Effects of Cyclic Changes in Relative Humidity on the Sorption Hysteresis of Thermally Modified Spruce Wood

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Thermal modification of spruce wood (*Picea abies* L.) was conducted at three different temperatures (160, 200, and 240 °C) and treatment times (1, 3, and 5 h). The cyclic sorption experiments were performed for relative humidity changes of 30 to 85%. The equilibrium moisture content of the thermally modified wood was reduced up to 50% after treatment at 240 °C for 5 h. The sorption isotherms were described with the Guggenheim, Anderson, and De Boer (GAB) model. Cyclic sorption increased the monolayer capacity. Thus, the monolayer sorption was increased, while the multilayer sorption was limited. The dependence of the mass loss, hysteresis loop, and the maximum difference of equilibrium moisture content on the modification temperature and duration was modeled by response surface methodology. There was a very strong correlation between the modification temperature and mass loss, while the relationship between treatment time and mass loss was insignificant. The correlations between the modification parameters and the descriptors of sorption hysteresis were stronger after cyclic sorption. The sorption hysteresis decreased after cyclic sorption. This result was mainly caused by the increase of the monomolecular sorption for the adsorption processes.

Keywords: Equilibrium moisture content; Sorption isotherms; GAB model; Hysteresis loop; Cyclic sorption; Spruce wood (Picea abies L.)

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INTRODUCTION

Thermal modification is the simplest and cheapest way for improving wood dimensional stability. The stability improvement is primarily obtained from decreased equilibrium moisture due to the thermal decomposition of hemicelluloses, which are the most hydroscopic constituent of wood cell walls. The intensity of this degradation is strongly related to the treatment temperature (Alén *et al.* 2002; Kocaefe *et al.* 2008; Mitani and Barboutis 2014). Olek and Bonarski (2014) found that paracrystalline cellulose was additionally reorganized after the modification at 180 °C and its decomposition started only at 220 °C. Zauer *et al.* (2014) stated that the decrease in equilibrium moisture content was permanent, and thermally treated wood is more durable in correlation to its moisture content (Hill 2006; Esteves *et al.* 2007). Contrary information on the influence of thermal modification on sorption hysteresis has been reported. Hill (2006) stated that hysteresis is reduced after thermal treatment, whereas Militz (2002) found no effect. Olek *et al.* (2013) clearly demonstrated that the hysteresis coefficient increased with the modification temperature.

The hygroscopic properties of thermally modified wood depend on variations in air temperature and relative humidity. Hill *et al.* (2012) observed that the sorption isotherms of unmodified wood were not changed appreciably after subsequent sorption cycles, while those of thermally treated wood were largely affected by changes in relative air humidity. The sorption hysteresis of the thermally treated wood substantially decreased depending on the treatment condition parameters, *i.e.* modification temperature and time. Willems (2015) mentioned that sorption isotherms depend on sorption history and analyzed among others the influence of temperature on the isotherm shape change and therefore, on changes of the sorption hysteresis.

Previous studies performed on the influence of cyclic sorption on the properties of thermally treated wood were conducted over a very limited number of cycles. Some improvement of wood properties, such as dimensional stability, is expected after a long exposure of the material to moist air (*e.g.* Esteban *et al.* 2005; Kránitz *et al.* 2016). Hill *et al.* (2015) recommended that a minimum of two full sorption cycles be conducted to determine sorption isotherms. Esteban *et al.* (2005) tested the accelerated ageing of wood and observed reduced equilibrium moisture content for relative humidity values close to saturation. However, the investigated hygroscopic properties were determined for only three levels of relative humidity. Therefore, the objective of the present study was to determine and quantify the sorption properties, particularly the sorption hysteresis, of thermally modified wood. The hygroscopic properties were determined after long-lasting cyclic sorption experiments performed at various relative humidity values, as is typical for the in-service conditions of treated wood products.

EXPERIMENTAL

Hemicelluloses are the most hygroscopic components of wood, and their degradation starts in the early stages of thermal modification (Hill 2006; Esteves and Pereira 2009). Therefore, spruce (*Picea abies* L.) wood was selected due to its natural high hemicellulose content (Fengel and Wegener 1989). Green quarter-sawn spruce timber was sampled and air-dried to a moisture content of 12 to 14%. The cut sample dimensions were 35 mm thickness, 60 mm width, and 350 mm length; the samples were carefully dried to an oven-dry state to prevent hydrolysis of the material during the initial stage of thermal modification (Fengel and Wegener 1989; Kocaefe *et al.* 2008; Rautkari and Hill 2014). Nine of the ten samples were subjected to thermal treatment under laboratory conditions during which the air humidity was carefully controlled. The temperature and time of the thermal modification were carefully selected according to a two factor three level full factorial design of experiments to obtain a response surface statistical model (Box and Draper 2007). Three temperatures, *i.e.*, 160, 200, and 240 °C, and three durations, *i.e.*, 1, 3, and 5 h, were used during thermal treatments. Each modification process consisted of three phases. The initial phase of heating was made in moist air only, and superheated steam was added to the kiln after obtaining a temperature of *ca.* 130 °C. The heating phase was ended when the target temperature was obtained and the modification had proceeded. After completing the modification phase cooling started and it was firstly done with superheated steam and next in moist air only. Mass loss of the modified wood was determined in relation to the oven-dry mass of unmodified wood.

Each sample was cut into 12 sub-samples, each with dimensions of 1.5 mm (tangential) × 30 mm (radial) × 45 mm (longitudinal). Thus, 10 sets of samples were prepared for the combination of 3 target temperatures and 3 treatment times, as well as untreated wood, for an overall total of 120 sub-samples. The sub-samples were stored in desiccators and dried to constant mass over phosphorus pentoxide (P_2O_5). Half of the samples of each set were subjected to the cyclic sorption experiment at temperature of 20 ± 1 °C. The samples were alternately placed in containers above solutions of calcium chloride ($CaCl_2 \cdot 6H_2O$) and potassium chloride (KCl). It assured two different levels of relative humidity, *i.e.*, 30 and 85%, respectively. The range of relative humidity values corresponded to typical in-service outdoor conditions. The sorption experiment consisted of 90 cycles of desorption and adsorption phases. Each cycle lasted 2 days; thus, the duration of the cyclic sorption experiment was 6 months. After the cyclic sorption experiments were completed, 60 samples were again stored in desiccators over phosphorus pentoxide to obtain constant mass.

The experiments for determining sorption isotherms were performed at 22 ± 1 °C. The samples were first used to obtain adsorption and then desorption isotherms. The relative humidity of the air was controlled during the experiments by salt solutions (Majka and Olek 2007). All wood samples were weighed at least twice after obtaining the equilibrium value. After finishing the sorption experiments, the samples were placed in a laboratory dryer in order to obtain their oven-dry mass. The equilibrium moisture content was calculated for each relative humidity level as a mean value of 6 measurements.

RESULTS AND DISCUSSION

The measured values of the equilibrium moisture content were used to obtain adsorption and desorption isotherms. The Guggenheim, Anderson, and De Boer (GAB) model was used to parameterize the isotherms, as shown in Eq. 1 (Basu *et al.* 2006),

$$EMC = M_m \frac{K \cdot C \cdot RH}{\left(1 - K \cdot RH\right) \cdot \left(1 - K \cdot RH + C \cdot K \cdot RH\right)} \qquad (1)$$

where EMC is the equilibrium moisture content (kg water / kg dry matter), RH is the relative humidity, M_m is the monolayer capacity (kg water / kg dry matter), C is the equilibrium constant related to the monolayer sorption (also known as the Guggenheim constant), and K is the equilibrium constant related to the multilayer sorption. The model was selected in the present study as it explained the mechanism of the sorption phenomenon and the equation was characterized by high goodness of fit. The results of the fitting of the GAB model for wood before and after cyclic sorption are presented in Table 1.

The direct comparison of the isotherms provides important information on the influence of cyclic sorption of the wood-water system (Fig. 1). The study considered three target temperatures and three times of thermal treatment, as well as the untreated control, which yielded ten individual plots. Each plot within the sets was constructed for isotherm loops before and after cyclic sorption. Figure 1 shows that the shape of the sorption isotherms and the reduction of the equilibrium moisture content strongly depended on the temperature of heat treatment. Thermal modification reduced the equilibrium moisture content by about 50% for the highest temperature (240 °C) and the longest treatment time (5 h). This observation is consistent with the earlier findings of Metsä-Kortelainen *et al.* (2006) and Olek *et al.* (2013).

The influence of the treatment time was practically insignificant when analyzing sorption isotherms for a treatment temperature of 160 °C. However, increased temperature revealed its influence on the isotherms, *i.e.* mainly by the reduction of the sorption hysteresis.

The calculated *C* coefficient values of the GAB model were greater than 2 for all isotherms before and after cyclic sorption (Table 1). This finding implied that the isotherms could be classified as type II. Moreover, Lewicki (1997) proposed additional conditions for classifying isotherms as type II and for estimating the monolayer capacity (M_m) accurately. The relations given by Lewicki (1997), *i.e.* $5.57 < C < \infty$ and $0.24 < K < 1$, were also satisfied for all studied isotherms, and the isotherms were confirmed as type II. The monolayer capacity decreased as the treatment time and temperature increased, and consequently it was depending on the mass loss (*ML*) increased (Table 2). Furthermore, the monolayer capacity was always lower for adsorption *versus* desorption in the same treatment conditions.

For the desorption isotherms, the M_m coefficient did not significantly change after cyclic sorption. The maximum reported change in monolayer capacity was ± 0.003 kg/kg (Table 1), which probably preserved the shape of the desorption isotherms. The same analysis for the adsorption isotherms showed an increased M_m coefficient after cyclic sorption, *i.e.*, from 0.0066 to 0.015 kg/kg. This result suggested that cyclic sorption was responsible for increasing the monolayer sorption and limiting the multilayer sorption. It can be interpreted that cyclic sorption caused the reorganization of wood ultrastructure (Olek and Bonarski 2012), which was responsible for forming additional primary sorption sites. After the cyclic sorption the values of the *C* coefficient significantly increased for all adsorption isotherms (Table 1). Therefore, the cyclic sorption caused the increase of the strength of binding of water molecules to the primary sorption sites (Quirijns *et al.* 2005). This is consistent with the previous observation on the formation of the additional primary sorption sites and the increase of the equilibrium moisture content for the adsorption processes only. Simultaneously, the changes in the values of the *K* coefficient were insignificant after the cyclic sorption for both adsorption and desorption isotherms. Thus, the mobility of multilayer molecules is not influenced by the cyclic sorption.

The equilibrium moisture content changes due to cyclic sorption were observed for relative humidity values lower than 0.8 (Fig. 1) and for higher relative humidity values the changes were insignificant. This result was observed for both unmodified and thermally modified wood, as reported previously by Hill *et al.* (2012).

The observed changes in the equilibrium moisture content and sorption isotherms also affected the sorption hysteresis (Fig. 1). The fitted sorption isotherms were used to derive the descriptors for quantifying the hysteresis (*i.e.*, the hysteresis loop) (Popper *et al.* 2009), the maximum difference of equilibrium moisture content for desorption and adsorption, and the relative change of the hysteresis loop, as follows,

$$\delta H(\%) = \frac{H^{*} - H}{H} \times 100 \tag{2}$$

where H^* is the hysteresis loop for wood after cyclic sorption and *H* is the hysteresis loop for wood before cyclic sorption. The results of the mass loss (*ML*) and hysteresis quantification are presented in Table 2.

Table 1. Estimated Coefficients of the GAB Model of Thermally Treated Spruce Wood Before and After Cyclic Sorption

Treatment Option		Sorption Phase	Before Cyclic Sorption				After Cyclic Sorption			
Temp. (°C)	Time (h)		M_m	C	K	R^2	M_m	C	K	R^2
Unmodified		Adsorption	0.0473	8.16	0.864	0.996	0.0528	26.03	0.827	0.986
		Desorption	0.0832	8.34	0.727	0.999	0.0833	9.81	0.719	0.997
160	1	Adsorption	0.0443	8.35	0.871	0.997	0.0502	24.82	0.832	0.984
		Desorption	0.0821	8.07	0.721	0.999	0.0789	9.98	0.728	0.997
	3	Adsorption	0.0450	6.86	0.871	0.998	0.0518	25.04	0.826	0.986
		Desorption	0.0811	7.69	0.728	0.999	0.0823	9.55	0.716	0.997
	5	Adsorption	0.0413	7.73	0.876	0.998	0.0540	29.08	0.806	0.982
		Desorption	0.0811	8.14	0.709	0.999	0.0818	12.56	0.703	0.998
200	1	Adsorption	0.0384	7.21	0.873	0.998	0.0486	21.37	0.805	0.987
		Desorption	0.0783	7.81	0.693	0.999	0.0806	8.74	0.675	0.997
	3	Adsorption	0.0340	6.77	0.872	0.997	0.0488	20.68	0.767	0.989
		Desorption	0.0816	7.03	0.637	0.998	0.0847	8.66	0.605	0.997
	5	Adsorption	0.0332	6.58	0.873	0.998	0.0466	19.43	0.777	0.988
		Desorption	0.0779	7.34	0.645	0.998	0.0811	8.40	0.618	0.997
240	1	Adsorption	0.0279	6.13	0.864	0.996	0.0411	21.31	0.750	0.986
		Desorption	0.0709	7.29	0.598	0.998	0.0732	8.38	0.574	0.995
	3	Adsorption	0.0261	6.91	0.867	0.997	0.0390	23.95	0.752	0.986
		Desorption	0.0680	6.84	0.599	0.998	0.0695	8.74	0.574	0.996
	5	Adsorption	0.0246	6.59	0.859	0.997	0.0372	19.71	0.732	0.990
		Desorption	0.0647	7.13	0.575	0.998	0.0639	8.65	0.556	0.996

Table 2. Mass Loss (*ML*), Hysteresis Loop (*H*), Relative Change of Hysteresis Loop (*δH*) and the Maximum Difference of Equilibrium Moisture Content for Desorption and Adsorption (ΔEMC_{max}) with Corresponding Relative Humidity (*RH*) for Thermally Treated Wood Before and After Cyclic Sorption

Treatment Option		*ML* (kg/kg)	Before Cyclic Sorption			After Cyclic Sorption			*δH* (%)
Temp. (°C)	Time (h)		*H* (arb. units)	ΔEMC_{max} (kg/kg)	*RH* (-)	*H** (arb. units)	ΔEMC_{max}* (kg/kg)	*RH* (-)	
Unmodified			0.0264	0.039	0.79	0.0165	0.036	0.83	-37.5
160	1	0.002	0.0270	0.041	0.76	0.0165	0.037	0.84	-38.9
	3	0.013	0.0269	0.039	0.76	0.0164	0.036	0.83	-39.0
	5	0.006	0.0288	0.043	0.71	0.0164	0.035	0.83	-43.1
200	1	0.016	0.0282	0.042	0.73	0.0157	0.033	0.82	-44.3
	3	0.028	0.0293	0.044	0.73	0.0149	0.030	0.79	-49.1
	5	0.039	0.0288	0.043	0.73	0.0147	0.029	0.80	-49.0
240	1	0.071	0.0256	0.037	0.73	0.0117	0.024	0.79	-54.3
	3	0.097	0.0237	0.036	0.73	0.0110	0.022	0.78	-53.6
	5	0.129	0.0222	0.033	0.72	0.0090	0.018	0.75	-59.5

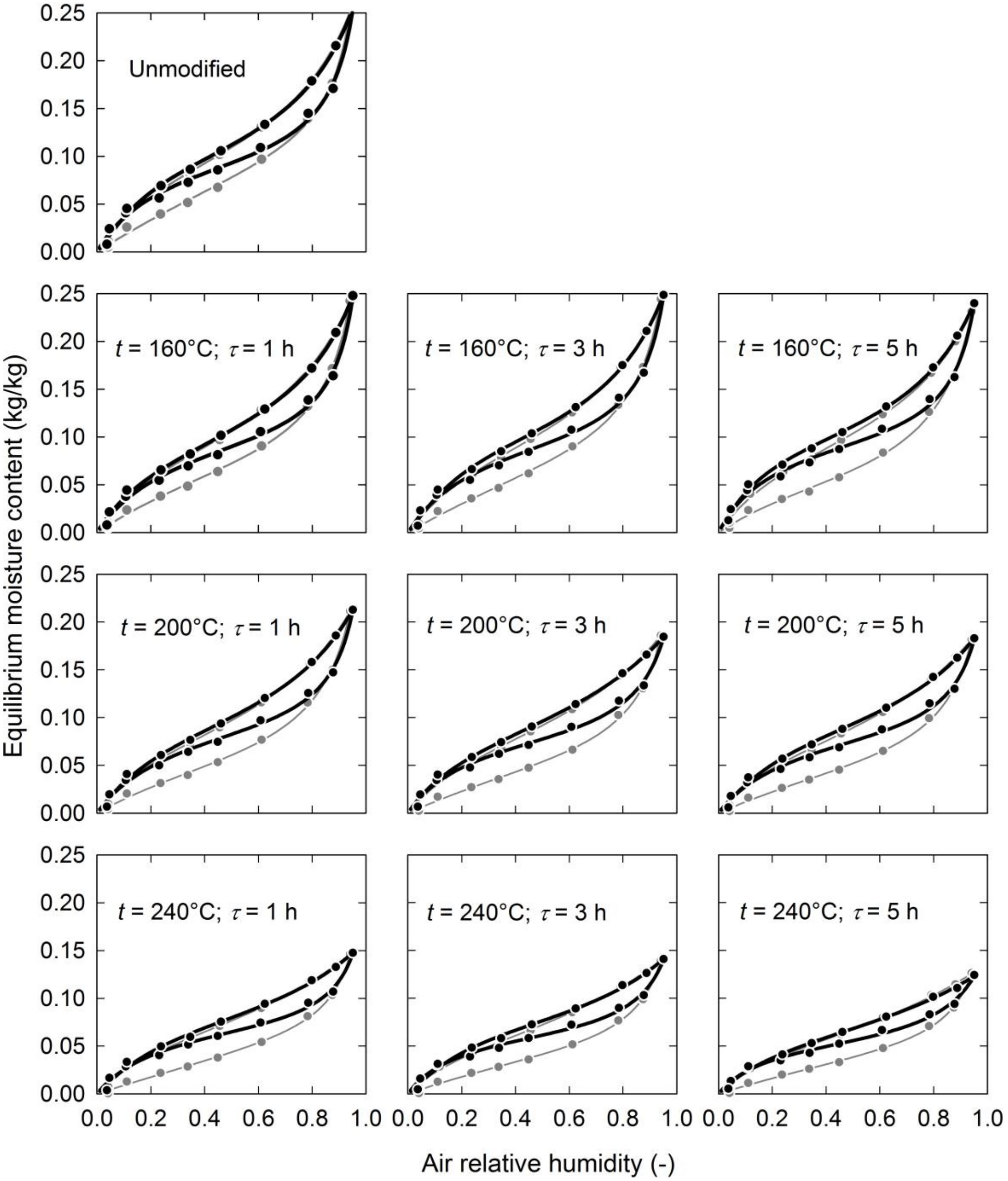

Fig. 1. The equilibrium moisture content (dots) and modeled sorption isotherms (curves) of thermally treated spruce wood. Black curves, after cyclic sorption; gray curves, before cyclic sorption

The dependence of the mass loss (*ML*), hysteresis loop (*H*), and the maximum difference of equilibrium moisture content (ΔEMC_{max}) on the modification temperature (t) and time (τ) was generalized *via* response surface methodology (Box and Draper 2007). A quadratic polynomial of the following form was fitted,

$$y = b_0 + b_1 t + b_2 \tau + b_{11} t^2 + b_{22} \tau^2 + b_{12} t \tau \quad (3)$$

where y is the predicted response (ML, H, or ΔEMC_{max}); b_0 is the y-intercept; and b_1, b_2, b_{11}, b_{22}, and b_{12} are the first-order, quadratic, and interaction effects, respectively, for treatment time (τ) and temperature (t). The results of the RSM modeling for ML, H, and ΔEMC_{max} are presented in Table 3. A stepwise regression approach was employed, and a backward elimination approach was used for excluding statistically insignificant model terms (Chatterjee and Hadi 2006) with the confidence interval of 0.95 (denoted as dashes in Table 3). The response surface model plot for mass loss is given in Fig. 2. Wood mass loss primarily depended on the temperature of thermal treatment, with much less dependence on treatment duration. The increase in thermal treatment temperature from 200 to 240 °C resulted in 3- to 4-fold higher mass loss depending on the treatment time. This result supported earlier observations on the influence of modification conditions on cellulose thermal decomposition (Poletto *et al.* 2012; Olek and Bonarski 2014).

Table 3. Estimated Coefficients of the Response Surface Methodology Model of Mass Loss (*ML*), Hysteresis Loop (*H*), and Maximum Difference of Equilibrium Moisture Content for Desorption and Adsorption (ΔEMC_{max})

Model Coefficients or Statistics	*ML* (kg/kg)	Before Cyclic Sorption		After Cyclic Sorption	
		H (arb. units)	ΔEMC_{max} (kg/kg)	*H** (arb. units)	ΔEMC_{max}* (kg/kg)
b_0	0.0276	0.0288	0.0429	0.0151	0.0307
b_1	0.0462	-0.0019	-0.0028	-0.0029	-0.0073
b_2	0.0143	–	–	-0.0006	-0.0021
b_{11}	0.0256	-0.0031	-0.0048	-0.0016	-0.0020
b_{12}	–	–	–	–	–
b_{22}	0.0135	-0.0013	-0.0015	-0.0007	-0.001
R^2	0.995	0.958	0.920	0.993	0.995
Adj R^2	0.990	0.933	0.871	0.986	0.991
Std error	0.0044	0.0006	0.0013	0.0003	0.0006

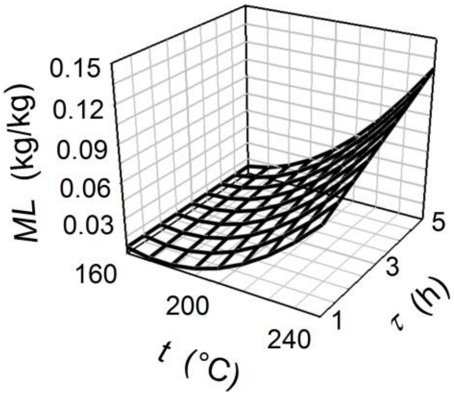

Fig. 2. Response surface model of mass loss as a function of treatment temperature and time

The treatment parameters had no clear influence on the hysteresis loop (H) observed for wood immediately after the thermal modification (Table 2). However, the estimated response model indicated that the lowest hysteresis loop was observed for the highest modification temperature (Fig. 3, right plot).

Cyclic sorption resulted in an appreciable reduction of the hysteresis loop for both untreated and thermally treated wood. This effect was quantified by the relative change in the hysteresis loop (δH), which is presented in Table 2. The fitted empirical model clearly showed that the hysteresis loop after cyclic sorption decreased with thermal treatment temperature but was relatively unaffected by treatment time (Fig. 3, left plot). A similar correlation was observed when analyzing the response surface model of the maximum difference of equilibrium moisture content (Fig. 4).

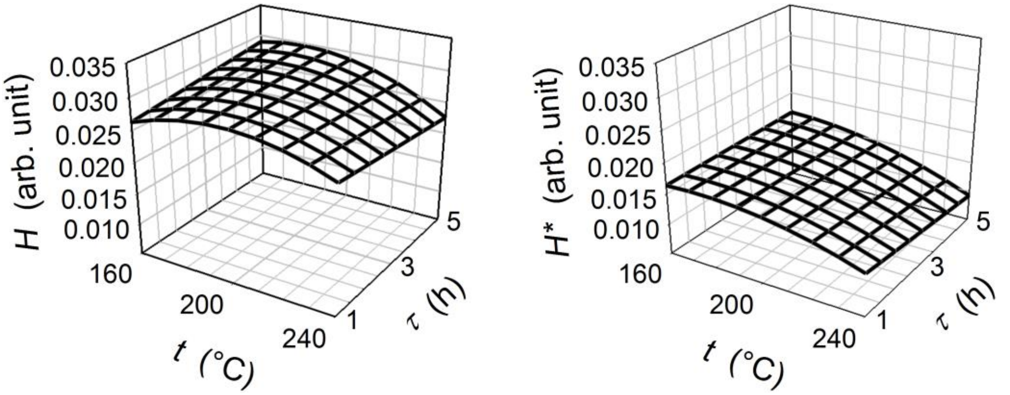

Fig. 3. Response surface model of hysteresis loop as a function of treatment temperature and time before (left) and after (right) cyclic sorption

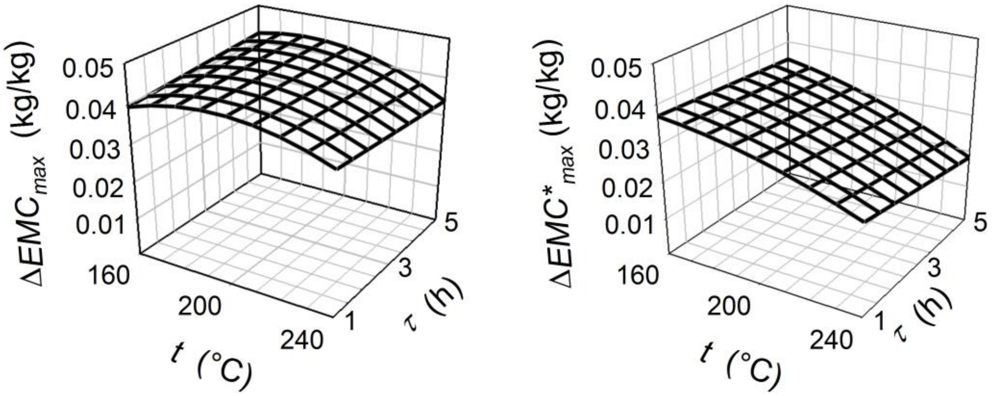

Fig. 4. Response surface model of maximum difference of equilibrium moisture content as a function of thermal treatment temperature and time before (left) and after (right) cyclic sorption

A correlation table (Table 4) was constructed to analyze the influence of the modification parameters on sorption hysteresis before and after cyclic sorption. A very strong correlation was found between the modification temperature and mass loss (*ML*), while the correlation between treatment time and mass loss was insignificant. The correlations between the modification parameters and the descriptors of the sorption hysteresis were stronger after the cyclic sorption. This observation lends support to the statement that the water sorption characteristics of the thermally modified spruce wood are finally established after the cyclic sorption; *i.e.*, the final wood-water relations are obtained after the in-service use of heat-treated wood products.

Table 4. Correlation between Descriptors of Sorption Hysteresis and Parameters of Thermal Modification

	Temp.	Time	*ML*	*H*	ΔEMC_{max}	*RH*	*H**	ΔEMC_{max}*	*RH**	*δH*
Temp.	1.00	0.00	**0.89**	-0.66	-0.65	-0.44	**-0.92**	**-0.95**	**-0.87**	**-0.94**
Time		1.00	0.27	-0.06	-0.04	-0.52	-0.20	-0.26	-0.36	-0.28
ML			1.00	**-0.87**	**-0.85**	-0.39	**-0.99**	**-0.98**	**-0.91**	**-0.93**
H				1.00	**0.98**	0.05	**0.86**	**0.76**	0.63	0.65
ΔEMC_{max}					1.00	0.03	**0.84**	**0.75**	0.62	0.64
RH						1.00	0.40	0.48	0.50	0.58
*H**							1.00	**0.99**	**0.91**	**0.95**
ΔEMC_{max}*								1.00	**0.94**	**0.98**
*RH**									1.00	**0.96**
δH										1.00

Note: Statistically significant correlations for the confidence interval of 0.95 are given in bold.

CONCLUSIONS

1. The thermal modification significantly reduced the equilibrium moisture content, and the extent of the reduction highly depended on the temperature of heat treatment.
2. The influence of the cyclic sorption on the properties was revealed by the applied set of descriptors. Most of the correlations between the parameters of the thermal modification and the sorption descriptors were significant after the cyclic sorption, which supports the conclusion that wood sorption properties strongly depend on the cyclic sorption.
3. The water sorption hysteresis decreased after the cyclic sorption in moist air; this was primarily caused by increased equilibrium moisture content during adsorption. This increase was due to the monomolecular sorption for the adsorption processes, as the monolayer capacity of the GAB model had unchanged desorption values but significantly increased for adsorption values.
4. The sorption properties of the thermally modified wood were not fixed immediately after the treatment. The properties depended on cyclic changes in relative humidity that are typical for the outdoor exposure of wood products. Therefore, the proper application of thermally modified wood requires information on the properties determined after a long-lasting cyclic sorption.

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