Annals of Warsaw University of Life Sciences – SGGW
Forestry and Wood Technology No 75, 2011: 19-25
(Ann. WULS-SGGW, Forestry and Wood Technology 75, 2011)

Influence of high pressing temperature on dimensional stability of beech wood.

JOZEF KÚDELA, MAREK REŠETKA

Faculty of Wood Science and Technology, Technical University in Zvolen, Slovak Republic

Abstract. This work was aimed at determining the influence of high pressing temperatures (180–220 °C) and pressing time on dimensional stability of beech wood specimens. The specimens, whose initial moisture content was 17 %, were pressed in tangential direction. The results show that the dimensional stability significantly improved with increasing pressing temperature and time. Under the given conditions, the optimum temperature was 200 °C and the optimum heating time was 6 minutes.

Keywords: pressing, dimensional stability, beech wood, temperature, pressing time

INTRODUCTION

Native wood is pressed with the purpose to increase its density, to improve its mechanical properties, and to shape its relief. One of advantages of the pressed wood is its dimensional stability. This stability is significantly related to the wood's moisture content, temperature, pressing time, pressing pressure or the degree of compression.

Former works, such as CHUCHRJANSKIJ (1953), STAMM AND SEBORG (1941) and SEBORG *et al*. (1962) show that wood with a moisture content below 13 %, pressed at 140°, suffered from lacking stability. After repeated wetting and heating, it turned almost to the original state. To ensure the dimensional stability of pressed wood, the wood was fixed also chemically, physically or mechanically (ITO *et al*. 1998a, b, JNOUE *et al*. 1998, DWIANTO *et al*. 1999, HIGASHIHARA *et al*. 2000, NAVI a GIRARDET 2000, REINPRECHT and VIDHOLDOVÁ 2011 and others). Another cause for re-starting the research was in emerging new possibilities for exploring the changes to wood induced by heat and moisture.

ITO *et al.* (1998) obtained, after preliminary plasticization, high dimensional stability for wood pressing temperatures above 180 ºC, while the wood stability was also significantly affected by pressing temperature at the given temperature. JOHANSSON *et al*. (2006) report that wood heat-treated at varying temperatures reached the lowest sorption capacity at 200 ºC. BÄCHLE (2007) documents an even ca 50% reduction of equilibrium moisture content of food subjected to heat treatment. Reduction of wood sorption capacity results in improving dimensional stability. INOUE *et al.* (1998) fixing pressed wood by high-frequency heating, reduced, in such a way, its instant reversible deformation significantly. The referred works show that the wood's dimensional stability is significantly influenced by interaction between moisture content and temperature. The effect of temperature is pronounced under high moisture content.

NAVI and GIRARDET (2000) show that better results can be obtained for beech wood pressed at 150 °C in saturated steam (thermo-hydro-mechanical treatment – THM) than for similar wood pressed at low moisture contents (thermo-mechanical treatment – TM). The authors also demonstrated that, in comparison with compressed wood, THM compressed wood exhibits markedly reduced sorption capacity and better dimensional stability after repeated wetting in water. There is evidence for a range of other factors improving the dimensional stability in THM beech wood. Under given conditions, the destruction of lignin-carbohydrate matrix is more advanced, and after removal of the moisture and heat load, the matrix molecules are cross-linked by restoration of hydrogen bonds distorted by wood plasticization and shaping. The stresses in the matrix are relaxed; the hygrophilous cell wall

components (primarily hemicelluloses) form polymers more resistant against water. There were also observed differences in forming cell elements. On the other hand, high wood moisture content has negative impacts – for higher pressing temperatures, a moisture content range of 15–20 % is recommended.

The results of the cited works do not allow setting the optimum moisture content and temperature for wood during pressing, due to the presence of a range of other factors. KÚDELA (2005), CLAIR *et al.* (2003) and ETEVES and PEREIRA (2009) show that molecular mechanisms underlying changes in wood is high-variable and high-complex. Several changes to wood structure have been explained satisfyingly (changes to chemical structure, degradation of hemicelluloses and of amorphous cellulose, lignin networking, reduction of the amount of hydroxyl groups followed by changes in sorption properties), many several, nevertheless, are still unanswered questions (CLAIR *et al.* 2003).

The aim of our work was to find out, by experiments, the influence of temperature ranging 160–220 ºC on the dimensional stability of beech wood after pressing followed by conditioning, and to determine the optimum value of pressing temperature under the given conditions.

MATERIÁL A METODIKA

The experiments were carried out on beech wood test specimens with dimensions of 50 × 50 × 20 mm (R × L × T) – Fig. 1. The pressing direction was decided as to follow CHUCHRJANSKYJ (1953), PERELYGIN (1965) and KÚDELA (1990), who report for beech wood better compressibility in tangential direction – due to a high portion of pith rays.

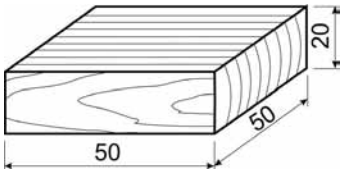

Fig. 1 Dimensions and shape of test specimens

The test specimens were conditioned at a relative air humidity of 80 % and a temperature of 20 °C, corresponding to an equilibrium moisture content of 17 %. The acclimated specimens were pressed in testing compression equipment. Before the pressing, the specimens were weighed with a precision of 0.01 g and measured with a precision of 0.01 mm.

The pressing appliance was equipped with two pressing plates, with controlled heating. The heating was electric, controlled with a thermostat whose gauge was inserted directly in the pressing plate. For the pressing process, we chose four temperature regimens: 160, 180, 200 and 220 °C and three pressing periods: 2, 4 and 6 minutes. The specimens were pressed by 40 %. This extent was obtained with the aid of restraining metal mats with the thickness corresponding to the desired compression extent. By combining these factors, we obtained 12 different pressing regimens. For each regimen, there were used 10 specimens, altogether 120.

After picking from the equipment, the specimens were weighed and measured with the same precision as above. Then they were placed in a conditioning box with a relative air humidity of 65 % and a temperature of 20 °C until reaching the equilibrium state. The conditioned specimens were weighed and measured again. Finally, the specimens were dried out to zero moisture content – to obtain the dry mass m_0.

The dimensional change was determined based on the change in the test specimen's thickness in pressing direction, immediately after the removal from the pressing equipment, according to the following equation:

$$D_1 = \frac{H_1 - H_0}{H_0} \cdot 100 \qquad (1)$$

and after the acclimation of the test specimens in an environment with parameters (φ, t), according to the equation

$$D_2 = \frac{H_{2(3)} - H_0}{H_0} \cdot 100, \qquad (2)$$

where H_0 is the thickness of specimen after compression (12 mm), H_1 – is the thickness of specimen removed from the pressing equipment, $H_{2(3)}$ – is thickness of specimen after acclimation at φ = 65 % (80 %).

In accordance with the standards 490103, we determined the moisture content before pressing, after pressing and after repeated acclimation.

RESULTS AD DISCUSSION

The average moisture content of the test specimens before the pressing was 17 % (Table 1). This moisture content was chosen based on the results of our former experiments. The conditioned specimens were pressed under conditions described in the methods. Mechanical, moisture and heat loading in their interactions, posed on the specimens in pressing, caused instant as well as permanent changes in wood structure, which means also changes in its properties. The immediate changes in wood properties influenced the wood compression during the pressing process itself. The permanent modifications of wood structure and properties had impact on its dimensional stability.

Table 1 Moisture content in specimens, corresponding to the phases of experiment (n =10)

Pressing time [min.]	Statistical character. m.c.	Temperature [°C]			
		160	**180**	**200**	**220**
Conditioning before pressing (φ = 80 %, t = 20 °C)					
	$\bar{x}$ [%]	17,02			
	s [%]	0,15			
After pressing					
2	$\bar{x}$ [%]	10.68	9.85	6.92	5.16
	s [%]	0.68	1.00	0.70	0.57
4	$\bar{x}$ [%]	7.33	6.69	3.25	1.55
	s [%]	0.64	0.76	0.56	0.42
6	$\bar{x}$ [%]	5.08	3.36	1.07	–
	s [%]	0.95	0.69	0.48	–
I-st conditioning after pressing (φ = 65 %, t = 20 °C)					
2	$\bar{x}$ [%]	11.08	10.25	9.69	8.94
	s [%]	0.52	0.53	0.23	0.30
4	$\bar{x}$ [%]	9.94	9.52	8.75	8.22
	s [%]	0.20	0.44	0.19	0.26
6	$\bar{x}$ [%]	9.20	8.40	8.41	–
	s [%]	0.16	0.16	0.16	–
II-nd conditioning after pressing (φ = 80 %, t = 20 °C)					
2	$\bar{x}$ [%]	16.71	16.32	16.25	15.72
	s [%]	0.51	0.48	0.22	0.35
4	$\bar{x}$ [%]	16.34	15.84	15.95	15.38
	s [%]	0.42	0.33	0.33	0.38
6	$\bar{x}$ [%]	15.85	15.35	15.85	–
	s [%]	0.53	0.56	0.37	–

The moisture content of the test specimens after pressing decreased from 11 to 1 % (Table 1). For all specimens, the testing has confirmed an important decrease in the moisture content in the pressing process. The final moisture content during pressing depended on the pressing temperature and time. Moisture content was found markedly decreasing with decreasing density; the influence of pressing period has been confirmed, too. The longer was the influence of temperature on the pressed specimens; the lower was the moisture content in these specimens.

The test specimens were pressed to a constant thickness of 12 mm (40% compression). After removing from the pressing equipment, the springback of specimens was determined according to the Eq. (1). We also monitored the changes in specimen thickness after conditioning at φ = 65 %, followed by further conditioning at 80 %. The results are represented in Fig. 2.

The dimensional stability of specimens after pressing correlated with varying moisture content. The lowest dimensional stability was observed at a pressing temperature of 160 °C and a pressing time of 2 min. In this case, the specimens were drought-up to a lower extent. The corresponding springback was 27 %. The springback for the given pressing time decreased linearly with increasing temperature, reaching at 220 °C almost one-third value (10 %). The springback also decreased significantly with prolonged pressing

time. Fig. 2a shows that at 180 °C and 6 min. reached the springback almost zero (the specimen's dimensions were fixed). At pressing temperature 220 °C, the zero springback was reached already after 4 min. pressing. The perfect dimensional stability of beech specimens at 17 % m. c. was attained already after pressing at 180 °C. To reach the full effect, the temperature needs to affect across the whole cross-section, for 6 min. at minimum. The heating time can be reduced by increasing the heating temperature. Considering additional properties, first of all the wood surface colour, the most appropriate pressing temperature and time seem 200 °C and 6 min. The temperature of 220 °C is associated with a high risk of considerable heat degradation of wood surface layers.

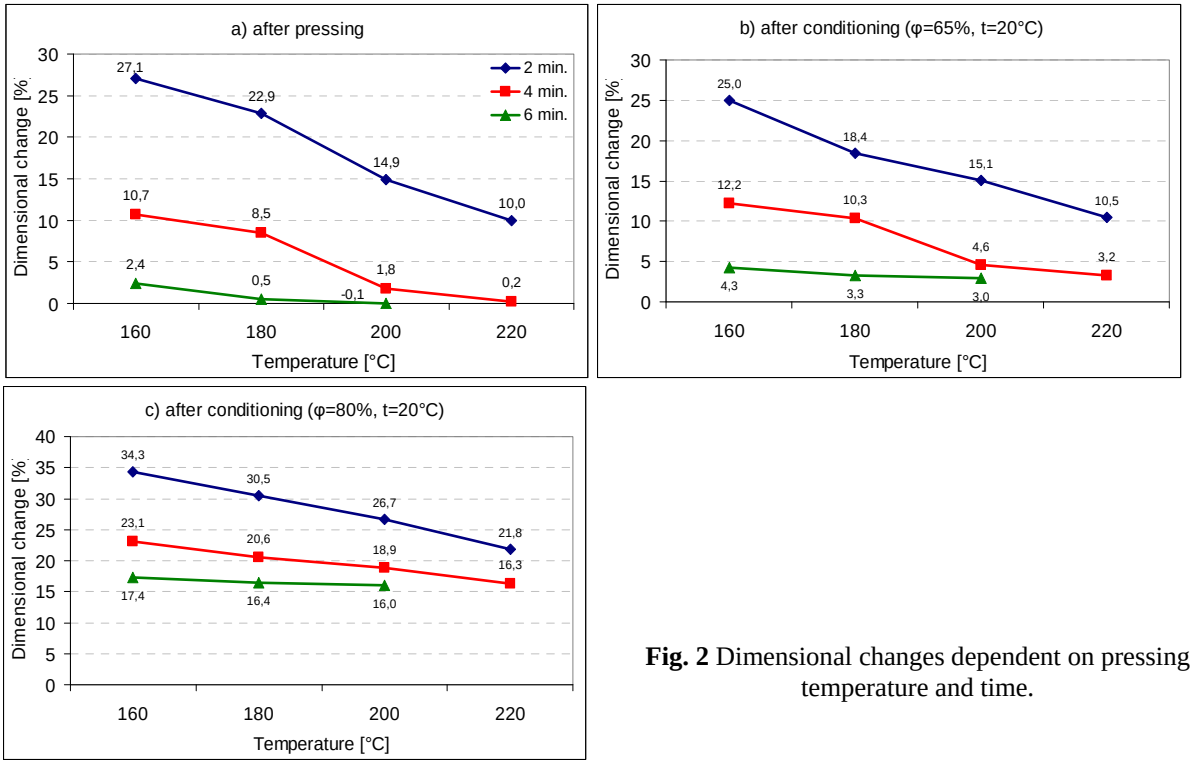

Fig. 2 Dimensional changes dependent on pressing temperature and time.

The pressed specimens were conditioned at a relative air humidity φ = 65 % and temperature t = 20 °C. These values are responded by an equilibrium wood moisture content of 12 %. The moisture content after conditioning ranged from 11 to 8 % (Table 1). Increasing temperature and prolonged pressing time had reducing effect on equilibrium moisture content. Pressing under specific moisture content partly reduced the beech wood sorption capacity.

After pressing at φ = 65 %, the dimensional changes were more pronounced, and they correlated with the varying moisture content (Fig. 2b). The following acclimation at φ = 80 % increased the moisture content in specimens significantly, close to the equilibrium moisture content of 17 %. The dimensional changes, however, were less discernible (Fig. 2c). The biggest change in thickness (by 1/3) was recorded for a pressing temperature of 160 °C and a pressing time of 2 min. With increasing pressing temperature and time, the dimensional stability improved. In the both cases, the best results were obtained for 220 °C and 4 min.; and for 180–200 °C and 6 min. In this cases, the backspring values were ca 3 % (conditioning at φ = 65 %), and ca 16 % (conditioning at φ = 80 %).

Several-fold compression of wood (compared to its shrinkage associated with moisture content varying in pressing process) results in extensive permanent mechanical strains. The springback after removing the load is assigned to elastic strains (immediate and elastic in time) as well as restoration of hydrogen bonds. The restoration of hydrogen bonds entails

reorganisations at the macromolecular and sub-microscopic structural level. The result is hygroscopic deformation – wood "swelling". The larger amounts of water in wood after pressing, the more pronounced these changes.

From the results it follows that these deformations can be influenced by temperature. The temperature-driven molecular mechanism of the changes in wood structure is different from the one driven by moisture content. When wood is heated, mechanical movements of its basic structural elements (heat movements) are intensified, which is connected with significant physical and chemical changes in the material (KAČÍKOVÁ and KAČÍK 2011).

Wood polymers are in general characterised by three temperature regions and three corresponding phases: glassy, viscous-elastic (transition area) and viscous flow (rubbery state) (KÚDELA 1992). IRVINE (1984) gives, for some polymers, even five temperature intervals. Five temperature ranges can also be identified for some lignins in broadleaved wood species (KUBO *et al.* 1997). The ranges differ in the polymers properties and performance. From this viewpoint is important T_g temperature (glass transition).

For dry hemicelluloses and lignin, T_g > 100° C (IRVINE 1984, SOLÁR 1997, OLSON and SALMÉN 1997 and other). However, the last cited works indicate that the moisture content exerts the chief controlling effect on T_g. The temperature T_g in wet lignin ranges from 60 to 100° C, in hemicelluloses from 0° to 100° C – depending on moisture content. With increasing moisture content, the difference in T_g between these two components increases. The underlying cause is also the high hygroscopic and the highest water-absorbing capacity of hemicelluloses in wood.

In cellulose, within the discussed temperature area, we do not observe T_g, due to the highly crystallised structure of this material. WOODWARD (1980) reports for cellulose T_g temperature of 230° C. Several authors cited in LINDSTRÖM *et al.* (1987) give for T_g in dry cellulose values about 200 °C, decreasing with increasing temperature.

The glass transition temperature for wood after plasticization has been primarily put in relation with temperature T_g of lignin. In case of wet beech wood, T_g is ranging 65–75 °C (OLSSON and SALMÉN 1997). The last cited authors suggest that the low T_g values in beech wood are mostly due to the high presence of methoxyl groups in lignin. From KÚDELA (2005), it follows that the temperature T_g may be to some extent influenced by the duration of heat or hydro-thermal treatment.

Obtaining rubbery state of lignin in wood pressing requires much higher temperature (180–200 °C). It is in accordance with ITA *et al.* (1998b), INOUE *et al.* 1998 and DWIANTA *et al.* 1999.

We were working with an initial moisture content of 17 % (air-dried wood). The wood moisture content decreased significantly during pressing. Considering these two facts, we choose for pressing temperature 160–220 °C. These parameters should provide guaranty that polymers in wood under pressing maintain within the third transition phase – rubbery.

The results have revealed that, from the viewpoint of permanent changes, in our case, for 20 mm thick specimens, the optimal pressing temperature and time seem 200–220 °C and 4–6 min. For these pressing parameters, there was recorded almost 100% dimensional stability after pressing, as well as good stability after conditioning. The obtained results are showing for evidence that the studied wood polymers were, under the given pressing parameters, in rubbery state (mainly lignin). In or opinion, this fact played a crucial role for specimen dimensional stability.

Similar results were obtained by ITO *et al.* (1998a), studying the mechanism of permanent fixation of compressed wood. The authors have come to conclusion that higher pressing temperatures could reduce the pressing time. At 200 °C, the specimen required for stabilisation 4min pressing time; at 180 °C, the same stabilisation required three times longer.

CONCLUSIONS

The experimental results have confirmed important influence of pressing temperature and time on dimensional stability. The dimensional stability was found significantly improved with increasing pressing temperature and time.

The perfect stability after pressing was reached at 200 °C temperature and 6min pressing time, and at 220 °C and 4 min. Under these pressing conditions, the sorption capacity of the pressed wood was reduced significantly.

Acknowledgement: We highly acknowledge the support from the Scientific Grand Agency of the Ministry of Education SR and the Slovak Academy of Sciences (Grant No. 1/0565/10). We thank also Dipl.Ing. Ivan Figuli for assistance with experimental works.

LITERATURE

1. BÄCHLE, F., NIEMZ,P., SCHNEIDER, T.: 2007. Physical-mechanical properties of hard- and softwood heat treated in an Autoclave. In.: European Conference on Wood Modification 2007., Bangor, UK, BC, pp. 177—182.
2. CLAIR, B., ARINERO, R., LÉVÈQUE, G., RAMONDA, M., THIBAUT, B., 2003. Imaging the mechanical properties of wood cell wall layers by atomic force modulation microscopy. IAWA Journal, *24*(3):223–230.
3. DWIANTO, W., MOROOKA, T., NORIMOTO, M., KITAJIMA, T., 1999. Stress relaxation of sugi (*Cryptomeria japonica*, D. Don) wood in radial compression under high temperature stem Holzforschung, *53*:541–546.
4. ESTEVES, B. M., PEREIRA, H. M., 2009. Wood modification by heat treatment. Bio Resourses, *4*(3): 370–404.
5. HIGASHIHARA, T., MOROOKA, T., NORIMOTO, M., 2000. Permanent fixation of transversally compressed wood by steaming and its mechanism. J. Jap. Wood Res. Soc., *46*(4):291–297.
6. CHUCHRJANSKIJ, P. N., 1953. Lisovanie dreva. Bratislava: Práca 156 pp.
7. INOUE, M., KODAMA, J., YAMAMOTO, Y., NORIMOTO, M., 1998. Dimensional stabilization of compressed wood using high-frequency heating. J. Jap. Wood Res. Soc., *44*(6):410–416.
8. IRVINE, G. M., 1984: The significance of the glass transition of lignin and hemicelluloses and their measurement by differential thermal analysis. Tappi J., *67*(5):118–121.
9. ITO, Y., TANAHASHI, M., SHIGEMATSU, M., SHINODA, Y., OHTA, CH., 1998a: Compressive-moulding of wood high-pressure steam-treatment. Part 1. Development of Compressively moulded squares from thinnings. Holzforschung, *52*(2):211–216.
10. ITO, Y., TANAHASHI, M., SHIGEMATSU, M., SHINODA, Y., 1998b: Compressive-moulding of wood high-pressure steam-treatment. Part 2. Mechanism of permanent fixation. Holzforschung, *52*(2):217–221.
11. JOHANSSON, D., PERSSON, M., MORÉN, T., 2006: Effect of heat treatment on capillary water absorption of heat/treated pine, spruce and birch. In.: Wood structure and propetries ´06. Zvolen, Arbora publishers, pp. 251–255.
12. KAČÍKOVÁ, D., KAČÍK, F., 2011: Chemické a mechanické zmeny dreva pri termickej úprave. Zvolen, TU vo Zvolene, 71 pp.
13. KUBO, S., ISHIKAVA, N., URAKI, Y., SANO, Y., 1997: Preparation of lignin fibers from softwood acetic acid lignin. Relationship between fusibility and the chemical structure of lignin. Mokuzai Gakkaishi, *43*(8): 655−662.

14. KÚDELA, J., 1990: Vplyv vlhkosti a teploty na mechanické vlastnosti bukového dreva. [Kandidátska dizertačná práca.] Zvolen , DF Technickej univerzity, 150 pp.
15. KÚDELA, J. 1992: Štúdium vplyvu teploty na vlastnosti dreva. In.: Wood burning '92. Vysoké Tatry, Technická univerzita vo Zvolene, pp. 201–222.
16. KÚDELA, J., 2005: Vlhkostné a tepelné namáhanie bukového dreva. Zvolen, TU vo Zvolene, 2005, 141 pp.
17. LINDSTRÖM, T., TULONEN, J., KOLSETH, P., 1987: Swelling and mechanical properties of cellulose hydrogels. Part VI. Dynamic mechanical properties. Holzforschung, *41*(4): 225–230.
18. NAVI, P., GIRARDET, F., 2000: Effect of thermo-hydro-mechanical treatment on the structure and properties of wood. Holzforschung, 54(3):287–293.
19. OLSON, A. M., SALMÉN, L., 1997: Humidity and temperature affecting hemicellulose softening of wood. In.: Mechanical performance of wood and wood products – International Conference of COST Action E8, Copenhagen, pp. 267–279.
20. PERELYGIN, L. M., 1965: Náuka o dreve. Bratislava, SNTL, 448 pp.
21. REINPRECHT, L., VIDHOLDOVÁ, Z., 2011: Termodrevo. Ostrava, ŠMÍRA-PRINT, s.r.o., 89 pp.
22. SEBORG, R. M., MILLETT, M. A., STAMM, A. J., 1962: Heat-stabilized compressed wood (Staypak). FPL, Report No. 1580 (revised), 22 pp.
23. SOLÁR, R., 1997: Zmeny lignínu v procesoch hydrotermickej úpravy. Zvolen, TU vo Zvolene, 57 pp.
24. STAMM, A. J., SEBORG, R. M., 1941:. Resin treated laminated, compressed wood. Trans. Am. Inst. Chem. Eng., 37: 385–397.
25. WOODWARD, C., 1980: Fractured surfaces as indicators of cell wall behaviour at elevated temperatures. Wood Sci., 13(2):83–96.

Streszczenie: *Wpływ prasowania wysokotemperaturowego na stabilność wymiarową drewna buka.* Praca opisuje wpływ prasowania wysokotemperaturowego (180–220 °C) i czasu prasowania na stabilność wymiarową drewna bukowego. Próbki, o wyjściowej wilgotności 17%, były prasowane w kierunku stycznym. Wykazano że stabilność wymiarowa znacząco zwiększyła się wraz ze zwiększoną temperaturą oraz czasem. Optymalnymi warunkami okazały się temperatura 200 °C i czas grzania 6 minut.

Corresponding author:

Prof. Dipl. Ing. Jozef Kúdela, CSc.
Dipl. Ing. Marek Rešetka
Department of Wood Science
Faculty of Wood Sciences and Technology
Technical University in Zvolen
T. G. Masaryka 24, 960 53 Zvolen
Slovak Republic
e-mail: kudela@vsld.tuzvo.sk
resetka.marek@gmail.com