The Influence of Process Parameters on the Density Profile and Hardness of Surface-densified Birch Wood (*Betula pendula* Roth)

Agnieszka Laskowska *

This study examined the influence of temperature and time of treatment on the density profile and hardness of surface-densified birch wood (*Betula pendula* Roth). An analysis of the wood density profile was conducted on the basis of the following parameters: thickness, maximum density, and the distance between the maximum density and the wood surface. Depending on the technological parameters' values, the degree of compression of the wood was 13% to 22%, and its maximum density was 808 kg/m^3 to 994 kg/m^3. As a result of the modification of birch wood at a temperature of 100 °C and 125 °C, the wood was densified on one side. As the temperature of the thermo-mechanical treatment was raised from 150 °C to 200 °C, the wood became densified on both sides. The maximum density of the wood increased gradually with the increase of the temperature of the press plate. The longer the time of thermo-mechanical treatment, the more distant the maximum density area was from the wood surface. Depending on the temperature and the time of treatment, the hardness of the surface-densified birch wood was 1.4 to 2.2 times greater than the hardness of non-densified wood.

Keywords: Birch; Thermo-mechanical modification; Densification; Density profile

*Contact information: Department of Wood Science and Wood Preservation, Faculty of Wood Technology, Warsaw University of Life Sciences - SGGW, Nowoursynowska Str. 159, 02-776 Warsaw, Poland; *Corresponding author: agnieszka_laskowska@sggw.pl*

INTRODUCTION

Density is an important property of wood that correlates with its physical and mechanical properties and, consequently, determines its applicability. Furthermore, wood, as a porous material that varies in terms of its morphological and chemical structure, displays various properties within and among particular types or species. An improvement in the physical and mechanical properties of wood can be achieved through densification. Therefore, low-density wood, *i.e.* wood with a density below 500 kg/m^3 is densified (Kutnar and Kamke 2012; Tu *et al.* 2014; Zhan and Avramidis 2016). Research on the densification of high-density wood, *e.g.* European beech, is also being conducted to obtain even higher mechanical properties, in particular regarding hardness (Blomberg *et al.* 2005; Rautkari *et al.* 2010; Gašparik *et al.* 2016).

The density of wood is the resultant of the proportions of pores and wood substance. The maximum density of wood that can be achieved as a result of densification is 1500 kg/m^3 (Wilfong 1966; Kellogg and Wangaard 1969). Material-related and technological factors influence the course of the process of wood densification. Due to the structure, including the chemical structure of wood, its densification is a very complex process. Individual components of wood are characterized by diversified softening temperatures (Irvine 1984; Wolcott *et al.* 1990). Moreover, cellulose and lignin show different rheological properties. The course of the densification process is determined by the type of wood, density, moisture of wood, the method of cutting specimens, *i.e.* the arrangement of annual growth rings in the wood cross-section, the thickness of the specimens, the pressing pressure, and the temperature and time of pressing (Ülker *et al.* 2012; Budakçı *et al.* 2016; Gašparík *et al.* 2016).

Wood can be densified by applying a thermo-mechanical (TM) or thermo-hydro-mechanical (THM) treatment, or viscoelastic thermal compression (VTC) (Kutnar *et al.* 2009), using the linear vibration friction technology (LVFT) (Rautkari *et al.* 2009). Wood can be densified in its whole volume (Ülker *et al.* 2012; İmirzi *et al.* 2014) or on the surface (Belt *et al.* 2013; Laine *et al.* 2013b; Rautkari *et al.* 2013; Kariz *et al.* 2017). The densification of wood's surface is a less energy-consuming process than the densification of the whole volume of wood. If wood is densified in its whole volume, a loss of wood can be observed. Applying such a solution is unjustified if the main goal of densification is to obtain materials with a high wood surface hardness, for example flooring materials or wall strips. It should be noted that the main problem involved in thermo-mechanical treatment of wood is a tendency for dimensions to change after the modification, most frequently referred to as "set-recovery" (Fang *et al.* 2012; Popescu *et al.* 2014; Laine *et al.* 2016). Kutnar *et al.* (2009) stated that wood with the highest degree of compression shows the highest potential for compression strain recovery. The surface densification of wood may be an alternative to the densification of the whole volume, which may contribute to a set-recovery reduction of wood.

The distribution of density in the cross-section is particularly important from the point of view of the correctness of the conducted densification process and the expected properties of the densified wood. Current literature on the subject does not provide a comprehensive characteristic of the density profile of surface-densified wood. Conducting research as part of the presented topics is justified as there are no models that would define, in a comprehensive manner, the nature of the changes that occur in wood during varied densification conditions, *i.e.* the parameters of the production process. Due to the complex structure of wood, the parameters of the thermo-mechanical treatment should be determined individually for each wood species. Because of the lack of data in previous literature, the subject of the study was birch wood (*Betula pendula* Roth). The main objective of the study was to determine the influence of the temperature and the time of treatment on the parameters of the density profile and hardness of surface-densified birch wood.

EXPERIMENTAL

Material

Birch wood samples (*Betula pendula* Roth, according to the EN 13556 (2003) standard BTXX) with the dimensions of 130 mm (longitudinal) × 80 mm (tangential) × 20 mm (radial) were used for the study. For each variant of thermo-mechanical modification 30 samples were used. The birch wood was obtained from a forest in northeastern Poland, located in Horodnianka (53°15′20″N 23°14′45″E), managed by the State Forests National Forest Holding. After the samples were conditioned in a normal climate (temperature 20 °C ± 2 °C, relative humidity 65% ± 5%) to an air-dry condition, the moisture content of the wood was determined according to ISO 13061-1 (2014). The moisture content of the wood subjected to thermo-mechanical densification was 6.48% (± 0.15%). The density of the wood was determined using the stereometric method in accordance with the ISO 13061-2 (2014) standard requirements.

Methods

The thermo-mechanical treatment of the wood comprised of two stages. At the first stage, the top surface of the wood was contact-heated in a hydraulic press and then densified at a unit pressure of 90 N/mm^2. The wood was cooled in a hydraulic press, whose plates were not heated, in a normal climate (temperature 20 °C ± 2 °C, relative humidity 65% ± 5%). The wood samples were cooled until the wood surface reached the temperature of 70 °C. Then, the samples were conditioned in a normal climate for 7 days.

Depending on the birch wood densification variant, the temperature of the upper plate of the press was from 100 °C to 200 °C. The parameters of the birch wood densification process are presented in Table 1.

Table 1. Birch Wood Surface Densification Parameters

Temperature of the Upper Plate (°C)	Heating Time (s)	Pressing Time (s)	Total Time of Thermo-mechanical Modification (s)
100	120	120	240
125	240	240	480
150	360	360	720
175			
200			

The distribution of density over the thickness of the wood samples before and after the densification was determined using a laboratory device for measuring density profiles (Laboratory Density Analyzer DA-X) manufactured by GreCon Inc. (Tigard, OR, USA), which determines the density using X-rays. The tests were conducted on samples with a length and width of 50 mm x 50 mm, at a measurement speed of 0.05 mm/s. Density values were measured every 0.02 mm of the thickness of the birch wood samples. The following parameters of the density profile were determined (Fig. 1): the minimum density (DMin); the maximum density on the left-hand side (DMaxL), *i.e.* the maximum density of the wood determined in the wood area whose surface was heated by the press plate; the maximum density on the right-hand side (DMaxR), *i.e.* the maximum density of the wood determined in the wood area whose surface was not heated by the press plate; the distance between the maximum density area and the wood surface on the left-hand side (ADMaxL), *i.e.* the distance between the maximum density area and the heated wood surface; the distance between the maximum density area and the wood surface on the right-hand side (ADMaxR), *i.e.* the distance between the maximum density area and the non-heated wood surface.

The compression ratio (CR) was calculated according to Eq. 1, where t_o is the original thickness (mm), and t_d is the thickness of wood after densification (mm).

$$CR = [(t_o - t_d) / t_o] \cdot 100 \text{ (\%)} \qquad (1)$$

The springback (S) of the densified wood was calculated according to Eq. 2 (Xu and Tang 2012; Pelit *et al.* 2016), where t_d is the thickness of wood after densification (mm), and t_r is the thickness after releasing (mm). The thickness t_r was measured after conditioning wood samples in a normal climate (temperature 20 °C ± 2 °C, relative humidity 65% ± 5%) for 7 days.

$$S = [(t_r - t_d) / t_d] \cdot 100 \text{ (\%)} \quad (2)$$

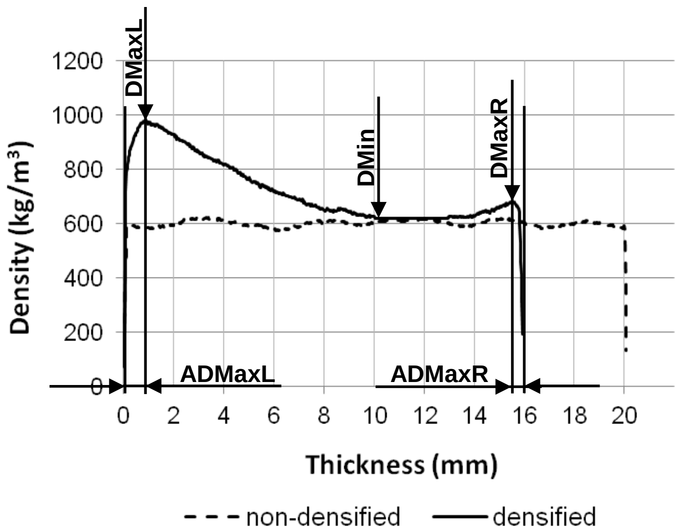

Fig. 1. Parameters of the density profile of surface densified birch wood.

The wood hardness was examined using the Brinell method in accordance with the requirements of EN 1534 (2010), after conditioning densified wood samples in a normal climate (temperature 20 °C ± 2 °C, relative humidity 65% ± 5%) for 7 days. The Brinell hardness was determined on the tangential surface of the sample. Hardness measurements were conducted using the universal testing machine CV-3000LDB manufactured by C.V. Instruments Ltd. (Sheffield, UK). The machine was equipped with an indenter with a 10-mm diameter. The maximum load was 1 kN. The wood hardness was determined for 30 samples of each variant of thermo-mechanical modification of birch wood. Statistical analysis was performed using STATISTICA Version-12 software of StatSoft, Inc. (Tulsa, USA). The statistical analysis of the results was performed at a significance level of 0.05.

RESULTS AND DISCUSSION

Examples of the density profiles of surface-densified birch wood at various levels of technological parameters are presented in Fig. 2. As a result of the modification of birch wood at a temperature of 100 °C and 125 °C, the wood was densified on one side. As the temperature of the thermo-mechanical treatment was raised from 150 °C to 200 °C, the wood became densified on both sides. The mean density of non-densified birch wood was 608 kg/m^3 ± 30 kg/m^3. This value was comparable to values cited in previous literature. According to Wagenführ (2007), birch wood density in an air-dry condition ranges from 510 kg/m^3 to 830 kg/m^3, with the mean value 650 kg/m^3. As a result of thermo-mechanical densification, the mean density of the wood (DMean) increased from 608 kg/m^3 to ca. 750 kg/m^3 in the case of modification conducted at a temperature of 200 °C.

The springback of the densified birch wood, regardless of temperature and time of thermo-mechanical modification, was found to be under 3%. Such low springback values resulted from the fact that the birch wood was surface-densified. Consequently, in contrast to volume-densified wood, only a part of the surface-densified wood could show a tendency for dimensions to change after the modification. Such interrelations were confirmed by literature data. Xu and Tang (2012) obtained under 2% springback for surface-densified plantation poplar wood. Such low springback values are among the typical characteristics of surface-densified wood.

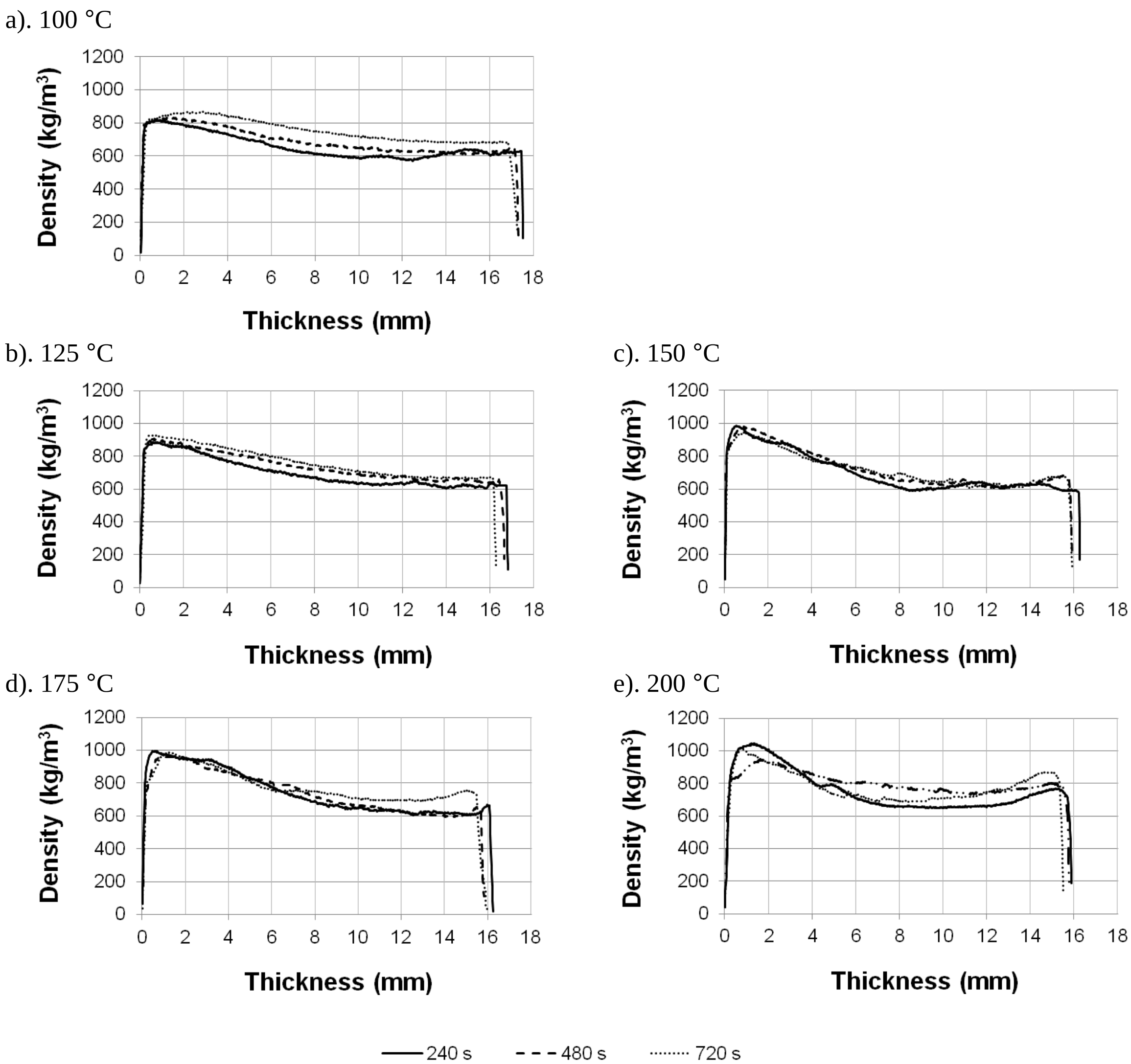

Fig. 2. Density profile of surface densified birch wood depending on the temperature of the press plate and the time of thermo-mechanical treatment

The statistical evaluation of the influence of factors on the parameters of the density profile and hardness of the birch wood is shown in Table 2. The temperature of the thermo-mechanical treatment showed a significant influence (based on ANOVA, Fischer's F-test) on all density profile parameters and on the hardness of birch wood (p <

0.05). The only parameter that was not significantly influenced by the time of thermo-mechanical treatment was DMaxL. The interaction between the temperature and the time of treatment significantly influenced the thickness, ADMaxL, ADMaxR, and hardness of birch wood.

When the percentage impact of factors on the properties of densified birch wood were analyzed, it is worth noting considerable differences in the operation of these factors (Table 2). The temperature of treatment influenced the parameters of the density profile and hardness of wood to a greater extent than the time of treatment. It should be noted that the temperature of treatment showed the greatest influence on the thickness of birch wood (84%), and the least significant (19%) on its hardness. The temperature and the interaction between the temperature and the time of treatment had an influence on ADMaxL and ADMaxR comparable between these parameters, which ranged from 34% to 44%. In contrast, the influence of the time of densification on the parameters of the profile and hardness of birch wood was minor and ranged from 3% to 21%. The greatest influence, in percentage terms, was recorded on ADMaxL and DMAxR, and the smallest on ADMaxR.

Table 2. Statistical Evaluation of the Factors Influencing the Parameters of Density Profile and Hardness of the Birch Wood

Properties	Factor	Sum of Squares	Degrees of Freedom	Variance	Fisher's F-test	Significance Level	Factor Influence (%)
		SS	Df	MS	F	p	
Thickness	Intercept	16004.05	1	16004.05	918600.0	0.000000	
	Temp. (1)	16.78	4	4.20	240.8	0.000000	84
	Time (2)	2.10	2	1.05	60.4	0.000000	11
	(1) × (2)	0.23	8	0.03	1.6	0.139379	1
	Error	0.78	45	0.02			4
ADMaxL	Intercept	70.02721	1	70.02721	1309.083	0.000000	
	Temp. (1)	10.61558	4	2.65389	49.612	0.000000	35
	Time (2)	6.35594	2	3.17797	59.409	0.000000	21
	(1) × (2)	10.65467	8	1.33183	24.897	0.000000	36
	Error	2.40720	45	0.05349			8
ADMaxR	Intercept	2503.476	1	2503.476	1256.779	0.000000	
	Temp. (1)	209.859	4	52.465	26.338	0.000000	44
	Time (2)	16.062	2	8.031	4.032	0.024515	3
	(1) × (2)	160.673	8	20.084	10.082	0.000000	34
	Error	89.639	45	1.992			19
DMaxL	Intercept	49927497	1	49927497	18571.97	0.000000	
	Temp. (1)	102822	4	25706	9.56	0.000011	41
	Time (2)	7442	2	3721	1.38	0.261022	3
	(1) × (2)	17434	8	2179	0.81	0.596878	7
	Error	120975	45	2688			49
DMaxR	Intercept	31188066	1	31188066	27797.20	0.000000	
	Temp. (1)	57216	4	14304	12.75	0.000001	39
	Time (2)	30834	2	15417	13.74	0.000022	21
	(1) × (2)	8578	8	1072	0.96	0.481915	6
	Error	50489	45	1122			34
Hardness	Intercept	170492.2	1	170492.2	3985.081	0.000000	
	Temp. (1)	782.1	4	195.5	4.570	0.003498	19
	Time (2)	458.7	2	229.4	5.361	0.008159	11
	(1) × (2)	969.0	8	121.1	2.831	0.012375	23
	Error	1925.2	45	42.8			47

The tests conducted show that the higher the temperature of the press plate was, the higher degree of wood compression achieved and, consequently, the smaller the thickness value of the sample (Fig. 3a). The degree of compression of wood densified at a temperature of 100 °C ranged from 13% to 14%, whereas in the case of wood densified at a temperature of 150 °C it ranged from 17% to 20%, and at a temperature of 200 °C from 20% to 22%. As a result, the densification of wood at a temperature of 100 °C in a time of 240 s led to obtaining samples with a thickness of 17.44 mm ± 0.11 mm. Where the temperature of the press plate was 200 °C and the treatment time 720 s, the samples reached a thickness of 15.50 mm ± 0.14 mm. The influence of the time of treatment on the thickness of densified wood was not as pronounced as the influence of the temperature of treatment (Fig. 3b).

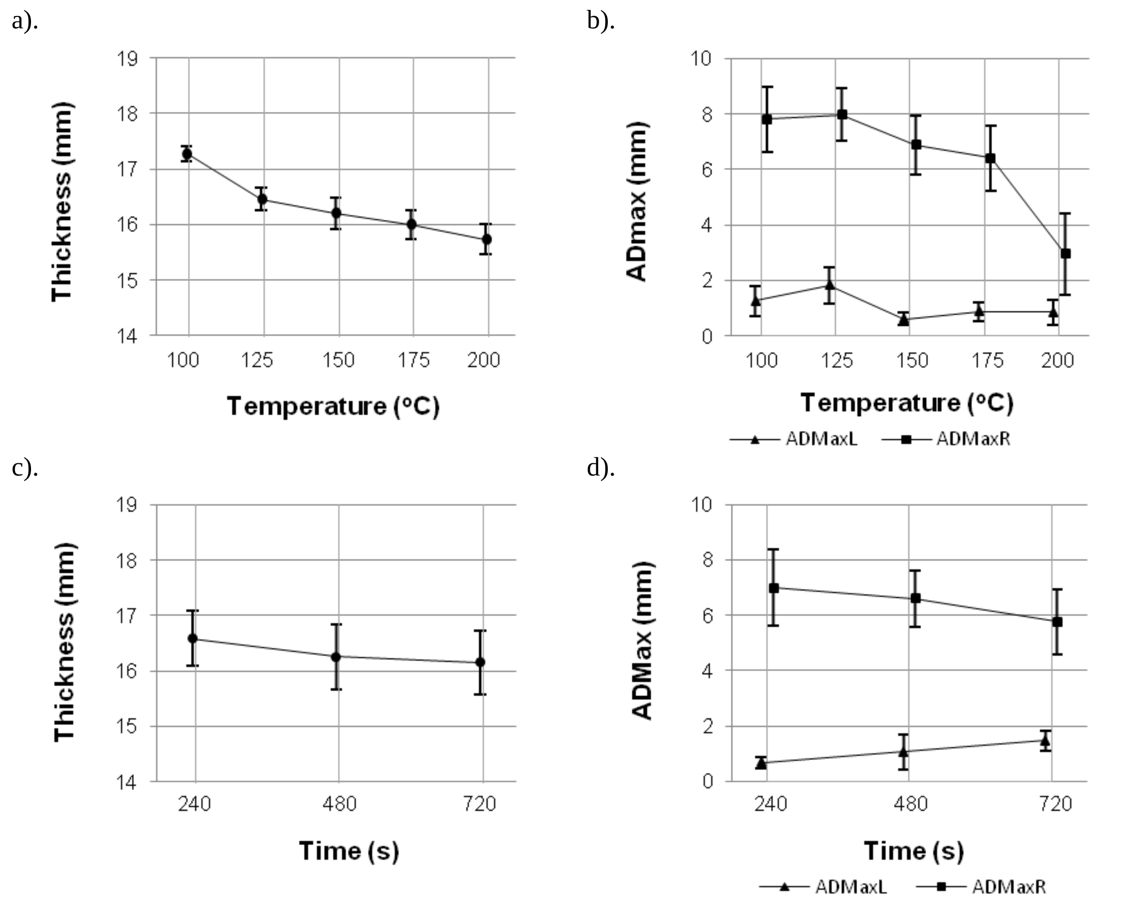

Fig. 3. Influence of the temperature and the time of thermo-mechanical treatment on the thickness, ADMaxL, and ADMaxR of birch wood

The longer the heating time and pressing of the wood, the bigger the distance between the maximum density area of the wood was from the side of the heated plate DMaxL and the wood surface. The mean value of ADMaxL after 240 s of treatment of birch wood was 0.69 mm ± 0.21 mm, whereas after 720 s it was 1.49 mm ± 0.36 mm (Fig. 3d). From the perspective of the user of wooden floors, it was important to be able to remove the worn out surface layer and to apply a new layer after a specific period of use of the floor. For this reason, it should be presumed that, in this case, it was justified to obtain materials characterized by a wide maximum density area DMaxL as a result of thermo-mechanical treatment. As a result of a longer treatment time, the wood tissue was plasticized to a greater extent and, consequently, the maximum density area DMaxL was “shifted” inwards. The longer the time and the temperature of treatment, the more thermal energy remained cumulated in the system. As a consequence, under the influence of the pressure and also the wood surface adhering directly, the non-heated plate of the press was densified. There might be some thermo-chemical reaction at 200 °C for the lignin. As a result, the maximum density area DMaxR was “shifted” closer to the surface of the wood sample. This was reflected in the decreased ADMaxR values as the temperature and the time of treatment increased (Figs. 3b, d). The mean value of ADMaxR after 240 s of treatment of birch wood was 7.00 mm ± 1.36 mm, whereas after 720 s it was 5.75 mm ± 1.18 mm.

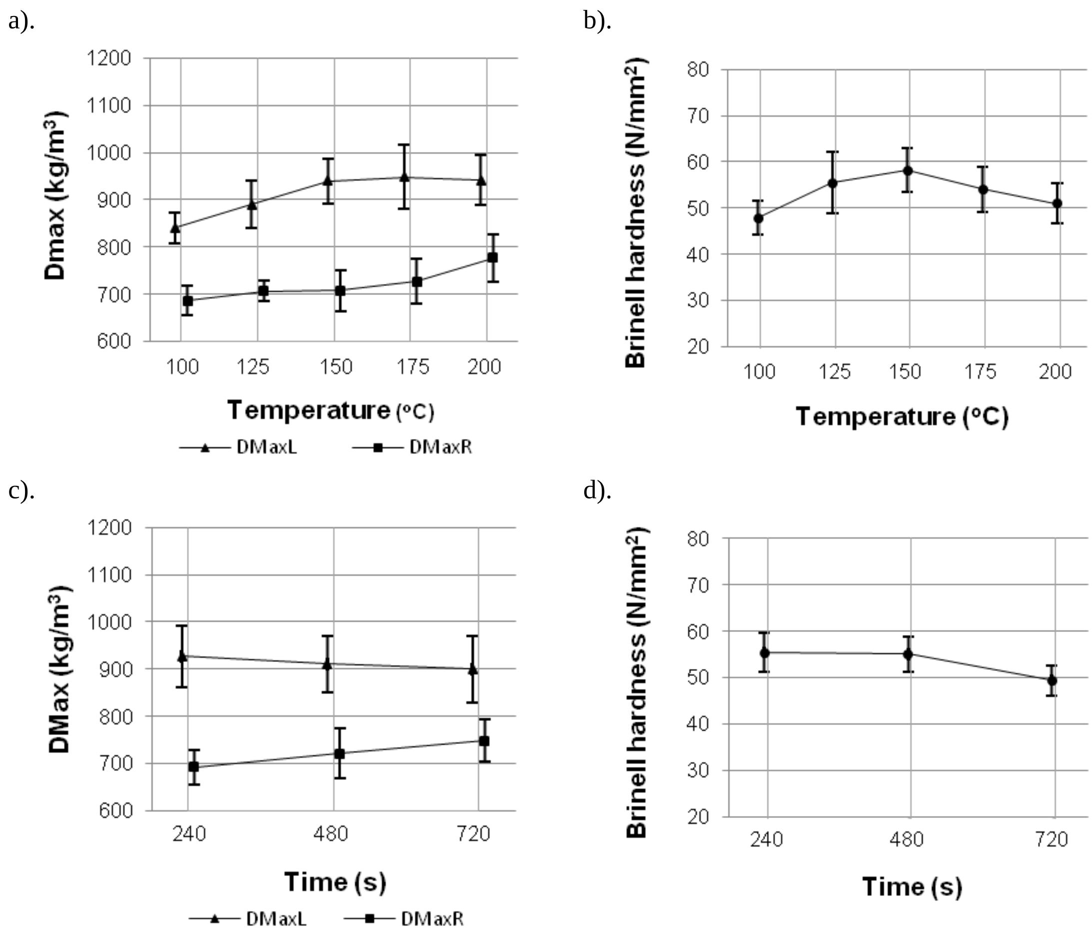

Fig. 4. Influence of the temperature and the time of thermo-mechanical treatment on the hardness, DMaxL, and DMaxR of birch wood

The most important values in the context of the density profile of surface-densified wood were the maximum density on the left-hand side (DMaxL), and the distance between this maximum density area and the wood surface (ADMaxL). The higher the temperature of the press plate, the greater were the values of DMaxL and DMaxR (Fig. 4a). The DMaxL of the surface-densified birch wood at a temperature of 100 °C was 841 ± 33 kg/m^3, which reached 941 kg/m^3 ± 53 kg/m^3 when the pressing temperature was 200 °C. It was approximately from 45% to 60% higher density than the surface density of non-densified birch wood. The DMaxR of the birch wood surface-densified at a temperature of 100 °C and 200 °C was 686 kg/m^3 ± 32 kg/m^3 and 777 kg/m^3 ± 50 kg/m^3, respectively. No significant DMaxL differences at a given temperature were found that depended on the treatment time. An inverse interdependence was found with DMaxR (Fig. 4c).

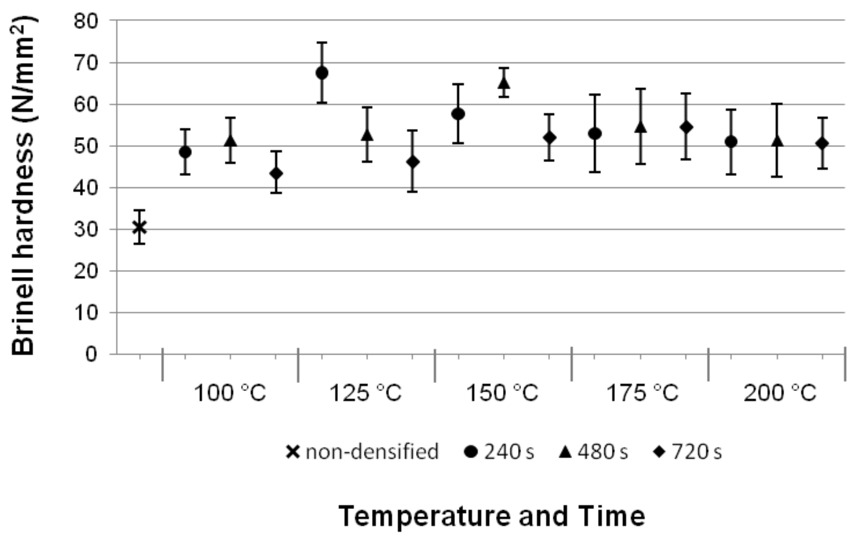

Fig. 5. Brinell hardness of surface-densified birch wood

The hardness of non-densified birch wood was 31 N/mm^2 ± 4 N/mm^2. This value was comparable to the data presented by Wagenführ (2007), according to whom the hardness of birch wood ranges from 22 N/mm^2 to 49 N/mm^2. The lowest hardness 44 N/mm^2 ± 5 N/mm^2 was recorded with birch wood densified at a temperature of 100 °C for 720 s. The greatest hardness was 68 N/mm^2 ± 7 N/mm^2, which was characterized by birch wood densified for 240 s at a temperature of the press plate of 125 °C (Fig. 5). Generally, it should be concluded that, depending on the temperature and the time of treatment, the hardness of surface-densified birch wood was from ca. 1.4 to 2.2 times greater than the hardness of non-densified wood and the differences were statistically significant ($p < 0.05$). The factors that had material influence on the hardness of birch wood were the temperature and the time of thermo-mechanical treatment, although this effect was not as great (Figs. 4b, d). The temperature and the time of treatment accounted for 30% of the variability in wood hardness, whereas the interaction between the temperature and the time of treatment accounted for 23% of the variability in wood hardness (Table 2).

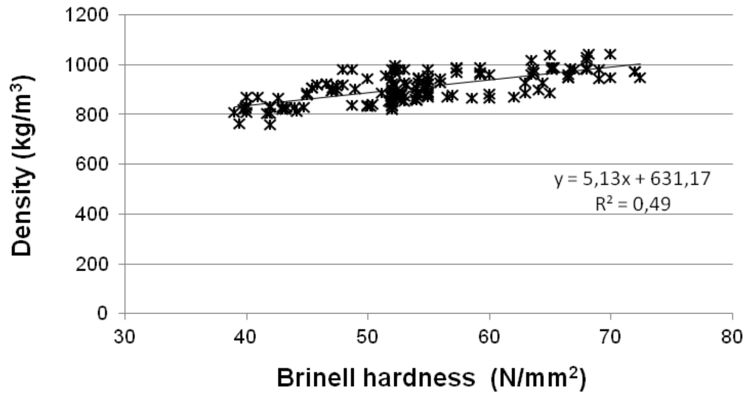

Fig. 6. Relationship between density and Brinell hardness of surface-densified birch wood

It was also found that the greater the DMaxL value, the greater the hardness of the densified birch wood (Fig. 6). However, no significant interdependence was found between the ADMaxL and the hardness of birch wood. This was due to the fact that when the intender was pressed into the sample, the wood surrounding the intender was deformed and entirely densified. Consequently, this additionally affects the density and hardness of the near-surface layer (Gašparík *et al.* 2016). As a result, the interdependence between DMaxL, ADMaxL, and the hardness of surface-densified wood was in some measure disturbed. Nevertheless, if wood was surface-densified in the course of the system being subjected to the operation of a high temperature for a long time, the non-heated wood layer was also densified. This results in a lesser degree of densification of the heated wood layer and its lower hardness value. Rautkari *et al.* (2011, 2013) and Laine *et al.* (2013a) stated that the holding time and pressing temperature result in a decrease in peak density, an increase in peak width, and an increase in peak distance. The effect is particularly strong if both parameters are increased at the same time.

CONCLUSIONS

1. The tests conducted have shown that the parameters of thermo-mechanical treatment have a significant influence on the properties of the density profile of birch wood. The thermo-mechanical treatment temperature had a significant influence on all density profile parameters and on the hardness of birch wood. The only parameter that was not significantly influenced by the time of thermo-mechanical treatment was the maximum density on the left-hand side (DMaxL).
2. Various birch wood density profiles have been obtained as a result of surface densification. When treatment temperatures of 100 °C and 125 °C were applied, wood was densified on “one side”. Along with an increase in the thermo-mechanical treatment temperature from 150 °C to 200 °C, the wood was densified on “both sides”.
3. The DMaxL of the surface densified birch wood at a temperature of 100 °C and 200 °C was 841 kg/m^3 ± 33 kg/m^3 and 941 kg/m^3 ± 53 kg/m^3, respectively. It was approximately from 45% to 60% higher density than the surface density of non-densified birch wood.

4. Depending on the temperature and the time of treatment, the hardness of the surface-densified birch wood was from ca. 1.4 to 2.2 times greater than the hardness of non-densified wood and the differences were statistically significant. The greatest hardness of 68 N/mm^2 ± 7 N/mm^2 characterized birch wood that was densified for 240 s at a press plate temperature of 125 °C.

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