Application of Microwave Heating for Acetylation of Beech (*Fagus sylvatica* L.) and Poplar (*Populus* hybrids) Wood

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Microwave and conventional acetylation of wood was carried out to determine its efficacy on the material properties. Beech (*Fagus sylvatica* L.) and poplar (*Populus* hybrids) samples with dimensions 14 mm × 14 mm × 14 mm were impregnated using acetic anhydride, and chemical reactions were initiated by microwave and conventional heating. The microwave acetylation process was carried out using laboratory equipment at a frequency of 2.45 GHz in several testing modes to reduce time of the reaction. The uptake of substance, equilibrium moisture content, wood swelling, and dimensional stability were determined in order to evaluate the efficacy and degree of acetylation. Both microwave and conventional heating positively affected the selected material properties. The results showed that no significant differences were found between microwave and conventional heating; therefore, microwave heating can be used as a valid replacement in the acetylation process. Microwave power of 2 kW and 0.1 $m \cdot min^{-1}$ conveyor speed were the optimum conditions for microwave acetylation. These process parameters resulted in 39.4% ASE_T and 35.2% ASE_R for beech and 38.0% ASE_T and 16.3% ASE_R for poplar samples. This work provides insight into the details of wood acetylation using microwave heating.

Keywords: Acetic anhydride; Chemical reactions; Dimensional stability; Wood impregnation; Microwave treatment; Wood modification

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INTRODUCTION

Wood, as an important renewable resource, has a broad range of material properties for various applications, *e.g.*, relatively high strength and stiffness, low specific weight, natural appearance with interesting texture, insulation properties, and machinability (Kollmann 1951; Stamm 1964; Wagenführ 2000; Skodras *et al.* 2004). However, natural wood also has undesirable properties that might limit the range of feasible applications. Wood is a natural heterogeneous composite and is considered to be dimensionally unstable when exposed to wet conditions (Kumar 1957; Rowell 1983; Skaar 1988; Hunter 1995). Hydroxyl (-OH) groups of hemicellulose and cellulose chains are mainly responsible for the highly hygroscopic behaviour of wood (Stamm 1964; Rowell 1983). The dimensional instability of wood under different moisture/humidity conditions is considered a major drawback of wood performance (Stamm 1964; Hill and Jones 1996; Homan and Jorissen 2004; Popescu *et al.* 2013).

Wood modification techniques can be applied in order to improve certain wood properties, *e.g.*, bio-durability, dimensional stability, colour, wettability, *etc.* (Hill and Jones 1996; Militz 2002; Rowel 2005; Hill 2006; Čermák *et al.* 2015). Chemical modification can, for instance, be used as an efficient way to transform hydrophilic OH groups into larger hydrophobic groups (Kollmann 1951; Rowel 1983; Skaar 1988; Bodîrlău *et al.* 2009). Little to no water can penetrate the permanently swollen cell wall of wood as a result of chemical treatment (Homan and Jorissen 2004).

Acetylation of wood is one of the most commonly used chemical treatments to improve the dimensional stability and biological durability of wood (Tarkow *et al.* 1950; Larsson and Simonsons 1994; Popescu *et al.* 2013). Moreover, this treatment retains to wood its original colour and improves acoustical, dielectric, and strength properties (Tarkow *et al.* 1950; Dreher *et al*.1964, Homan *et al.* 2000).

Acetylation effectively changes free hydroxyls within the wood into acetyl groups (Rowel 1983; Hill and Jones 1996; Rowell *et al.* 2013). This is done by reacting wood with acetic anhydride (Ac_2O) (Militz 1991; Sander *et al.* 2003). The standard acetylation process includes impregnation of oven-dried wood with Ac_2O, followed by conventional heating to initiate the chemical reactions with wood polymers (Bongers and Beckers 2003). Acetic acid is then released as a by-product of the reactions (Homan and Jorissen 2004). Application of the Ac_2O without a catalyst or cosolvent is the preferred method for wood acetylation (Rowell 2013). Time consumption is an important issue for the proposed acetylation method (Yang *et al.* 2014).

In order to reduce the reaction time and make the process more effective, an innovative wood acetylation process that uses microwave (MW) energy has been recently studied (Larsson *et al.* 1999; Larsson and Simonson 1999; Larsson 2002; Li *et al.* 2009; Diop *et al.* 2011; Yang *et al.* 2014). The application of microwave energy rapidly heats the material throughout the whole cross-section using the dielectric properties of wood (Torgovnikov 1993; Larsson *et al.* 1999) instead of commonly used convection and conduction heat flux (Koskiniemi *et al.* 2013). The principle behind microwave heating is based on the polar characteristic of molecules and their ability to absorb and transform microwave radiation into heat (Metaxas and Meredith 1983; Torgovnikov 1993; Hansson and Antti 2003). Permanent dipoles of molecules begin to move with the same frequency as the electromagnetic field. Therefore, rapid changes in the field polarity cause vibration and rotation of molecules, which transforms the microwave energy into frictional heat (Makovíny 2000; Hansson and Antti 2003; Dömény *et al.* 2014). The polarizability of the Ac_2O in the MW field has been extensively studied (Baghurst and Mingos 1992; Larsson *et al.* 1999). Baghurst and Mingos (1992) stated that during MW heating, the temperature rise of the acetic anhydride (Ac_2O) was about two times higher than water (H_2O).

Unfortunately, published studies related to the acetylation process conducted using MW heating are still limited. Therefore, the present study aims to (1) analyze the acetylation process using MW heating, (2) evaluate the efficacy of MW heating on the chemical reactions during the process and its similarities with conventional methods, and (3) evaluate material properties (uptake of substances, equilibrium moisture content, wood swelling, and anti-swelling efficiency). This work should provide a better insight into details of the wood acetylation.

EXPERIMENTAL

Materials

Beech (*Fagus sylvatica* L.) and poplar (*Populus hybrids*) sapwood were studied. Samples with dimensions 14 mm × 14 mm × 14 mm were oven dried to 0% moisture content (MC), according to EN 13183-1 (2002). The average oven-dry density (ρ_0) of testing samples was 694 kg·m^{-3} for beech and 316 kg·m^{-3} for poplar. Afterward, samples were sorted into groups of 10 for each species and acetylation treatment (Table 1).

Table 1. List of Treatments and Process Parameters

Material	No. of samples	Treatment	Mode	Speed of Conveyor	Time
Beech/Poplar	10/10	Control	-	-	-
Beech/Poplar	10/10	MW I	1.5 kW	0.1 m·min^{-1}	15 min
Beech/Poplar	10/10	MW II	2.0 kW	0.1 m·min^{-1}	15 min
Beech/Poplar	10/10	MW III	2.0 kW	0.025 m·min^{-1}	60 min
Beech/Poplar	10/10	Conventional Heat	100 °C	-	60 min

Methods

Acetylation process

Prior to treatment, samples were pressure impregnated with Ac_2O (Sigma-Aldrich, analytical grade ≥ 99%) in a laboratory plant JHP1-0072 (Fig. 1).

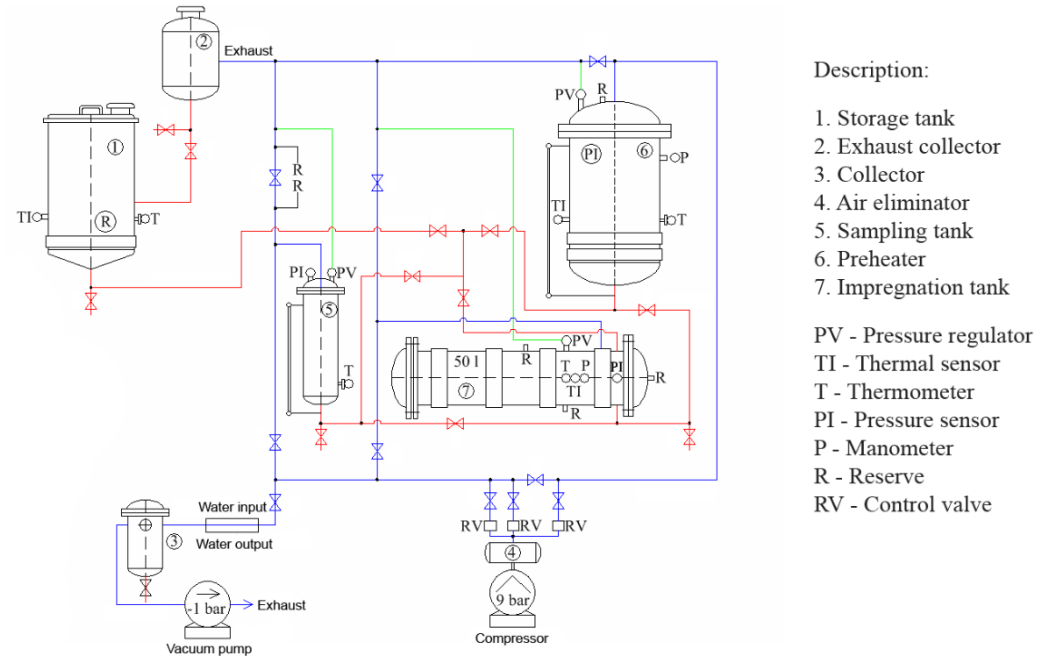


Fig. 1. Scheme of vacuum – pressure impregnation equipment

Wood species were impregnated separately using same process parameters, *i.e.*, 0.8 MPa pressure for 120 min at 20 °C. Weight percentage gain (WPG) and retention (R)

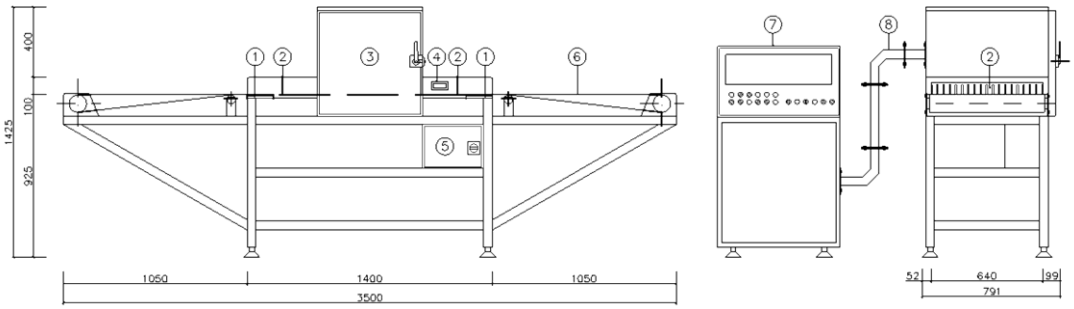
values were used as an indicator of substance uptake according to the following formulas (Eqs. 1 and 2),

$$WPG\ (\%) = (m_2 - m_1)/ m_1 \quad (1)$$

$$R\ (kg \cdot m^{-3}) = (m_2 - m_1)/V \quad (2)$$

where m_1 is the sample weight before impregnation, m_2 is the sample weight after impregnation, and V is volume of the sample.

Afterward, MW and conventional heating were used to induce chemical reactions taking place during the acetylation process (Table 1). A continuous laboratory microwave device (Fig. 2) that operates at a frequency of 2.45 GHz with adjustable power from 0.6 to 5 kW was used for the MW heating. Conventional heating was carried out in a standard laboratory drying oven (Sanyo MOV 112).



Description: 1. Duced ceramics 2. Flexible copper plate 3. Modification chamber 4. Speed control panel 5. Power button 6. Conveyor 7. Microwave generator with control panel 8. Waveguide

Fig. 2. Scheme of continuous microwave device

Once chemical reactions took place, the testing samples were placed into the impregnation plant in vacuum at 10 kPa for 60 min to eliminate the residual acetic anhydride and acetic acid from wood structure.

Uptake of the substance was determined in two steps: after the impregnation of testing samples and after the chemical reaction when residuals were eliminated.

Surface temperature

The surface temperature of the testing samples was measured by a contactless infrared thermometer (Voltcraft IR-380, accuracy ±1.0 °C) during a short interruption of the chemical reactions (turning off the power and opening the modification chamber for a few seconds) for both methods of heating. Measurement was done in the middle of the reaction time.

Equilibrium moisture content and Dimensional stability

The samples were conditioned in a climate chamber (Sanyo MTH 2400) at different relative humidities (30%, 65%, and 99%) at 20 °C until the equilibrium moisture content (EMC) was reached for certain conditions. Afterward, the moisture content was determined using the oven-dry method according to EN 13183-1 (2002).

Dimensional stability in radial and tangential directions was determined by estimating wood swelling ($S_{T,R}$) and anti-swelling efficiency ($ASE_{T,R}$). The swelling was calculated in dry and equilibrium states at the relative humidity (RH) under study. ASE

represents difference between the swelling of the treated and untreated wood. $S_{T,R}$ and $ASE_{T,R}$ were calculated according to the following formulas,

$$S_{T,R}\ (\%) = 100\ (D_{T,R,2} - D_{T,R,1})/\ D_{T,R,1} \quad (3)$$

$$ASE_{T,R}\ (\%) = 100\ (S_{T,R,u} - S_{T,R,t})/S_{T,R,u} \quad (4)$$

where $D_{T,R,1}$ is the radial or tangential dimension of the oven-dried sample, $D_{T,R,2}$ is the radial or tangential dimension of the conditioned sample, and $S_{T,R,u}$ and $S_{T,R,t}$ are wood swelling of the untreated and treated sample, respectively.

Statistical analysis

The data were processed in STATISTICA 10 software (StatSoft Inc., USA) and evaluated using a one-factor analysis of variance (ANOVA), completed with Tukey's honest significance test (HSD test).

RESULTS AND DISCUSSION

Uptake of Substance

Average WPG and retention values are shown in Table 2. The amount of Ac_2O impregnated within the wood structure was identical for all testing groups (MW I, MW II, MW III, and conventional heating) after the impregnation process within the same wood species group. The results showed significant differences between the WPG of beech (66%) and poplar (211%), even though the impregnation process parameters were the same. The major differences can be explained by different densities of wood species, which is reflected in calculating of WPG (formula 1). In fact it was caused also by the structural and chemical composition of the different wood species. For practical applications, it is rather important to know the amount of the impregnated substance expressed by weight in the wood volume. Therefore, retention was used as a second indicator of the substance uptake.

Based on the Tukey's HSD test, statistically insignificant WPG and retention were found between conventional heating, MW I, and MW III treatments for beech and conventional heating, MW II, and MW III treatments for poplar. All other acetylation treatments were statistically significant in terms of substance uptake (Table 2). After the chemical reaction, the WPG decreased by ~57% for beech and ~202% for poplar. This shows that a relatively high uptake of substance was reached but a small amount of the Ac_2O reacted within and remained in the wood structure. This can also be seen from the retention results. After the chemical reaction, the retention decreased from 656.2 $kg \cdot m^{-3}$ to ~30 $kg \cdot m^{-3}$ for poplar and from 452.7 $kg \cdot m^{-3}$ to ~65 $kg \cdot m^{-3}$ for beech. Values of beech retention (after the reaction) were two times higher than poplar. However, this does not mean that the efficacy of acetylation was also higher because of the higher retention. The efficacy depends on the degree of substitution and the amount of free hydroxyl groups, which is associated with wood density. Therefore, WPG is an appropriate indicator of the substance uptake and the acetylation process efficacy. Some authors have evaluated the effectiveness of acetylation by acetyl content using HPLC analysis (Larsson and Simonson 1999) or by degree of substitution determined by a back-titration method (Li *et al.* 2009). However, WPG is a well-known indicator used in most studies dealing with wood acetylation. In the present study, WPG values of the acetylated beech and poplar

samples were similar for all treatment modes, on average ~9%. Eranna and Pandey (2012) published data of the substance uptake for rubberwood (*Hevea brasiliensis*) conventionally treated at a temperature of 120 °C, after 15 min, 30 min, 60 min, and 120 min and reported that the WPG reached ~7%, ~8%, ~9%, and ~12%, respectively. Yang *et al.* (2014) compared the different types of acetylation reactions: liquid, microwave, and vapour phase after 60 min of reaction time. Sugi wood (*Cryptomeria japonica*) acetylated using a microwave reaction exhibited the highest WPG (19.4%), followed by the liquid phase reaction (19.1%), and vapour phase reaction (18.0%). Li *et al.* (2009) acetylated cellulose by MW heating with iodine as a catalyst. Results of the WPG after 15 min of the treatment at temperatures of 80 °C, 100 °C, and 130 °C were 13%, 16%, and 25%, respectively (Li *et al.* 2009). Pries *et al.* (2013) conventionally acetylated beech for a fungal decay test by Ac_2O at 120 °C for 120 min and observed 10.2% WPG. Results of the substance uptake (WPG, R) were difficult to compare with previously published data because of different wood species and process parameters used.

Table 2. Results of Substance Uptake (Weight Percentage Gain and Retention)

Uptake of substance	Treatment	WPG (%)		Retention (kg·m^{-3})	
		Beech	Poplar	Beech	Poplar
After impregnation	-	66.0 (3.8) ^{A}	211.3 (12.0) ^{A}	452.7 (23.2) ^{A}	656.2 (17.2) ^{A}
After chemical reaction	MW I	9.3 (1.1) ^{B}	7.7 (1.8) ^{B}	65.4 (6.4) ^{B}	24.2 (5.4) ^{B}
	MW II	10.2 (0.5) ^{C}	9.6 (0.8) ^{C}	70.0 (3.3) ^{C}	30.3 (2.3) ^{C}
	MW III	8.8 (0.8) ^{B}	9.3 (1.4) ^{C}	61.4 (5.5) ^{B}	30.1 (6.1) ^{C}
	Conventional Heat	9.5 (0.8) ^{B}	9.6 (1.3) ^{C}	64.7 (4.9) ^{B}	30.1 (4.1) ^{C}

Means sharing same letter in column are not significantly different (Tukey's HSD, $p < 0.05$)
Numbers in parentheses indicate standard deviation

Temperature

A maximum surface temperature was observed when microwave treatment at 2 kW with 0.025 m·min^{-1} conveyor speed (mode MW III) was used, as well as conventional heating. The surface temperature increased from 20 °C to 103 °C and 100 °C, respectively. Moreover, when milder MW heating modes were used, the surface temperature increased from 20 °C to 83 °C (MW II) and 69 °C (MW I). The results showed that the MW power and conveyor speed had a substantial effect on the sample temperature, influenced by various radiation intensities converted directly to thermal energy by frictional heating.

Equilibrium Moisture Content

By converting the hydroxyl groups of cell wall polymers into hydrophobic acetyl groups, the hygroscopicity of the wood was reduced. Values of the EMC at different relative humidities are presented in Fig. 3. Significant differences were found between the control, MW, and conventionally heated samples at all relative humidity levels (30%, 65%, and 99%). However, insignificant differences were found between various modes of MW treatment. In the case of poplar, there were insignificant differences between MW II and MW III treatments. Similar results were recorded for beech in 99% RH. In that respect, 60 min (MW II) and 15 min (MW III) treatments had identical results for EMC.

Yang *et al.* (2014) reported that the acetylation efficacy increased with reaction time. Such a statement was not confirmed in the present study. From the results, it can be

concluded that the MW treatment provided the same degree of acetylation independent of the duration of the reaction in the range of 15 to 60 min. However, it should be considered that only two time modes (conveyor speeds) were used in the experiment and deeper investigation is needed to confirm this statement. Larsson *et al.* (1999) studied the MW acetylation of pine and stated that the microwave-heated wood gives a higher degree of acetylation during the initial phase of the reaction compared to the conventional method. With a prolonged reaction time, the degree of acetylation was about the same, because only thermal effects are included when MW heating is applied to wood acetylation (Larsson *et al.* 1999).

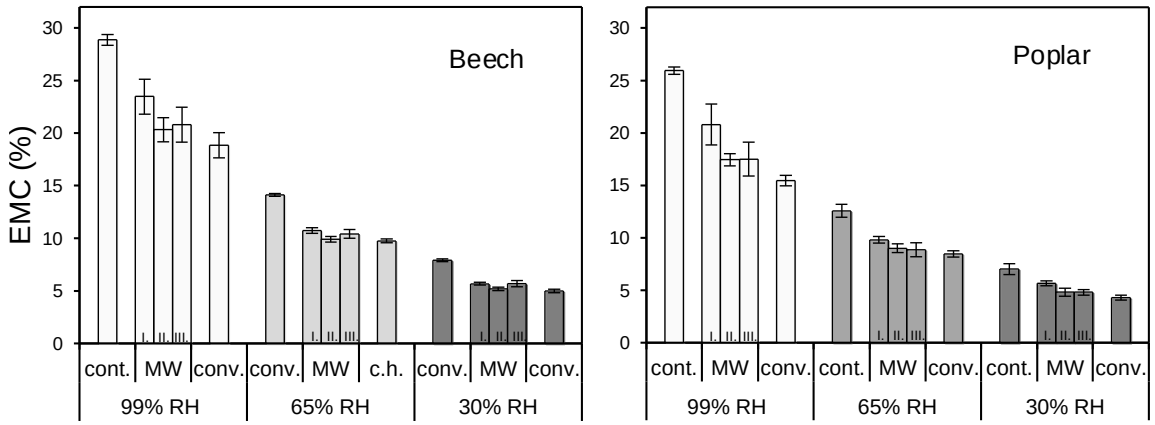


Fig. 3. EMC of beech and poplar under different RH; roman numerals indicate MW modes (MW I-III)

Using conventional heating for 60 min, the EMC of beech samples in 99% RH decreased by 35% when compared to the control. By using 2 kW microwave heating for 15 and 60 min (MW II and MW III), the EMC decreased by ~29%. Similar results were found for poplar samples. The EMC of conventionally heated poplar decreased by ~40% and by about 32% for MW heating samples (MW II, MW III). The MW I mode provided a lower degree of acetylation even though a longer period (60 min) of heating was used. This was probably caused by a low increase of temperature during the acetylation process. EMC values at 65% and 30% RH showed a similar trend as 99% RH (Fig. 3). The degree of acetylation increased with an increase in the MW power; therefore, it can be stated that MW power has a significant effect on wood acetylation.

Dimensional Stability

The radial and tangential swelling of wood (S_R and S_T) at different relative humidity levels are presented in Fig. 4. Results at 99% RH were used for the ASE, indicating the effectiveness of treatments on the dimensional stability (Fig. 5).

The control samples had a higher radial and tangential swelling than acetylated samples. Since acetyl groups occupy space within the cell wall, the wood is not able to absorb water molecules and therefore wood swelling is reduced (Tarkow *et al.* 1950; Rowell 1983). When conventional and MW acetylation are compared, only very minor differences in the wood swelling can be found. Moreover, statistically insignificant differences were found between 60 min (conventional, MW III) and 15 min treatments. Heating by MW can accelerate chemical reactions in the acetylation process, whereby the

same dimensional stability is reached. Therefore, the reaction time of acetylation can be reduced and the process made more effective.

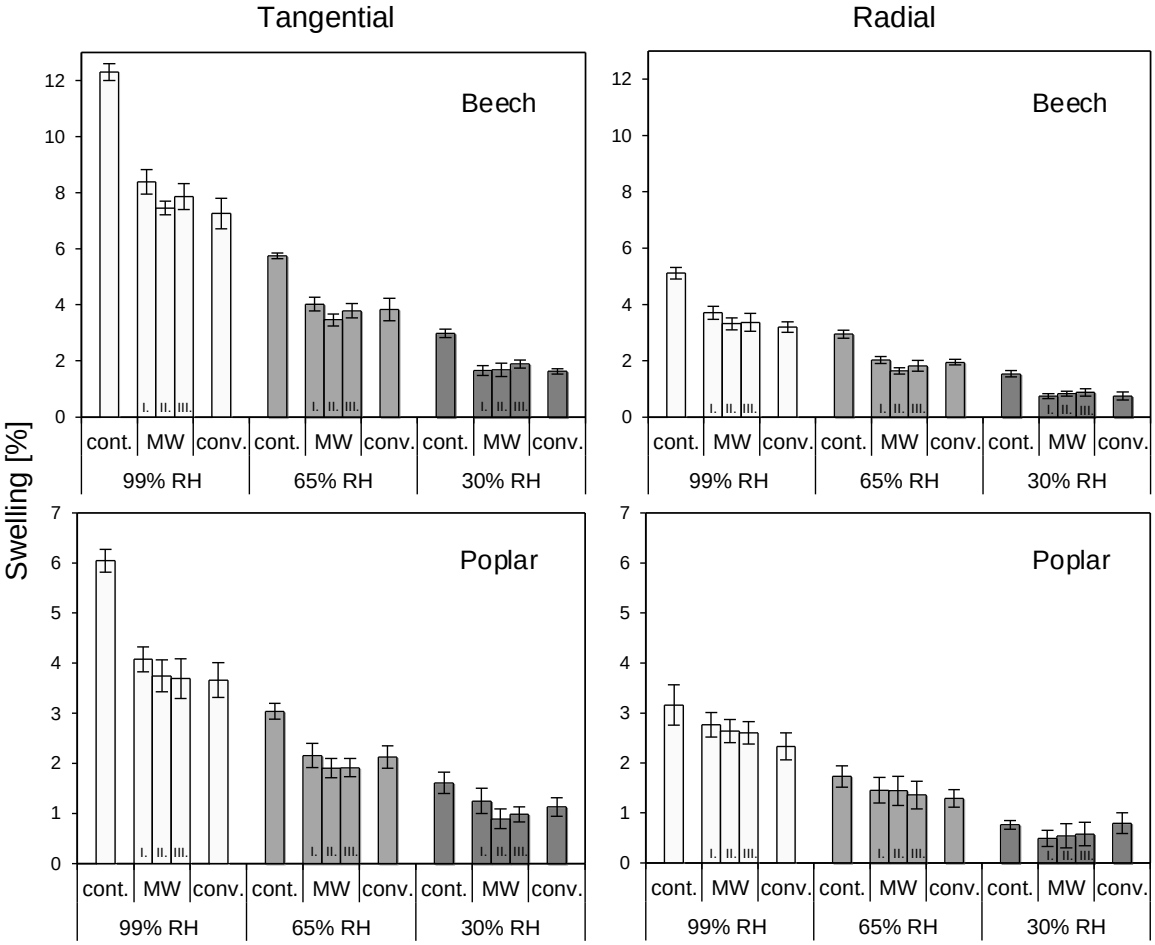


Fig. 4. Swelling of beech and poplar in the tangential and radial directions

Anti-swelling efficiency is the most commonly used method to evaluate the dimensional stability of the modified wood (Santos 2000; Čermák *et al.* 2015). The dimensional stability was considerably improved for all applied acetylation treatment modes. The ASE of the acetylated beech in modes MW I, MW II, MW III, and conventional heating were 31.8%, 39.4%, 36.1%, and 41.0% in the tangential and 27.7%, 35.2%, 34.3%, and 37.6% in the radial direction, respectively. Similar values of ASE_T and lower values of ASE_R were found for poplar samples, *i.e.*, 35.8%, 38.0%, 38.9%, and 39.4% in the tangential and 12.4%, 16.3%, 17.0%, and 26.1% in the radial direction, respectively.

Unfortunately, it was difficult to compare the ASE data of the acetylated wood, because no study dealing with the same wood species was found. Nevertheless, the data are comparable with previous studies published by Hill and Jones (1996) and Rowell *et al.* (2008). Hill and Jones (1996) stated that after the acetylation of Corsican pine (*Pinus nigra*), ASE reached 35% at 10% WPG. Similar results were found by Rowell *et al.* (2008), who reported 34% ASE (at 10% WPG) for southern pine (*Pinus taeda*).

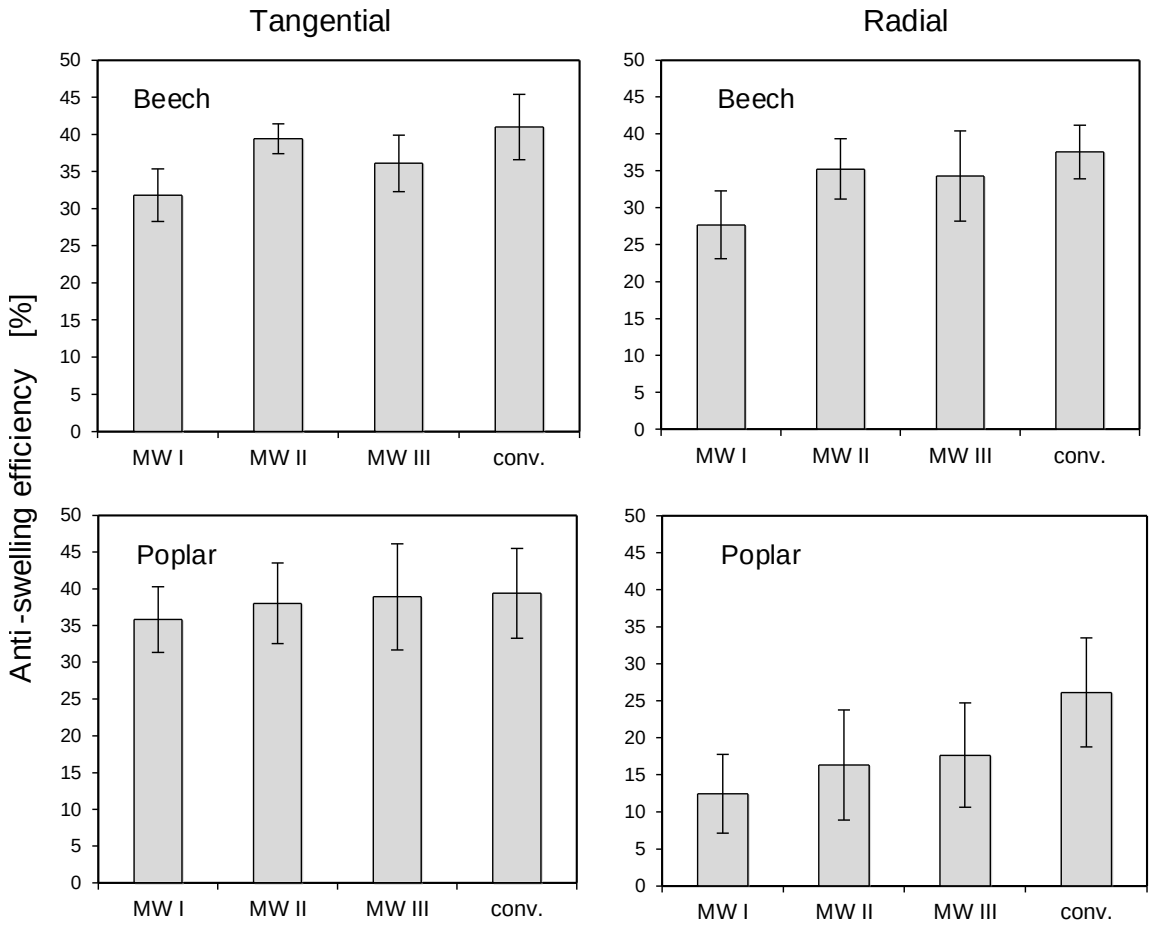


Fig. 5. ASE of beech and poplar in tangential and radial directions in 99% relative humidity

It is well known that wood is an anisotropic material with different dimensional changes in different anatomical directions (Kollmann 1951; Boutelje 1962; Stamm 1964; Skaar 1988). According to Niemz *et al.* (1993), swelling in the tangential and radial directions can be expressed by a 2:1 (S_T:S_R) ratio. The tangential and radial swelling ratios ($S_{T,99\%}$:$S_{R,\ 99\%}$) for control, MW, and conventional heating were 2.4:1, 2.3:1, and 2.3:1 for beech and 1.9:1, 1.4:1, and 1.6:1 for poplar. From the results, it can be concluded that acetylated beech specimens provided negligible swelling ratio improvement. However, poplar samples showed much more significant swelling ratio improvement (S_T:S_R). This improvement is attributed to the lower ASE of poplar in the radial direction (Fig. 5) compared to the tangential direction. The acetylated wood (species-dependent) can be therefore considered more homogenous, but anisotropy of swelling remains.

CONCLUSIONS

1. The acetylation of samples using different treatment modes (MW I, MW II, MW III, and conventional heating) provided similar WPG values after chemical reactions (~9%). Retention, expressed by weight in the wood volume, has been suggested as a

better indicator of substance uptake. Beech wood had approximately two times higher substance retention compared to poplar, due to its structural and chemical composition.

2. The microwave and conventional acetylation positively affected equilibrium moisture content, wood swelling, and consequently the dimensional stability. The improvement in the investigated properties was nearly identical in both types of treatment. Therefore, the efficacy of the acetylation process carried out using microwave or conventional heating is comparable.
3. The rate of acetylation increased with an increase of microwave power. However, the treatment time (15 and 60 min) did not affect the degree of acetylation. These results were confirmed for all relative humidity levels (30%, 65%, and 99%).
4. The optimum mode of microwave acetylation was found at the microwave power of 2 kW using 0.1 $m \cdot min^{-1}$ conveyor speed (15 min). This mode resulted in 39.4% ASE_T and 35.2% ASE_R for beech and 38.0% ASE_T and 16.3% ASE_R for poplar samples.
5. Microwave heating was found to be an efficient rapid acetylation process (15 min, 0.1 m/min). However, more detailed investigation of the time-dependency of microwave heating should be done in the future.

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