al. 1994, Weise 1998). The consequences of the irreversible hydrogen bonding might well be denoted as hornification.

Hornification is generally associated with changes in moisture content being most pronounced at high temperatures (Crawshaw and Cameron 2000; Weise 1998). Intermediate relative humidity at the setup temperature was produced by placing dry specimens in the pressure vessel along with a predetermined amount of liquid water. Then, the water penetrated the specimens entering more readily the large pores and cavities, but also entering smaller pores along with time. Further on, along with increased temperature, the relative humidity within the vessel became reduced. Thus, some of the water that had entered the wood vaporized inducing a drying sequence in the disordered pore system of the cell walls.

High-temperature drying obviously did not happen in the case of specimens heated in water-saturated conditions. On the other hand, specimens treated in a dry atmosphere were not wetted during the experiment. Only those specimens treated at intermediate relative humidity experienced a wetting and high-temperature drying cycle during the experiment with the consequent formation of irreversible hydrogen bonds within the wood cell walls. This is likely to explain the lower hygroscopicity of such specimens, as shown in Fig. 1.

The experiments reported here were batch-experiments. In other words, all the constituents involved in the heat-bath treatment, *i.e.*, dry wood and water, were added at the beginning of the experiment and extracted at the end of the experiment. This induced a sequence of wetting and high-temperature drying in the case of the specimens treated at intermediate relative humidity. The authors hypothesize that intermediate relative humidity during a heat treatment probably would not result in reduced hygroscopicity if the relative humidity remained constant during the heat treatment. Such an experiment would require steaming of the specimens in the course of the temperature increment.

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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	*Relative	Isothermal	Loss of mass	EMC [%]
[°C]	humidity [%]	treatment [hours]	[%]	
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	1.5 (0.5)	8.2 (0.2)
		15	2.1 (0.7)	7.8 (0.3)
		15	2.8 (0.9)	7.3 (0.3)
		55	2.4 (0.4)	6.5 (0.2)
		50	3.2 (0.5)	6.2 (0.2)
		61	7.2 (0.5)	5.6 (0.1)
		100	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

Figure 1. Equilibrium moisture content (EMC) of heat-treated wood at 19 °C and 65 % relative humidity, plotted as a function of the mass loss for each heat-bath treatment. *Filled circles*, dry conditions at 150 °C; *open circles*, dry conditions at 170 °C; *filled squares*, intermediate relative humidity at 150 °C; *open squares*, intermediate relative humidity at 170 °C; *filled triangles*, water-saturated conditions at 150 °C; *open triangles*, water-saturated conditions at 170 °C; marker on the y-axis: mean value for reference wood

Abb. 1 Gleichgewichtsfeuchte (EMC) von wärmebehandeltem Holz bei 19°C und 65% relativer Feuchte in Abhängigkeit des Masseverlustes für jede Wärmebadbehandlung. *Ausgefüllte Kreise*: trockenes Klima, 150°C, *leere Kreise*: trockenes Klima, 170°C, *ausgefülltes Rechteck*: mittlere relative Luftfeuchte, 150°C, *leere Rechtecke*: mittlere relative Luftfeuchte, 170°C, *ausgefüllte Dreiecke*: wassergesättigt, 150°C, *offene Dreiecke*: wassergesättigt, 170°C. Markierung auf der y-Achse: Mittelwert für Kontrollprobe

