

## Analysis of Dimensional Stability of Thermally Modified Wood Affected by Re-Wetting Cycles

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The dimensional stability of thermally modified wood exposed to several wetting-drying cycles was analyzed. Specimens of dimensions 15×15 ×15 mm were thermally modified at 180 and 200 °C. The mass loss and chemical composition of the wood were determined in order to evaluate the effect and degree of modification. Afterwards, the radial, tangential, and volumetric swelling, anti-swelling efficiency, water absorption, water repellence efficiency, and mass loss due to wetting-drying cycles were determined and compared. The specimen's mass tended to decrease with each additional rewetting cycle. Additional extractives that were formed *via* thermal decomposition leached out during wetting cycles. Thermal modification positively affected the dimensional stability of all investigated species. The wood's swelling was reduced, a result attributed to hemicellulose degradation. Dimensional stability was improved by 24 to 30% following mild treatment and by 26 to 54% following more severe treatment. When specimens were exposed to six consecutive rewetting cycles, the swelling of the modified wood increased, whereas it slightly decreased for the control (hornification). The effective dimensional stability of thermally modified wood was reduced by 34 and 28.4% for beech, 47 and 19.6% for poplar, and 19.3 and 24.5% for spruce compared to the initial anti-swelling efficiency following the first wetting cycle.

*Keywords:* Thermal modification; Anti-swelling efficiency; Dimensional stability; Wetting-drying cycle; Chemical composition; Shape stability; Heat treatment

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### INTRODUCTION

Wood is a biodegradable, dimensionally unstable material. These fundamental properties of wood can cause problems when using certain wood products. Wood that is subjected to outdoor conditions, or even to humidity changes indoors, will shrink and swell, causing issues. Several common wood modification techniques are applied to fundamentally change the properties of wood (its bio-durability, dimensional stability, and others). Thermal modification of wood has long been recognized as a potential method to improve the natural properties of the wood and has been one of the most successful methods of modification for many years (Viitanen *et al.* 1994; Viitaniemi *et al.* 1997; Militz 2002; Hill 2006 and 2011; Čermák *et al.* 2014; Yan and Morrell 2014).

Heating wood to a relatively high temperature (160 to 260 °C) results in degradation associated with chemical changes in its structure (Syrjänen and Kangas 2000; Esteves and Pereira 2009). If the process is controlled carefully, the properties obtained following thermal modification can be tailored for further use of the wood in specific applications.

One of the most important characteristics of thermally modified wood is a decrease in its equilibrium moisture content (EMC) and, consequently, greater dimensional stability. Such reduction in the EMC has already been reported by Tiemann (1920), who showed that drying at high temperatures decreased the EMC of wood, inhibiting swelling and shrinking. The principal driver of the enhanced dimensional stability and reduced water absorption is likely a reduction in the number of hydroxyl groups of the hemicelluloses, as well as decreased accessibility of water molecules to cellulose hydroxyl groups due to the increase in cellulose crystallinity and cross-linking in lignin (Weiland and Guyonnet 2003; Hakkou *et al.* 2005; Boonstra and Tjeerdsma 2006). According to Militz (2002) and Tjeerdsma (1998), the dimensional stability of wood depends on the species, conditions of use, and the anatomical direction. Yildiz (2002) reported that the ASE reached 50% for beech wood treated at 200 °C. Tjeerdsma (1998) and Sailer *et al.* (2000) reported ASE values of about 40% for spruce and pine. Similar results were presented by Militz (2002) and Giebelier (1983), in which the ASE was determined to be 35 to 40% for pine and beech and 50% for poplar. Sailer *et al.* (2000) and Esteves *et al.* (2007; 2008) stated that the dimensional stability was greater in the tangential direction (by about 2.5%).

Nearly all of the thermally modified timber commercially available today is used for outdoor applications (*i.e.*, cladding, decking, flooring, garden furniture, and others). In such applications, wood-water interaction has always been the main issue. These products are commonly exposed to cyclic moisture conditions during seasonal changes and the issue of dimensional stability of modified wood in these types of environments is of fundamental importance. Unfortunately, studies of the dimensional stability of thermally modified wood exposed to cyclic moisture conditions are scarce (Rowell and Ellis 1978, Charani *et al.* 2007). The present study is therefore focused on the basic analysis of the dimensional stability of modified wood as affected by several wetting-drying cycles. The present study was conducted in order to evaluate material performance in such conditions.

## EXPERIMENTAL

### Materials

Beech (*Fagus sylvatica* L., 0.72 g/cm<sup>3</sup>), poplar (*Populus alba* L., 0.39 g/cm<sup>3</sup>), and spruce (*Picea abies* L. Karst., 0.41 g/cm<sup>3</sup>) wood obtained from Czech forest enterprises were studied. Specimens of dimensions 15×15×15 mm were cut from a single log with the growth rings parallel to two edges. Afterwards, the specimens were sorted into three groups (control, modified at 180 °C, and modified at 200 °C). Each group consisted of 6 specimens. Prior to treatment, the specimens were conditioned in a climate-controlled chamber at 65% relative humidity and 20 °C, resulting in initial moisture content (MC) of 12±2%.

### Methods

#### *Thermal modification*

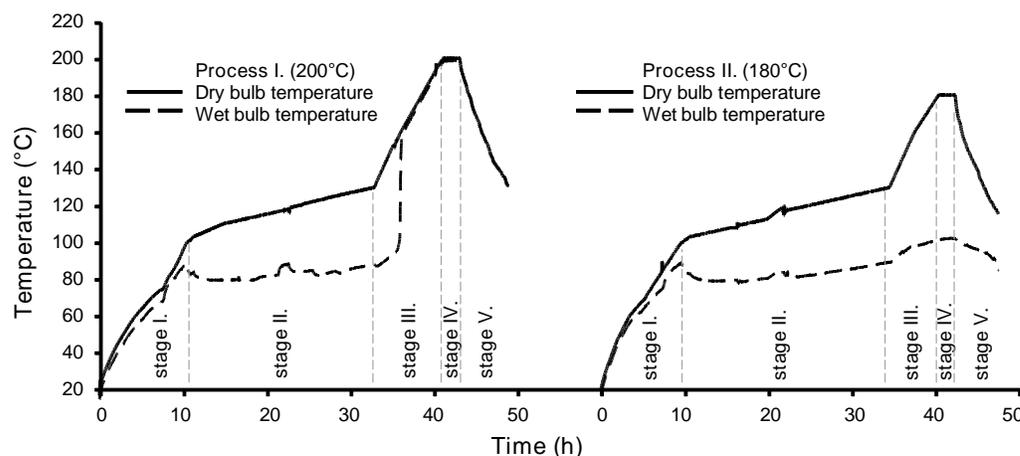
Thermal modification (TM) was carried out using a small-scale laboratory heat-treatment chamber (Katres spol. s r.o., CZ) at 180 or 200 °C. The schedule of the five-stage thermal modification process throughout 50 h was achieved with a temperature control system as shown in Fig. 1. In additional Fig. 1 shows information about wet-and-dry-bulb thermometer, consists of two thermometers, one that is dry and one that is kept moist with

water on a sock. Values gives an indication of atmospheric humidity i.e. psychrometer difference.

The maximum temperatures (180 or 200 °C) were maintained for 3 h. The mass loss (ML) of thermally modified specimens was determined immediately after the end of the modification process in order to evaluate the degree of the modification. The ML of the modified specimens was calculated as follows,

$$ML (\%) = (m_{u,O.D.} - m_{m,O.D.}) / m_0 \cdot 100 \quad (1)$$

where  $m_{m,O.D.}$  is the oven-dry mass of the specimens following thermal modification and  $m_{u,O.D.}$  is the oven-dry mass of the specimens prior to thermal modification.



**Fig. 1.** Thermal modification processes at 200 and 180°C, including the dry-bulb and wet-bulb temperatures and stages

### Chemical analysis

The chemical composition (cellulose, hemicelluloses, and lignin) of the specimens was determined. Around 200 mg of sample was pre-hydrolyzed with 2 mL of 72% H<sub>2</sub>SO<sub>4</sub> (30 °C, 1 h). The reaction mixture was diluted with 56 mL additional ultra-pure water, and post-hydrolysis was performed in an autoclave at 120 °C and 1.2 bar for 30 min. For the HPEAC borate analysis, wood sugars were separated in a 5.6 mm bore column 115 mm in length (omnifit) filled with strong anion exchange resin 114 (MCL gel CA08F (Mitsubishi) at 60 °C). The mobile phase (0.7 mL/min) consisted of A 0.3 M potassium borate buffer with pH 9.2 (A) and 0.9 M potassium borate buffer with pH 9.5 (B). After the samples were injected, separation started with 90% (A) and 10% (B). A linear gradient was run to transition the mobile phase to 10% (A) and 90% (B) over the course of 35 min. Data acquisition ceased after 50 min. For quantification a post-column derivatization of monosaccharides with Cu-bichinoninate (0.35 mL min<sup>-1</sup>) was applied. The reaction was performed at 105 °C in a 30 m crocheted Teflon coil of 0.3 mm inner diameter. This enabled the subsequent detection of sugars at 560 nm (Sinner et al. 1975; Sinner and Puls 1978). Data were processed using Dionex Chromeleon software.

The extractives content was also determined. Ground samples were extracted using a fexIKA vario control apparatus. Dried specimens were milled to pass through a 0.5-mm sieve. 10±0.01 g of each sample was extracted using 100 mL of a mixture of methanol and distilled water (1:1, v/v) at 110 °C for 1.5 h for 4 extraction cycles. After the extraction process, the extracts were diluted to 100 mL and analyzed.

*Wetting-drying cycles and dimensional stability*

The dimensional stability was evaluated using a wetting-drying test until the difference between the previous two measurements was less than 1% (6 cycles). Selected specimens were submerged in water at 20 °C for 24 h for each cycle. The radial, tangential, and longitudinal dimensions and weights of the saturated specimens were determined. After wetting, the specimens were oven-dried to 0% MC in three phases (to prevent cracks from occurring). Phase (1) involved heating at 40 °C for 24 h; Phase (2), at 60 °C for 12 h; and Phase (3), at 103 °C for 12 h. When 0% MC was reached, the specimen dimensions (to within 0.01 mm) and weights (to within 0.001 g) were determined. The entire wetting-drying cycle was repeated 6 times.

The radial ( $S_R$ ), tangential ( $S_T$ ), and volumetric ( $S_V$ ) swelling, anti-swelling efficiency ( $ASE$ ), water absorption ( $WA$ ), and water repellence efficiency ( $WRE$ ) were calculated after each cycle. The swelling ( $S$ ) was determined as follows,

$$S_{R,T,V} (\%) = (V_1 - V_0) / V_0 \cdot 100 \quad (2)$$

where  $S$  is the radial, tangential, or volumetric swelling,  $V_1$  is the radial, tangential, or volumetric dimension of the specimen after soaking, and  $V_0$  is the oven-dry dimension before submersion. The  $ASE$  was then calculated as follows,

$$ASE_V (\%) = (S_u - S_m) / S_u \cdot 100 \quad (3)$$

where  $ASE_V$  is the volumetric anti-swelling efficiency,  $S_u$  is the swelling of control (untreated) wood, and  $S_m$  is the swelling of modified wood. Water absorption was calculated as follows,

$$WA (\%) = (W_W - W_{O.D.}) / W_{O.D.} \cdot 100 \quad (4)$$

where  $WA$  is the water absorption of the wood,  $W_W$  is weight of the wet specimen, and  $W_{O.D.}$  is the weight of the oven-dried specimen. The water repellence efficiency was then calculated as follows,

$$WRE (\%) = (WA_u - WA_m) / WA_u \cdot 100 \quad (5)$$

where  $WRE$  is the water repellence efficiency,  $WA_u$  is the water absorption of the control (untreated) specimens, and  $WA_m$  is the water absorption of the modified specimens.

## RESULTS AND DISCUSSION

### Mass Loss and Chemical Changes due to Thermal Modification

The mass loss behavior of wood is one of the most important parameters to consider when applying any thermal modification technology. It is considered an indicator of the degree of modification achieved and of the quality of the modification (Militz 2002; Hill 2006; Esteves and Pereira 2009). The mass loss (ML), density, and contents of cellulose, hemicelluloses, lignin, and extractives are shown in Table 1.

The ML generally depends on the wood species, heating medium, temperature, and time, with temperature having the most dramatic effect. As expected, higher ML was observed as more severe treatments were applied (200 °C), whereas significant differences were found for beech (6.9%) and poplar wood (10.4%) compared to spruce wood (4.5%). It is possible that the different structures and chemical compositions of hardwoods and softwoods (*i.e.*, higher hemicelluloses and extractives content in hardwoods) caused these discrepancies because extractives and hemicelluloses are susceptible to thermal decomposition (Tjeerdsma 1996).

**Table 1.** Average Mass Loss and Amount of Cellulose (CL), Hemicelluloses (HL), Lignin (L), and Extractives (E) in the Investigated Species

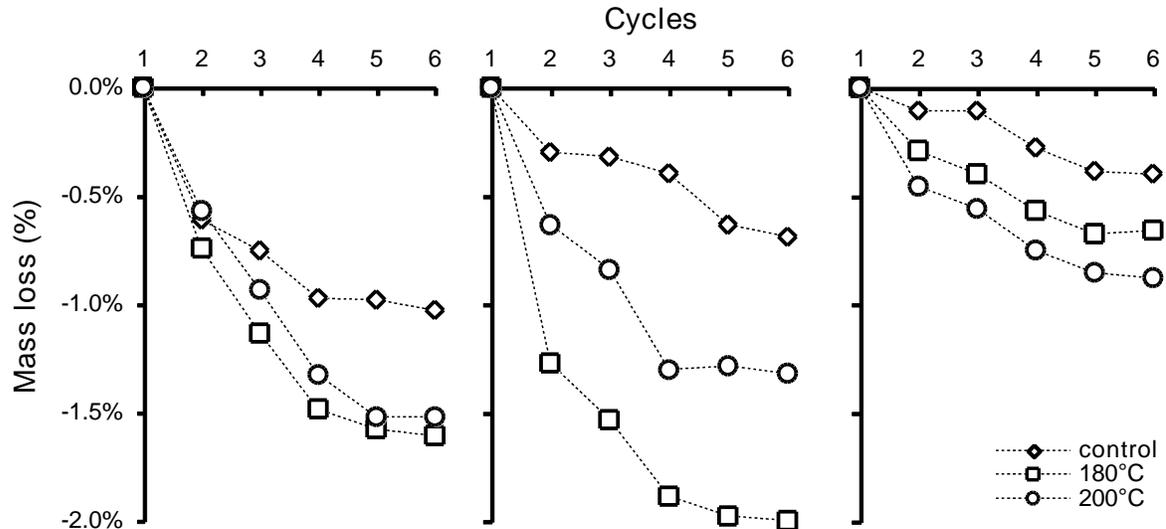
	Mass Loss (%)	Density (kg/m <sup>3</sup> )	CL (%)	HL (%)	L (%)	E (%)
<b>Beech</b>	–	723	41.0	22.5	34.2	2.2
<b>B 180°C</b>	3.8	695	42.4	22.4	31.2	4.1
<b>B 200°C</b>	6.9	673	44.9	16.4	29.0	9.7
<b>Poplar</b>	–	395	53.5	16.2	27.9	2.4
<b>P 180°C</b>	3.2	382	51.5	15.6	27.9	5.0
<b>P 200°C</b>	10.4	354	50.4	8.3	32.4	8.9
<b>Spruce</b>	–	405	46.6	20.6	31.4	1.4
<b>S 180°C</b>	2.8	394	46.5	18.4	31.9	3.2
<b>S 200°C</b>	4.5	387	48.6	16.3	30.9	4.3

Most of the data (mass loss and chemical compounds content) were difficult to compare to previously published figures because of the different thermal modification processes, conditions (heating medium, temperature, and time), and wood species used. Hakkou *et al.* (2005) reported 4, 2.5, and 5% mass loss following TM at 180 °C and 7, 7.5, and 8% mass loss after TM at 200 °C for beech, poplar, and spruce, respectively. Alén *et al.* (2002) observed 1.5% ML in spruce thermally modified at 180 °C for 4 h. Based on a patent by Vittaniemi *et al.* (1997), at least 4% ML should be achieved to improve the dimensional stability of wood. The results of the present study are in general agreement with those of previously published studies (Zaman *et al.* 2000; Alén *et al.* 2002; Weiland and Guyonnet 2003; Rautkari and Hill 2014).

### Mass Loss and Dimensional Stability after Re-wetting Cycles

An average mass loss during rewetting cycles was determined after each cycle, as shown in Fig. 2, based on oven-dry weights. The specimens tended to decrease in mass with each incremental cycle. After six wetting-drying cycles, the initial mass decreased differently for each of the species studied. The highest mass losses were 1.5 and 2% for beech and poplar, respectively, and only 0.5 to 0.7% for spruce.

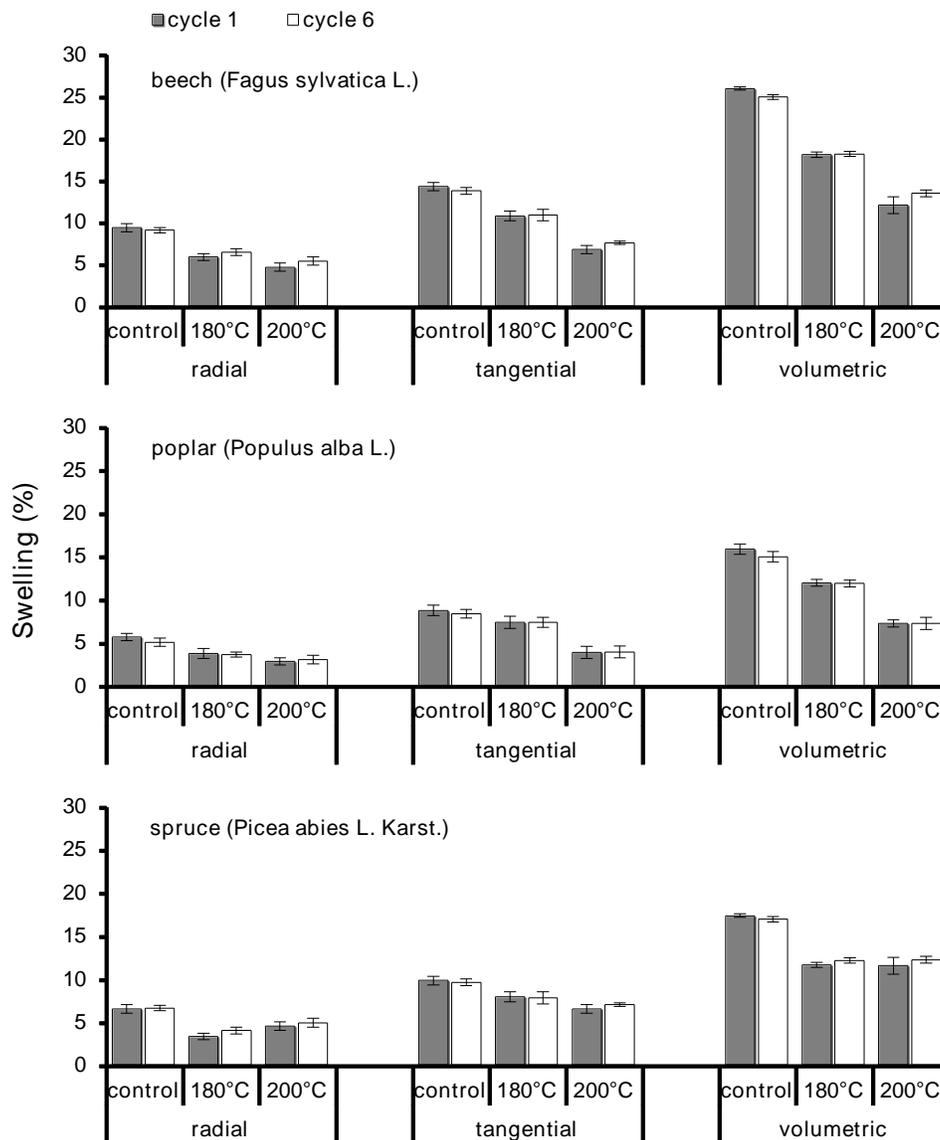
Thermal modification led to an increase in extractives content (Table 1), which can leach out during wetting-drying cycles, resulting in decreased dry mass. The extractives content increased with treatment temperature, so the leachable content was expected to be higher at higher temperatures. The highest mass losses occurred following mild treatment.



**Fig. 2.** Mass loss due to wetting-drying cycles exposure for control and thermally modified wood

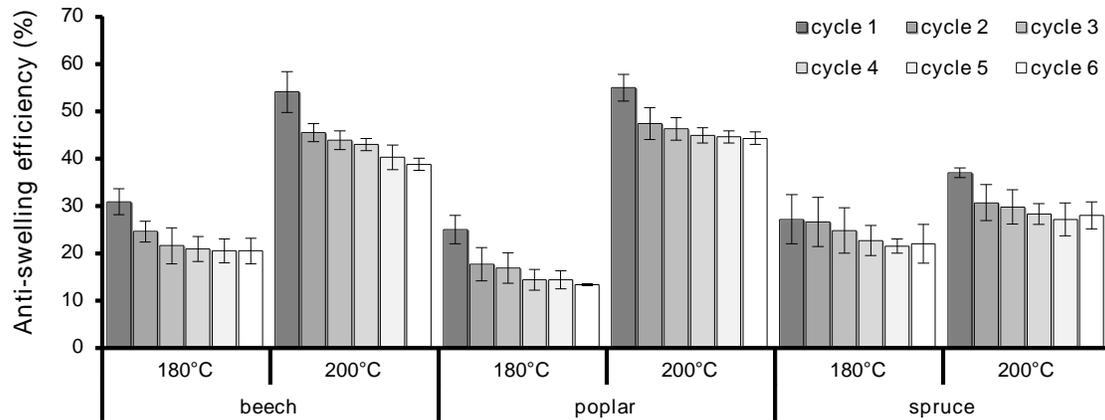
Furthermore, the radial, tangential, and volumetric swelling of the investigated species, exposed to six wetting-drying cycles, are shown in Fig. 3. The control (untreated) specimens had greater radial, tangential, and volumetric swelling than the thermally modified specimens. Certain thermal modification processes decreased swelling *via* the degradation of the hemicelluloses with the -OH groups primarily responsible for the highly hygroscopic behaviour of wood. Burmester (1975) concluded that thermal modification of wood results in significant reduction of hemicelluloses content, thus improving the dimensional stability of the wood.

After exposure to 6 wetting-drying cycles, the swelling of control specimens decreased slightly (2.1 to 3%), whereas that of thermally modified specimens increased (3.6 and 7.9% for 180 and 200 °C, respectively). The reduction of swelling in the control specimens was likely due to hornification (Matsuda *et al.* 1994; Park *et al.* 2006; Rautkari *et al.* 2013). Hornification results in a tightly bound fiber structure in which some of the polar sites previously available for water sorption do not open upon rewetting, decreasing -OH group accessibility (Scallan 1977; Crawshaw and Cameron 2000; Esteban *et al.* 2005; Rautkari *et al.* 2013; Pönni *et al.* 2014). Irreversible hydrogen bonding decreases the capability of water sorption into wood fibers (hygroscopicity). According to Weise (1998) and Crawshaw and Cameron (2000), hornification occurs progressively with additional drying cycles. The higher swelling values after several rewetting cycles can be explained by the formation of extractives, which can leach out during soaking cycles, during thermal decomposition (Table 1).



**Fig. 3.** The changes in the radial, tangential, and volumetric swelling of investigated species after first and sixth wetting cycle

Even though several authors (Tjeerdsma *et al.* 1998; Esteves *et al.* 2007; Esteves *et al.* 2008) mentioned that swelling values are superior in the tangential direction, the present study did not reach such a conclusion. The severe treatment at 200 °C confirmed such results: the ratio of tangential-to-radial swelling was 1.52, 1.66, and 1.45 for the control specimens and 1.42, 1.30, and 1.43 for thermally modified beech, poplar, and spruce specimens, respectively. On the other hand, the mild treatment yielded ratios of 1.69, 2.03, and 1.95. Despite the more substantial dimensional changes following treatment at 200 °C, swelling anisotropy still remained in the thermally modified species investigated (Sailer *et al.* 2000; Esteves *et al.* 2008).

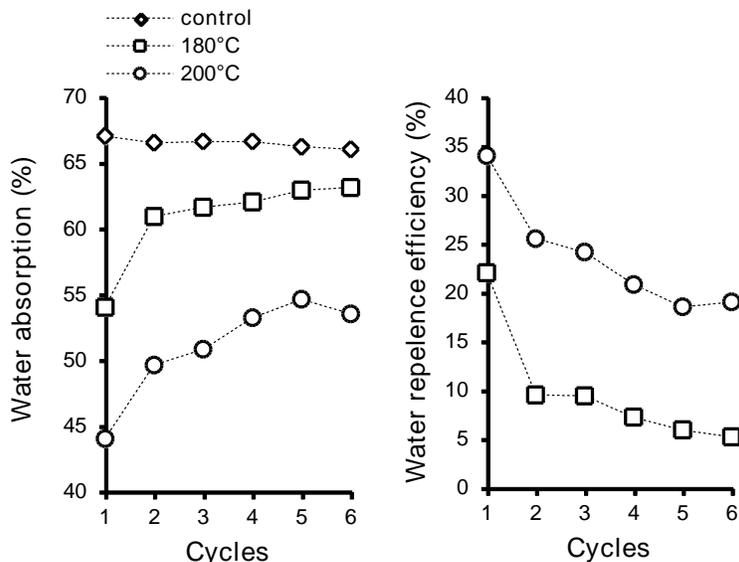


**Fig. 4.** Changes in the anti-swelling efficiency (ASE) of investigated species after six wetting-drying cycles

The anti-swelling efficiency (ASE) and water repellence efficiency (WRE) are the most common methods used to evaluate the dimensional stability of modified wood. For each specimen, ASE values were calculated for each wetting-drying cycle, as shown in Fig. 4. The dimensional stability improved in the species studied. The ASE was approximately 24 to 30% following the milder treatment and 36 to 54% following the more severe treatment. The differences between the applied treatment temperatures were determined to be approximately 23 to 30% of the ASE, except in the case of spruce wood, for which the treatment temperature did not noticeably affect the ASE (only a 9.5% improvement). There were only slight variations in mass loss after both treatments. According to Fengel and Wegener (1989), hardwoods are less thermally stable than softwoods, which may explain why the ASE values of beech and poplar increased to greater levels than that of spruce.

The ASE values declined as the specimens were exposed to six consecutive soaking cycles. The effective dimensional stability of the thermally modified wood was reduced by 34 and 28.4% for beech, 47 and 19.6% for poplar, and 19.3 and 24.5% for spruce, respectively, after pretreatments at 180 and 200 °C, compared to the initial ASE after the first soaking cycle. The highest decrease was observed after the first soaking cycle (an average of 17%). Moreover, the ASE decreased slowly and stabilized after five cycles. This decline was because some of the newly formed extractives leached out from the wood during rewetting, making more cell wall spaces accessible to water, which increases the capacity for water bonds in the cell wall.

The water absorption of thermally modified wood decreased compared to that of the control specimens. Figure 5 shows an average water absorption of the control and thermally modified beech wood samples during the wetting cycles. The WA of the control specimens decreased slightly (by 1%), while the TM increased with each incremental cycle. As wood is exposed to severe conditions such as high temperatures or outdoor dry-wet cycles, cracks can be formed (Tjeerdsma *et al.* 1998; Rapp and Sailer 2004). With increasingly severe conditions and greater potential for crack formation, water absorption increases. According to the water absorption test results, the WRE decreased from 22 to 6% following treatment at 180 °C and from 35% to 20% following treatment at 200 °C.



**Fig. 5.** Effect of wetting-drying cycles on water absorption and water repellency efficiency (WRE) of beech wood (control, modified at 180 and 200 °C)

## CONCLUSIONS

1. Thermal modification led to significant chemical changes, resulting in 2.8 to 10.4% of mass loss (species depended). Hemicelluloses were the most degraded wood component, while amount of extractives increased.
2. Thermal modification significantly improved the dimensional stability of the wood. The radial, tangential, and volumetric swelling of modified wood decreased, resulting in 24 to 30% ASE following mild treatment (180 °C) and 36 to 54% following severe treatment (200 °C).
3. When specimens were exposed to several rewetting cycles, the initial dimensional stability of the thermally modified wood was reduced by 34 and 28.4% for beech, 47 and 19.6% for poplar, and 19.3 and 24.5% for spruce for 180 °C and 200 °C, respectively.
4. Dimensional stability reduction following numerous soaking cycles can be partly attributed to the formation of leachable extractives within the thermally modified wood as a result of thermal degradation. This leaching rendered new regions within the cell wall accessible to water molecules.
5. The control specimens underwent lesser swelling during rewetting cycles, which is likely due to hornification. This resulted in a reduction of -OH group accessibility after repeated soaking cycles.
6. Although some of the phenomena observed in this study were explained, more detailed analysis of the -OH group accessibility of thermally modified wood exposed to cyclic conditioning should be done in the future.

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