

## **PHYSICAL AND MECHANICAL PROPERTIES OF THERMO-HYGRO-MECHANICALLY (THM) – DENSIFIED WOOD**

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### **ABSTRACT**

Physical and mechanical properties of thermo-hygro-mechanically (THM)-densified wood of *Picea abies* and *Fagus sylvatica* were investigated. Three different treatments were applied: thermo-hygro (TH), mechanical densification and THM densified and post-treated under saturated steam at different temperatures. The density of the specimens was significantly increased after THM and slightly reduced after TH-treatment. Densified and non-post treated samples showed a strong tendency of spring-back, while THM-densification eliminated compression set recovery. THM-treatment reduced the hygroscopicity of beech and Norway spruce wood. Brinell hardness, Compressive strength and MOE increased with increasing temperatures applied during the post-treatment phase. The studies show that TH-treatment has an adverse effect on the strength properties of wood as the mechanical properties are affected. Densification in combination with TH post treatment improved the mechanical performance of both species rendering wood more rigid but less elastic.

**KEY WORDS:** beech, hygroscopicity, mechanical properties, Norway spruce, set-recovery, thermo-hygro-mechanical (THM) – densification.

### **INTRODUCTION**

Due to environmental concerns regarding the use of certain types of preservatives, there has been a renewed interest in wood modification. Three types of wood modification have been investigated in recent years: thermal, chemical and physical. Improvements in dimensional stability, physical and mechanical properties, enhancement of durability against biological degradation are the primary concerns of wood modification.

Thermal modification has long been recognized as a potential method to improve the

dimensional stabilization of wood, increase the natural durability of solid wood and allow its use in outdoor applications (Boonstra et al. 1998, Zaman et al. 2000, Alén et al. 2002, Junghans et al. 2005). The main problem associated with thermal treatment is the reduced mechanical performance of the end product (Repellin and Guyonnet 2003, Welzbacher and Rapp 2005).

Wood densification is another process that was developed to significantly improve the mechanical properties of wood. One of the main problems associated with most types of densified wood (except those with high resin content) is the lack of dimensional stability. When soaked in water or exposed to high relative humidity, compressed wood has the tendency of irreversible swelling or spring back (Hillis 1984, Inoue et al. 1998, Schrepfer and Schweingruber 1998). This can be a serious problem when densified wood is used in high humidity environments. There have been many studies related to dimensional stabilization of densified wood with a range of treatments. Hillis (1984) reviewed the literature on stabilization of wood after applying a heating process. The effect of steam pre-treatment was investigated by Hsu et al. (1988), Inoue et al. (1993), Inoue et al. (1998) and more recently the effect of heat on the dimensional stability of compressed wood was evaluated by Norimoto et al. (1993).

To overcome the drawbacks of thermal treatment, researchers have considered the combination of densification and heat treatment for improving both the dimensional stability and mechanical performance of wood. In Japan, in particular, different researchers e.g. Tanahashi (1990), Inoue et al. (1993), Norimoto et al. (1993) have made considerable progress towards the permanent fixation of compression set by applying high temperature steam. Ito et al. (1998b) have successfully developed an innovative process with high temperature steam, for the permanent fixation of transformed and compressed logs of sugi into square samples. Morsing (2000) demonstrated that permanent fixation of compression deformation could be achieved at high temperature e.g. 190°C for 20 hours or 5 hours at 200°C. He also compared the total fixation time of set-recovery using heat and steam treatments with beech and demonstrated that steam treatment could improve the hardness and dimensional stability of wood much faster than heat treatment alone.

Thermo-hygro-mechanically (THM)-densified wood is a unique material in the field of engineered wood products. High temperature, moisture and compression are used to manufacture a wood product with superior strength properties than that of natural wood. An improved densification method was presented in 1998 that included steaming to provide shape fixation (Ito et al. 1998a). Further improvements to densification were published by Navi and Girardet (2000) that resulted in a compressed and stabilized wood product with improved set-recovery. Navi and Heger (2004) found that wood compressed by THM-densification showed increased shear and strength parallel to the grain as well as increased surface hardness. Increase in tensile strength after densification has also been reported by Navi and Girardet (2000). By contrast, Perkitny and Jablonski (1984) showed that densified wood was weaker than natural wood after testing bending and compression strength.

As an alternative to a hygro-post treatment, Norway spruce wood was densified in a common industrial scale process and thereafter thermally modified with an oil-heat treatment process (Welzbacher et al. 2005). The bending strength of densified and oil-heat treated material was only slightly reduced in comparison to untreated Norway spruce wood. Densified and thermally modified samples showed improved dimensional stability compared to untreated densified material. In contrast to steam post-treatment, durability was significantly increased after oil-heat treatment. As a drawback, the impact bending strength of densified and oil-heat treated spruce were reduced by 40% compared to controls, though static bending strength was equal to untreated spruce wood. Nowadays extensive work is carried out at the TU Dresden for the shaping of wooden parts by compression (Haller and Wehsener 2001, Haller 2004) and using the set recovery effect for obtaining desired shapes (Ziegler and Haller 2006).

The objective of the present study was to assess a range of physical and mechanical properties of THM-densified Norway spruce and beech wood and to determine the effect of each treatment phase of the THM process on the overall performance of the resulting wood product. For this purpose wood samples of Norway spruce (*Picea abies*) and beech (*Fagus sylvatica*) were subjected to physical and mechanical testing according to EN standards.

## MATERIAL AND METHODS

### Thermo-hygro-mechanical densification

Wood specimens (dimensions: 150 mm x 25 mm x 15 mm, R x T x L) were excised from the heartwood of living 40-50 year old trees of Norway spruce (*Picea abies* Karst.) and beech (*Fagus sylvatica* L.) from central Switzerland. Before treatment wood samples were conditioned at 20°C and 65% relative humidity (RH). The specimens were cut from the same stem and were free of knots and resin and showed no visible signs of microbial infection. Seven different optimised treatments on the basis of the THM-process developed by Navi and Heger (2004) were selected for wood treatment (Tab. 1). All test specimens were manufactured at EPFL, Switzerland. Non treated control specimens were included for all test series.

Tab. 1: Treatment conditions of beech and Norway spruce wood specimens

| Condition of wood |                                                                  | Temperature (°C) |                | Duration (min) |                |
|-------------------|------------------------------------------------------------------|------------------|----------------|----------------|----------------|
|                   |                                                                  | Densification    | Post-treatment | Densification  | Post-treatment |
| <b>Control</b>    | Untreated                                                        | -                | -              | -              | -              |
| <b>TH 160</b>     | Heat treatment at 160°C under SSC                                | -                | 160            | -              | 75             |
| <b>TH 180</b>     | Heat treatment at 180°C under SSC                                | -                | 180            | -              | 35             |
| <b>Densified</b>  | Mechanical densification under SSC without post-treatment        | 140              | -              | 20             | -              |
| <b>THM 140</b>    | Densification and post-treatment at 140°C under SSC              | 140              | 140            | 20             | 150            |
| <b>THM 160</b>    | Densification and post-treatment at 160°C under SSC              | 140              | 160            | 20             | 75             |
| <b>THM 180</b>    | Densification and post-treatment at 180°C under SSC              | 140              | 180            | 20             | 35             |
| <b>THM 180/80</b> | Densification under SSC and post-treatment at 180°C under 80% RH | 140              | 180            | 20             | 65             |

SSC, saturated steam conditions. RH, relative humidity.

During the first stage of the THM procedure the specimens were mechanically compressed in a multiparameter reactor (Fig. 1) under saturated steam and a controlled displacement mode in the radial direction. The process consists of 2 steps (Fig. 2): at first, the sample is steam heated for 10 min. at 140°C and 3.61 bar steam pressure (plasticisation) and then densified under controlled displacement mode at 5 mm.min<sup>-1</sup> until 5kN, 2.5 mm.min<sup>-1</sup> until 10 kN, 1.25 mm.min<sup>-1</sup> until 20 kN, 0.5 mm.min<sup>-1</sup> until 22 kN. When 22 kN is reached for the second time, the piston is stopped and the sample cooled down to 60°C and removed from the reactor if not subjected to further post treatment.

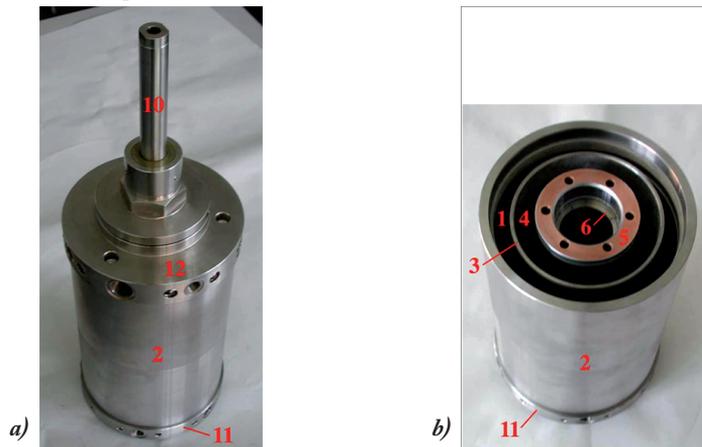


Fig. 1: a) THM-reactor closed by the compression piston b) Open reactor

After densification, the samples were post-treated under different conditions (Tab. 1). The post-treatments were carried out either under saturated steam (6.18 bar) or 80% relative humidity, which made it easier to control the temperature by varying the pressure. After densification the piston was stopped and maintained in the same position throughout the post-treatment stage. Afterwards the reactor was cleaned, the samples immediately cooled to 60°C and removed from the reactor (Fig. 3).

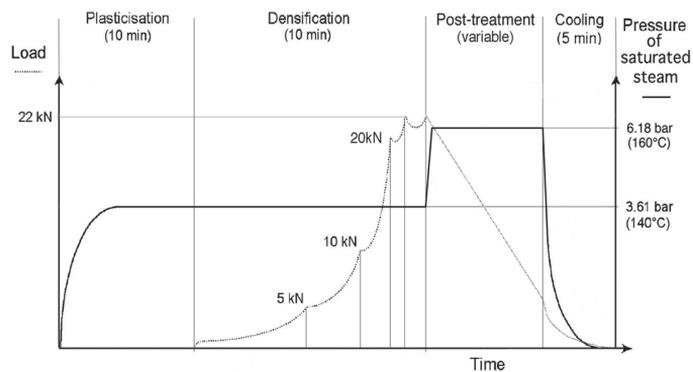


Fig. 2: Different stages of the THM-densification procedure (Navi and Heger 2005)

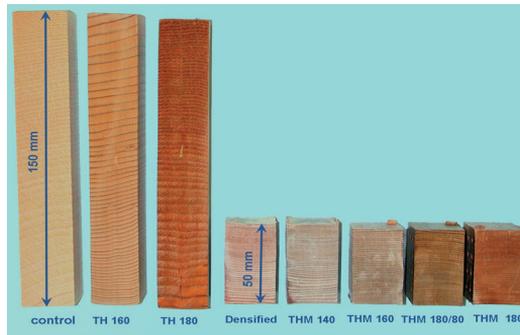


Fig. 3: Norway spruce wood specimens after different THM-treatments and temperatures of post treatment (140°C, 160°C, 180°C)

### Physical-mechanical properties

In most cases wood modification results in changes in its mechanical and physical properties. Alterations in wood density, Brinell hardness, compression strength and MOE as well as moisture-related properties were investigated on THM-densified beech and Norway spruce wood specimens. Due to the restrictions of the specimens' sizes, it was not possible to assess additional parameters.

### Density

Density increases under compression proportionally to the compression strength. The density of the specimens were assessed according to (DIN 52-182 1976) after preconditioning the wood at 20°C and 65% relative humidity until the constant mass was reached.

### Compression set

The value of compression set (C) was defined as follows:

$$C = \frac{R_o - R_c}{R_o} \times 100\% \quad (1)$$

where:

$R_o$  and  $R_c$ , dimensions of the samples in the direction of compression before and after densification respectively.

### Compression set recovery

Compression set recovery ( $C_{rec}$ ) (synonyms: spring-back, set-memory, set-recovery, shape-recovery) determines the irreversibly recovered thickness in the direction of compression due to shape-memory effect. It is calculated as follows:

$$C_{rec} = \frac{R_{c_{wet}} - R_c}{R_o - R_c} \times 100\% \quad (2)$$

where:

$R_o$  size of specimen before densification

$R_c$  size of specimen after densification

$R_{c_{wet}}$  size of specimen in the direction of compression after soaking in water for 3 days at 20°C, followed by oven-drying

## Moisture content

The percentage of moisture content was determined according to DIN 52-183 1977.

## Linear swelling

All specimens (dimensions approx. 10 mm x 25 mm x 30 mm, R x T x L) were placed into the climate chamber with a constant temperature of 20°C and a relative humidity of 35%. Each specimen was weighed at regular intervals until the alterations in weight over a 2 day period were less than 0.1%. After such a defined equilibrium moisture content was reached the dry weights as well as the dimensions were recorded. The relative humidity was subsequently increased according to the scheme: 35% → 50% → 65% → 80% → 93%. Finally the specimens were dried in the oven at 103 ± 2°C for 2 days and both the oven dry weight and the dimensions were measured. The linear swelling was calculated as follows:

$$\alpha_u = \frac{a_u - a_o}{a_o} \times 100\% \quad (3)$$

where:

$a_u$  size of the specimen in the particular anatomic direction at the moisture content  $u$

$a_o$  size of the oven dry specimen

## Brinell hardness

In order to estimate Brinell hardness (HB) a steel sphere with a diameter of 10mm was pressed against the surface of the wood specimen for 10 seconds with a defined force (500 N). The surface of indentation was measured, and the load divided by the area of the impression surface on the material. As different spheres produce different impressions, the ratio of the load to the square of sphere diameter needs to be constant so to maintain the same HB. The test was conducted in the radial and longitudinal direction on all specimens according to (DIN 1534 2000). The Brinell hardness (HB) was calculated as follows:

$$HB = \frac{2 \times F}{\pi \times D \times (D - \sqrt{D^2 - d^2})} \quad (4)$$

where:

F applied force (N)

D diameter of indenter (10 mm)

d diameter of indentation (mm)

The measurements were conducted with the Universal Testing Machine “Zwick” equipped with “Universalhärtemeskopf”. This device gives an opportunity to measure the depth of indentation and calculate the diameter of indentation out of it. This simplifies the procedure considerably and averts time consuming and inaccurate indentation diameter measurements. The HB was calculated as follows:

$$HB = \frac{F}{\pi \times D \times h} \quad (5)$$

where:

F applied force (N)

D diameter of indenter (10 mm)

h depth of indentation (mm)

### Compression strength perpendicular to the grain

The compressive strength of a material is determined by the value of uniaxial compressive stress reached when the wood fails completely. The compression strength value was obtained experimentally in a compressive test according to (DIN 52-192 1979). Wood specimens (dimensions 50 x 25 x 15 mm) were loaded in the Zwick (100 kN) Universal Testing Machine longitudinally or radially to the grain, applying the load at a displacement rate of 0.5 mm.min<sup>-1</sup>. The radial loading referred to loading applied in the radial direction (loading applied to the tangential surface). The compression strength ( $\sigma_{max}$ , MPa), Young's modulus in compression (MOE) were both determined from the recorded strain – stress curves using the pre-calibrated Zwick software. The maximum compression of 2% was defined for all wood specimens.

### Porosity

Mercury intrusion porosimetry was used for assessment of the porosity of the specimens (Junghans et al. 2005, Moura et al. 2005). Specimens of untreated, TH 180 and THM 180 Norway spruce were cut into sub-samples of about 10 mm x 7 mm x 5 mm (R x T x L). The specimens were fixed in dilatometer which was later filled with 450ml of mercury. During the measurement process the pressure on mercury was constantly raised. The alterations in the mercury level in the column were registered via the change of electrostatic capacity. The measurements were conducted on the PASCAL 140/440 (ThermoFinnigan, UK).

### Scanning electron microscopy (SEM)

Radial longitudinal specimens from wood blocks were extracted from THM-densified samples. These were then prepared for scanning electron microscopy (SEM), dried in a vacuum oven at 40°C and 10 mbar for 12 hours, glued on a specimen holder using a carbon adhesive and sputtered with a platinum layer of approx. 10 nm. The specimens were investigated with a Field Emission SEM (Jeol 6200F) at an acceleration voltage of 5 kV and a working distance of 24 mm.

### Statistical analysis

One-way analysis of variance (ANOVA) of the recorded dry weight losses was performed for wood specimens in Excel with the significance level set at  $p < 0.01$ . A Tukey HSD post-hoc test was performed in SPSS to demonstrate the differences in mean values.

## RESULTS

### Compression-set, density and spring-back

THM-densification induced a high degree of densification in treated wood specimens. Compression sets of approx. 73% and 45% for Norway spruce and beech wood, respectively, were obtained with the THM-densification process (Tab. 2). The values differed within treatments depending on the morphology of each treated sample. Densification and post-treatment of Norway spruce resulted in almost complete occlusion of the cell lumina in both early- and latewood tracheids (Fig. 4).

Tab. 2: Compression set and density of THM-densified beech and Norway spruce wood specimens (mean values,  $n = 12$ )

| Treatment of wood | Compression set (%) |       | Density ( $\text{g}\cdot\text{cm}^{-3}$ ) |       | Compression set recovery (%) |       |
|-------------------|---------------------|-------|-------------------------------------------|-------|------------------------------|-------|
|                   | Spruce              | Beech | Spruce                                    | Beech | Spruce                       | Beech |
| Control           | -                   | -     | 0.361                                     | 0.651 | -                            | -     |
| TH 160            | -                   | -     | 0.358                                     | 0.648 | -                            | -     |
| TH 180            | -                   | -     | 0.351                                     | 0.648 | -                            | -     |
| Densified         | 71.85               | 45.29 | 1.272                                     | 1.140 | 44.72                        | 61.38 |
| THM 140           | 72.85               | 46.21 | 1.324                                     | 1.223 | 11.5                         | 25.86 |
| THM 160           | 73.18               | 42.83 | 1.296                                     | 1.239 | 7.52                         | 23.11 |
| THM 180           | 73.33               | 44.74 | 1.279                                     | 1.194 | 4.07                         | 15.99 |
| THM 180/80        | 73.18               | 45.84 | 1.257                                     | 1.177 | 6.67                         | 18.22 |

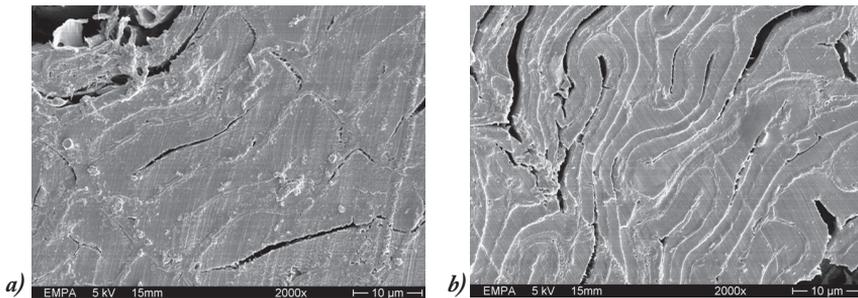


Fig. 4: Transverse section of Norway spruce densified and post-treated at  $180^{\circ}\text{C}$

a) Latewood tracheids. Note a complete occlusion of the cell lumina

b) Earlywood tracheids. Note that the cell lumina of most tracheids is completely occluded

The density of the specimens was significantly increased by THM-densification and slightly reduced by the TH-treatments. The maximum density of  $1.32 \text{ g}\cdot\text{cm}^{-3}$  for spruce wood and  $1.24 \text{ g}\cdot\text{cm}^{-3}$  for beech wood was obtained after THM 140 and THM 160 treatments respectively (Tab. 2).

THM-densification followed by post-treatment induced the elimination of compression-set recovery. THM 180 treated specimens showed the lowest compression set recovery values (Tab. 2). Densified and non post-treated specimens showed a strong tendency of spring-back, although they did not fully recover to their initial shape. Thus, spruce samples only recovered to approx. 45%, whereas beech wood showed a higher shape memory effect (approx. 62%).

### Water sorption

All TH- and THM-treatments altered the sorption behaviour of beech wood, resulting in reduction of wood moisture content in humid environment. This effect was least pronounced in densified samples that were not post-treated. However, the THM 180/80 treatment markedly reduced the hygroscopicity of beech specimens (Tab. 3).

Tab. 3: EMC in THM-densified beech wood (*Fagus sylvatica*) and Norway spruce wood (*Picea abies*) as a function of relative air humidity at 20°C (mean values, n = 6)

|                                         | Relative air humidity (%) | Treatment |        |           |         |         |         |            |         |
|-----------------------------------------|---------------------------|-----------|--------|-----------|---------|---------|---------|------------|---------|
|                                         |                           | TH 160    | TH 180 | Densified | THM 140 | THM 160 | THM 180 | THM 180/80 | Control |
| <b>Equilibrium moisture content (%)</b> |                           |           |        |           |         |         |         |            |         |
| Beech                                   | 35                        | 6.8       | 6.5    | 7.5       | 5.7     | 6       | 6.2     | 6.1        | 7.8     |
|                                         | 50                        | 8.4       | 7.8    | 9.3       | 7       | 7.4     | 7.5     | 7.3        | 9.9     |
|                                         | 65                        | 10.8      | 9.8    | 12.6      | 9.2     | 10      | 9.5     | 9.1        | 12.8    |
|                                         | 80                        | 16.1      | 15.7   | 18.8      | 14.4    | 15.6    | 15.3    | 13.4       | 19.1    |
|                                         | 93                        | 18.6      | 17.6   | 23.1      | 15.8    | 18.3    | 18.2    | 16.9       | 26.4    |
| <b>Equilibrium moisture content (%)</b> |                           |           |        |           |         |         |         |            |         |
| Norway spruce                           | 35                        | 5.9       | 5.5    | 7.9       | 6.5     | 6.7     | 6.9     | 5.3        | 8.1     |
|                                         | 50                        | 7.8       | 7.2    | 9.7       | 8.1     | 9.1     | 8.3     | 6.5        | 9.9     |
|                                         | 65                        | 10.9      | 10.1   | 12        | 10.3    | 11.9    | 11.9    | 8.7        | 12.2    |
|                                         | 80                        | 16.7      | 15.1   | 17.9      | 16.5    | 17.1    | 17.4    | 12.6       | 18.2    |
|                                         | 93                        | 18.7      | 18.8   | 22.4      | 18.7    | 19.8    | 19.3    | 15.1       | 26.4    |

THM180/80 treatment of Norway spruce wood rendered it less sensitive to fluctuations in the relative humidity. Increasing temperatures applied during the post-treatment phase led to reduction of EMC and decreased the rate of moisture-uptake compared to untreated control samples (Tab. 3).

## Swelling

Maximum swelling of the densified wood specimens was affected by TH-post-treatment, which markedly decreased it. However, the lowest swelling was recorded for TH 160 and TH 180-treated beech (Tab. 4) and Norway spruce specimens (Tab. 5). Densification without TH-post treatment did not markedly improve the swelling behaviour of both species, furthermore, the values recorded for the radial swelling are approx. 4 times higher than those recorded for controls.

Tab. 4: Tangential and radial swelling of THM-densified beech wood (*Fagus sylvatica*) as a function of relative air humidity at 20°C (mean values, n = 6)

| Relative air humidity (%)      | Treatment |        |           |         |         |         |            | Control |
|--------------------------------|-----------|--------|-----------|---------|---------|---------|------------|---------|
|                                | TH 160    | TH 180 | Densified | THM 140 | THM 160 | THM 180 | THM 180/80 |         |
| <b>Tangential swelling (%)</b> |           |        |           |         |         |         |            |         |
| 35                             | 1.25      | 0.85   | 1.14      | 2.17    | 2.16    | 1.63    | 1.92       | 1.35    |
| 50                             | 1.68      | 1.44   | 2.04      | 2.76    | 2.8     | 2.42    | 2.5        | 1.8     |
| 65                             | 2.58      | 2.37   | 3.39      | 3.78    | 3.69    | 3.31    | 2.82       | 3.07    |
| 80                             | 6.29      | 6.02   | 6.71      | 6.65    | 5.44    | 5.48    | 4.42       | 7.55    |
| 93                             | 8.69      | 8.59   | 8.95      | 8.1     | 7.15    | 7.04    | 5.72       | 10.53   |
| <b>Radial swelling (%)</b>     |           |        |           |         |         |         |            |         |
| 35                             | 0.82      | 0.86   | 1.32      | 1.62    | 1.16    | 1.03    | 1.48       | 0.96    |
| 50                             | 1.09      | 1.06   | 3.42      | 2.33    | 1.82    | 1.83    | 2.49       | 1.23    |
| 65                             | 1.41      | 1.38   | 5.13      | 3.16    | 3.83    | 3.61    | 3.77       | 1.91    |
| 80                             | 2.67      | 2.66   | 12.03     | 7       | 7.27    | 7.61    | 6.96       | 2.71    |
| 93                             | 3.55      | 3.57   | 20.34     | 11.79   | 11.28   | 11.26   | 11.23      | 4.95    |

Tab. 5: Tangential swelling of THM-densified spruce wood (*Picea abies*) as a function of relative air humidity at 20°C (mean values,  $n = 6$ )

| Relative air humidity (%) | Treatment                      |        |           |         |         |         |            |         |
|---------------------------|--------------------------------|--------|-----------|---------|---------|---------|------------|---------|
|                           | TH 160                         | TH 180 | Densified | THM 140 | THM 160 | THM 180 | THM 180/80 | Control |
|                           | <b>Tangential swelling (%)</b> |        |           |         |         |         |            |         |
| 35                        | 1.8                            | 0.91   | 1.23      | 1.23    | 1.36    | 1.17    | 1.29       | 1.99    |
| 50                        | 2.06                           | 1.41   | 1.7       | 1.96    | 1.85    | 1.75    | 1.65       | 2.18    |
| 65                        | 3.56                           | 2.84   | 3.04      | 2.89    | 2.76    | 2.82    | 2.36       | 3.81    |
| 80                        | 4.95                           | 4.63   | 5.22      | 5.1     | 5.13    | 4.67    | 3.61       | 4.64    |
| 93                        | 6.61                           | 6.12   | 7.9       | 5.99    | 6.15    | 5.14    | 4.63       | 7.51    |
|                           | <b>Radial swelling (%)</b>     |        |           |         |         |         |            |         |
| 35                        | 0.99                           | 0.4    | 1.73      | 1.15    | 2.96    | 2.76    | 2.32       | 1.31    |
| 50                        | 1.41                           | 0.74   | 2.74      | 1.68    | 3.54    | 3.29    | 2.45       | 1.58    |
| 65                        | 1.9                            | 1.7    | 4.61      | 3.75    | 5.6     | 5.63    | 5.01       | 2.53    |
| 80                        | 2.64                           | 2.8    | 11.44     | 8.91    | 10.38   | 10.05   | 8.37       | 2.86    |
| 93                        | 4.58                           | 4.46   | 26.29     | 12.77   | 13.46   | 12.65   | 11.54      | 5.06    |

### Brinell hardness

In comparison to the radial direction, control specimens of beech and Norway spruce wood showed a hardness approx. 3-fold higher in the tangential direction. The highest HB values were recorded for all densified samples i.e. for post treated and non post treated wood specimens. TH-treatments, on the contrary, did not noticeably affect Brinell hardness; hence both species did not show significant difference from controls. THM-densification in radial direction resulted in substantial improvement of HB and rendered spruce wood samples equally hard in both tangential and radial directions (Fig. 6). A similar tendency was observed for beech wood specimens (Fig. 5).

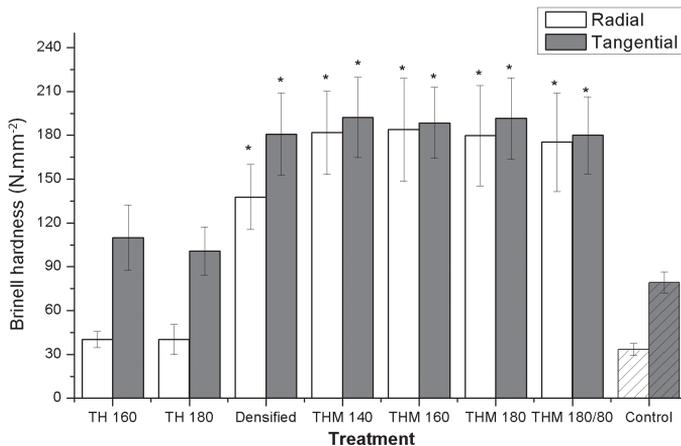


Fig. 5: Brinell hardness of untreated controls, TH- and THM-treated beech wood specimens. Columns marked with asterisk (\*) denote significant differences in comparison to untreated controls ( $p < 0.01$ )

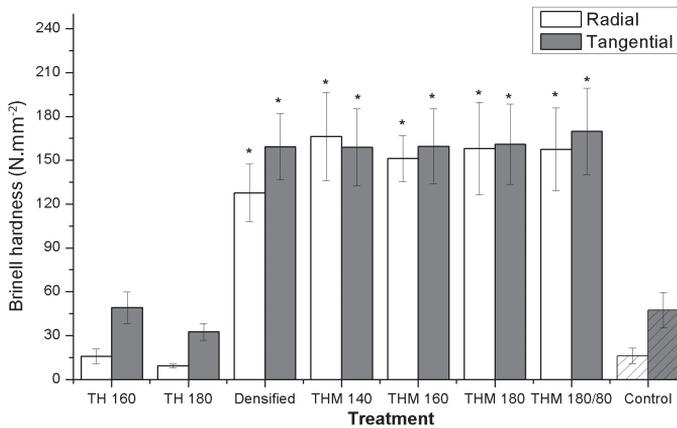


Fig. 6: Brinell hardness of untreated controls, TH- and THM-treated Norway spruce wood specimens. Columns marked with asterisk (\*) denote significant differences in comparison to untreated controls ( $p < 0.01$ )

### Compressive strength perpendicular to the grain

TH-treatments decreased both the compressive strength and MOE in compression of beech and Norway spruce specimens. THM-densification, on the other hand, substantially augmented compression strength by approx. 20-fold in spruce and approx. 3-fold in beech (Tab. 6). A marked improvement of compressive strength induced by THM-densification for Norway spruce wood is demonstrated in Tab. 6. Observations of the specimens behaviour under compression showed that till the very moment of fracture the THM-densified specimen no evidence of any deformation to the visible eye. TH-treated wood blocks, however, behaved as control specimens developing cracks and deformations at the early stages of compression. The elastic behaviour is more pronounced in Norway spruce wood than in beech. It is advisable to further investigate elastic properties for the potential application of THM –densified wood.

### Porosity

THM-densification induced major alterations in the wood porosity markedly reducing the average pore diameter and total porosity values. However, TH180 treatment without densification resulted in a slight increase of the porosity (Tab. 7). The pore size distribution indicates the impact of the treatment. The trend could be observed by dividing the pore's radius into two classes (class 1:  $r > 1 \mu\text{m}$  and class 2:  $r < 1 \mu\text{m}$ ). The amount of class 1 pores increased after TH treatment and decreased after THM-densification; on the contrary, the relative volume of smaller (class 2) pores decreased after TH process. However, the THM-densification induced the increase of relative amount of class 2 pores (Tab. 7).

Tab. 6: Compressive strength perpendicular to the grain ( $\sigma_{max}$ , 2% compression) and the modulus of elasticity in compression (MOE) of THM-densified beech and Norway spruce wood ( $n=3$ , wood humidity 13%)

| Species | Treatment  | $\sigma_{max}$<br>(N.mm <sup>-2</sup> ) | st. dev | MOE<br>(N.mm <sup>-2</sup> ) | st. dev |
|---------|------------|-----------------------------------------|---------|------------------------------|---------|
| Beech   | Control    | 12.96                                   | 2.45    | 1388                         | 86      |
|         | TH 160     | 10.87                                   | 3.74    | 1251                         | 124     |
|         | TH 180     | 8.42                                    | 1.52    | 986                          | 120     |
|         | Densified  | 50.16                                   | 2.18    | 1658                         | 127     |
|         | THM 140    | 56.73                                   | 2.12    | 1763                         | 102     |
|         | THM 160    | 51.31                                   | 3.76    | 1742                         | 147     |
|         | THM 180    | 53.67                                   | 3.03    | 1884                         | 115     |
|         | THM 180/80 | 49.48                                   | 3.39    | 1818                         | 152     |
| Spruce  | Control    | 3.45                                    | 1.22    | 1123                         | 131     |
|         | TH 160     | 3.25                                    | 1.43    | 951                          | 154     |
|         | TH 180     | 2.62                                    | 2.09    | 973                          | 203     |
|         | Densified  | 68.71                                   | 12.28   | 2567                         | 112     |
|         | THM 140    | 83.3                                    | 7.64    | 3470                         | 165     |
|         | THM 160    | 72.42                                   | 11.46   | 3774                         | 258     |
|         | THM 180    | 85.39                                   | 9.17    | 3078                         | 123     |
|         | THM 180/80 | 81.05                                   | 10.96   | 3719                         | 235     |

Tab. 7: Porosity of Norway spruce wood specimens

| Porosity                                | untreated                        | TH 180 | THM 180 |
|-----------------------------------------|----------------------------------|--------|---------|
| Mean pore diameter ( $\mu\text{m}$ )    | 1.69                             | 5.16   | 0.26    |
| Total porosity (%)                      | 77.02                            | 86.01  | 9.98    |
| Total pore volume (g.mm <sup>-3</sup> ) | 583.3                            | 125.1  | 49.5    |
| Density (g.cm <sup>-3</sup> )           | 0.42                             | 0.34   | 1.36    |
| Relative pore volume (%)                | Class 1 (1000-1 $\mu\text{m}$ )  | 54     | 62      |
|                                         | Class 2 (1-0.001 $\mu\text{m}$ ) | 46     | 38      |

## DISCUSSION

THM-treatment of Norway spruce and beech increased the density of both species much higher than that of most central European wood species. The differences in values for compression set and density between two species can be explained by structural dissimilarities of two selected species (higher resilience of the hardwood) and a unique nature of every single test specimen. Compared to the strong impact of the temperature during plasticization and compressive force during densification phase on the density of the final product, the influence of post-treatment conditions on density was not significant. The impact of temperature, duration and degree of steam saturation during the post-treatment phase was more important for the elimination of shape memory than any other step of the THM procedure. Post-treatment at 180°C appears to be most

conducive for elimination of the spring-back effect for both species with respect to mechanical requirements and constant strength properties of the densified wood product.

The spring-back effect of THM-densified wood decreased significantly with elevated post-treatment temperatures and shorter treatment durations, which is in good agreement with results of Tabarsa and Chui (2000), Heger (2004), Welzbacher and Rapp (2007). A comparable elimination of the shape memory and hence improved dimensional stability after steam post-treatment at temperatures exceeding 180°C has been reported by several authors in the past (Inoue et al. 1993, Dwianto et al. 1996, Ito et al. 1998b). The mechanism by which steam heated treatment fixes the compressive set is thought to be due to softening of lignin combined with degradation of amorphous polysaccharide content (Morsing 2000). Dimensional stabilisation is most likely caused by increasing cross linkage and relaxation of stored stresses by partial hydrolysis of hemicelluloses and degradation of lignin at elevated temperatures (Norimoto et al. 1993, Navi and Heger 2005). However, macrobuckling and damage of the wooden structure in densified and not post-treated specimens imparted wood cells being unable to recover completely to the initial dimensions (44% and 61% compression set recovery for beech and spruce wood, Tab. 2).

Our study has shown that hygroscopicity of THM-densified specimens is markedly reduced as a result of modification by steam post treatment. This reduction is related to the temperature of the process. Other studies indicate that the time and treatment atmosphere during the process also affect sorption behaviour (Alexiou et al. 1990, Poblete et al. 2005, Popper et al. 2005, Welzbacher et al. 2008). In steam post-treatments the inserted moisture contributes to the hydrolysis of the paracrystalline regions of celluloses (Ito et al. 1998a) and furthermore cause inner stress relaxation by hydrolysis of hemicelluloses that results in fixation of compression set (Heger et al. 2003). Navi and Heger (2005) have calculated the amount of –OH groups using the Dent model and showed that half of the –OH groups were suppressed after TH-post-treatment at 180°C during 16 min making the wood less hydrophilic.

Swelling of THM-densified beech and Norway spruce wood was significantly reduced by implementation of steam post-treatment. However, the lowest swelling was recorded for TH-treated beech and spruce specimens (TH 160 and TH 180), which is related to the fact that these specimens were not densified and hence did not exhibit any set recovery, that contributed to the swelling behaviour especially in the radial direction (the direction of compression). Steam post-treatment, in contrast to a dry post-treatment (in the environment excluding any contact with moisture) or oil-heat treatment (Treu et al. 2003), contributes to the fixation of compression set at lower temperatures and shorter durations, which is reflected by the energy demands for the production and consequently on the cost of the final product.

Reduction of MOE values in TH-treated specimens are in good agreement with the work of Yilgor et al. (2001) where compression strength decreased by 13.3% and MOE by 16.5%. However, many studies have shown that there is a slight increase in MOE when wood is thermally treated for short periods. In a study on the thermal degradation of beech and pine at various temperatures and time periods, it was found that although the reduction in strength and work to maximum load was proportionate to mass loss, irrespective of treatments and species reduction in MOE only became significant after mass losses exceeded 8% (Rusche 1973). In studies of Kubojima et al. (2000) where wood specimens were heated in a sealed reactor at 160°C under nitrogen or air for various time periods, MOE increased over short treatment periods and then remained relatively constant. Also Bekhta and Niemz (2003) found little change in MOE for spruce wood heated at 200°C. Chang and Keith (1978) reported on the increase of MOE for wood samples (elm, beech, aspen and maple) after thermal treatment, but also noted that more severe treatments resulted in a reduction of MOE. Radial compression of Norway spruce and beech, on the other hand, increased the density

and resulted in a dramatic increase in MOE. The same trend has been reported by Kubojima et al. (2004) who assessed the MOE and shear modulus of densified Japanese cedar (*Cryptomeria japonica* D. Don) in the free-free flexural vibration test. The noticeable increase of the MOE was probably due to the increase of the relative amount of crystalline cellulose after THM-densification, although degradation and modification of the hemicelluloses might also have an effect on MOE. Moreover, due to the anisotropic character of crystalline cellulose, the compressive strength is limited in the radial and tangential direction. The slight decrease of the radial compression strength after TH-treatment may be caused by small radial fissures. However, an approx. 25-fold dramatic increase in compressive strength perpendicular to the grain (direction of densification) for Norway spruce could be due to the careful selection of the specimens and hence an exceptional efficiency of densification. In the study of Beaud et al. (2008a, b, c) with the industrial scale densification of Norway spruce wood panels (dimensions: 1 m x 3 m x 10 cm), test specimens showed a 5-fold increase of compressive strength in the radial direction having a compression set of approx. 50% and density between 0.88 and 0.92 g.cm<sup>-3</sup>.

## CONCLUSIONS

The THM-densification procedure allows the compression of wood specimens in the radial direction to approx. 70% (Norway spruce) and 45% (beech) of their initial size inducing an increase in density of approx. 1.3 g.cm<sup>-3</sup> and 1.25 g.cm<sup>-3</sup> for spruce and beech respectively. Increase in density of the wood specimens induces changes in the mechanical and wood strength properties. Moreover, TH-post-treatment slightly alters wood chemistry and individual wood constituents contributing to changes in the mechanical properties (reduction in HB, MOE and compressive strength) and an improvement of dimensional stability.

The compression-set recovery of THM-densified specimens was not completely eliminated by the TH-post-treatment for 35 min and at temperatures not exceeding 180°C. TH- and THM-treatments reduced hygroscopicity of beech and spruce wood specimens. Increasing temperatures applied during the post-treatment phase led to a reduction of EMC and decreased the rate of moisture-uptake.

Results obtained in the present study indicate the importance of the TH-post-treatment phase, although the set-recovery was significantly reduced, it was not completely eliminated, causing re-expansion of cell lumina as the humidity in the environment increased. The fixation of the compressed wood structure and hence, the elimination of spring back is presumably a result of thermal modification of the hygroscopic components in the cell wall.

It is apparent that TH-treatment has an adverse effect on the strength properties of wood as the mechanical properties are affected. However, densification in combination with TH-post-treatment improved the mechanical performance of both wood species tested, rendering wood more rigid but less elastic.

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