Drewno 2020, Vol. 63, No. 205
DOI: 10.12841/wood.1644-3985.D06.08

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# DENSITY PROFILE AND HARDNESS OF THERMO-MECHANICALLY MODIFIED BEECH, OAK AND PINE WOOD

*Beech (Fagus sylvatica* L.*), oak (Quercus robur* L.*) and pine (Pinus sylvestris* L.*) wood were volume-densified by means of thermo-mechanical modification. At first stage the wood was heated in a hydraulic press at temperature 100°C for 720 s, and then one-step densified in order to obtain the target thickness. The wood was cooled in a hydraulic press with unheated plates. Density profiles parallel and perpendicular to the grain were examined. The analysis of the density profiles was carried out on the basis of the following parameters: mean density, minimum to mean density ratio, maximum density, and the distance between the maximum density area and the wood surface. Wood hardness was determined according to the Brinell method. Volume-densified pine wood was characterized by considerably lower susceptibility to densification than beech or oak wood. Densified beech wood had the highest mean density 921 ±7 kg/m$^3$, and the highest maximum density 968 ±12 kg/m$^3$. The Brinell hardness of densified beech, oak and pine wood was twice as high as before the densification. The greatest hardness after the densification 78.60 ±10.56 N/mm$^2$ was observed in beech wood.*

**Keywords:** beech, densification, density profile, European oak, hardness, Scots pine

## Introduction

Wood as an organic material is susceptible to elastic and plastic deformation. It is therefore possible to orientate wood properties. One of the solutions applied in order to improve the physical and mechanical properties of wood is the thermo-mechanical (TM) modification. TM modification of wood is a process involving compression by pressing heated wood. It is usually conducted in a hydraulic press equipped with heated plates [Ülker et al. 2012; Laine et al. 2016]. Sometimes machines are especially designed or modified, which allows the introduction of steam during processing [Pařil et al. 2014; Cruz et al. 2018; Fang

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et al. 2019]. Then the thermo-hydro-mechanical (THM) modification occurs. TM and THM are one of the wood densification method [Ülker et al. 2012]. The effect of applying such a solution is an increase in density, and consequently in the hardness of wood. These characteristics are of particular importance in wood which is to be used for flooring material. Densification improves the functional properties of wood.

Density profile is an indicator of physical and mechanical properties not only in wood-based panels, but also in wood [Ülker et al. 2012; Rautkari et al. 2013; Laskowska 2017]. The method of processing wood by means of thermo-mechanical modification is still subject to research whose aim is to find for it new industrial applications. A major issue is to match the pressing parameters with particular wood species because of the differences in their chemical and anatomical structures [Dogu et al. 2010; Budakçı et al. 2016].

Density profile of densified wood is influenced by many factors. The density profile of thermo-mechanically modified wood depends principally on the wood's anatomical structure and treatment parameters [Rautkari et al. 2011, 2013; Laine et al. 2014]. A material factor conditioning the course of wood densification, and consequently the density profile, is the moisture content [Ülker et al. 2012]. In principle, it is assumed that the higher the moisture content and temperature of thermo-mechanical modification, the greater the wood's susceptibility to densification [Kutnar and Šernek 2007]. These conditions ought to be adhered to in order to obtain in short time the glass transition temperature of the wood's structural components, and wood softening. It is stated that the glass transition temperature for lignin is 100°C at about 10% moisture content [Salmén 1982; Olsson and Salmén 1997]. It ought to be pointed out, however, that the higher the densification temperature, the greater the changes in wood colour [Fang et al. 2012; Bekhta et al. 2014]. Moreover, high temperature of thermo-mechanical modification may bring about deterioration in the wood's mechanical properties [Ülker et al. 2012]. Density profiles differ likewise depending on the degree of densification, which in turn depends principally on the treatment parameters such as temperature and pressure [Kutnar et al. 2009; Rautkari et al. 2011]. The density profile of wood is also affected by the method of thermo-mechanical modification. The profile of volume-densified wood shows less variability in transverse section than the profile of surface-densified wood [Rautkari et al. 2013; Laskowska 2017].

Hardness is an important feature of wood which determines its functional properties. This is particularly relevant to wood species which are used for flooring materials. Thermo-mechanical modification enables the orientation of the properties of low density wood, i.e. below 500 kg/m$^3$ [Kutnar and Kamke 2012; Tu et al. 2014; Zhan and Avramidis 2016]. Research on densification of high density wood is aimed at improving its mechanical properties, in particular the hardness, still further [Rautkari et al. 2010; Gašparik et al. 2016; Laskowska 2017]. The most important factor which affects the hardness of wood is its density [Pizzi et al. 2005; Gong et al. 2010; Rautkari et al. 2011]. Rautkari et al. [2013] stated that hardness was mostly affected by the density of the surface layer, while the depth of the layer with increased density did not influence the Brinell hardness.

The research object was to determine the impact of thermo-mechanical densification on the density profile and hardness of beech, oak and pine wood. An important aspect of this research was to determine the correlation between the density profile parameters and the hardness of wood subjected to densification. The wood selected for testing varied in anatomical structure. Consequently, it was possible to verify and compare the properties of diffuse-porous hardwood, ring-porous hardwood, and softwood subjected to densification.

## Materials and methods

The thermo-mechanical modification of wood was carried out in laboratory conditions in a hydraulic press. The samples used in the tests were of beech (*Fagus sylvatica* L.), oak (*Quercus robur* L.) and pine (*Pinus sylvestris* L.) wood, and had the following dimensions: 130 mm (longitudinal), 80 mm (tangential), 23 mm (radial). The wood was obtained from a forest in central Poland (Mazowieckie voivodship), 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 9.8% (± 0.6%). For each variant of thermo-mechanical modification 20 samples were used. Sample surfaces were finished by planing. The wood densification process consisted of three stages outlined in Figure 1.

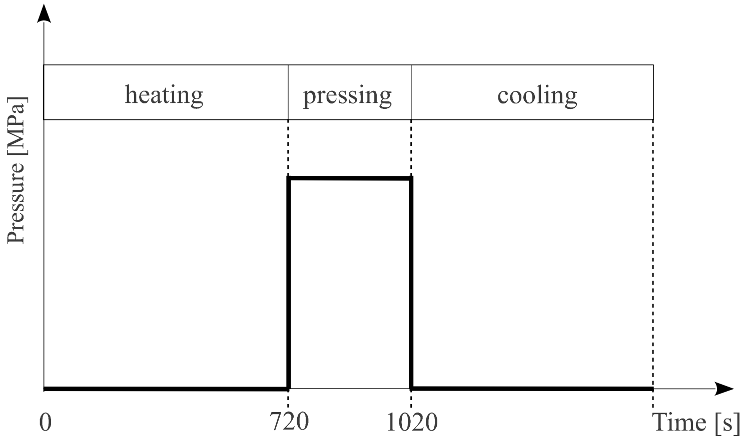


**Fig. 1. Thermo-mechanical wood densification**

At the first stage the wood was contact-heated in a hydraulic press to obtain 17 mm target thickness. The temperature of press plates was 100°C, wood heating time 720 s, wood pressing time 300 s. The wood samples were cooled in an unheated hydraulic press without exerting pressure. The wood samples were cooled until the wood surface reached the temperature of 70°C. Then, the samples were conditioned in a normal climate (i.e. 20°C (±2°C) at 65% (±5%) for 7 days.

The density distribution on the thickness of wood samples before and after densification was determined by using Laboratory Density Analyzer DA-X manufactured by GreCon Inc. (Tigard, Oregon, USA), which enabled the determinations to be carried out by using X-rays. The dimensions of the samples tested were 50 × 50 mm length and width, and the measurement speed 0.05 mm/s. The thickness of the samples corresponded to the thickness of non-densified and densified solid wood. Consecutive density values were measured every 0.02 mm of the thickness of the material tested. Wood density profiles were determined parallel and perpendicular to the grain in accordance with the outline presented in Figure 2a. The analysis of wood sample density profiles was carried out on the basis of the following parameters (Fig. 2b):

1. mean density (DMean);
2. minimum to mean density ratio (DMin/DMean);
3. maximum density on the left-hand side (DMaxL), i.e. the maximum density of wood determined in the area of wood whose surface touched the upper plate of the press;
4. maximum density on the right-hand side (DMaxR), i.e. the maximum density of wood determined in the area of wood whose surface touched the lower plate of the press;
5. 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 wood surface touching the upper plate of the press;
6. 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 wood surface touching the lower plate of the press.

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).

$$\mathrm{CR} = \frac{t_0 - t_d}{t_0} \cdot 100(\%) \tag{1}$$

Brinell hardness was determined on the tangential surface of the sample in accordance with the requirements of EN 1534:2010, after conditioning wood samples in a normal climate (temperature 20°C ±2°C, relative humidity 65% ±5%) for 7 days. Hardness measurements were conducted using the

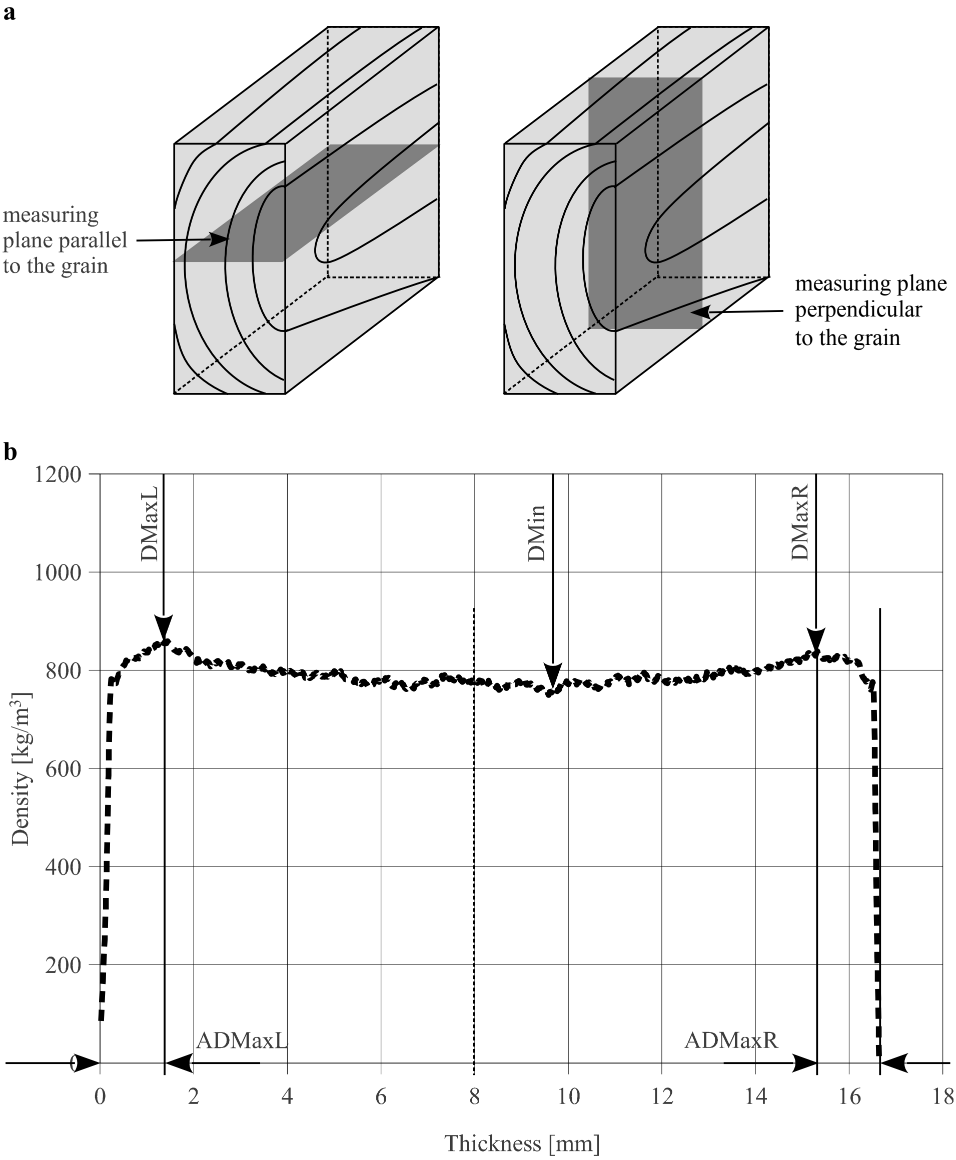
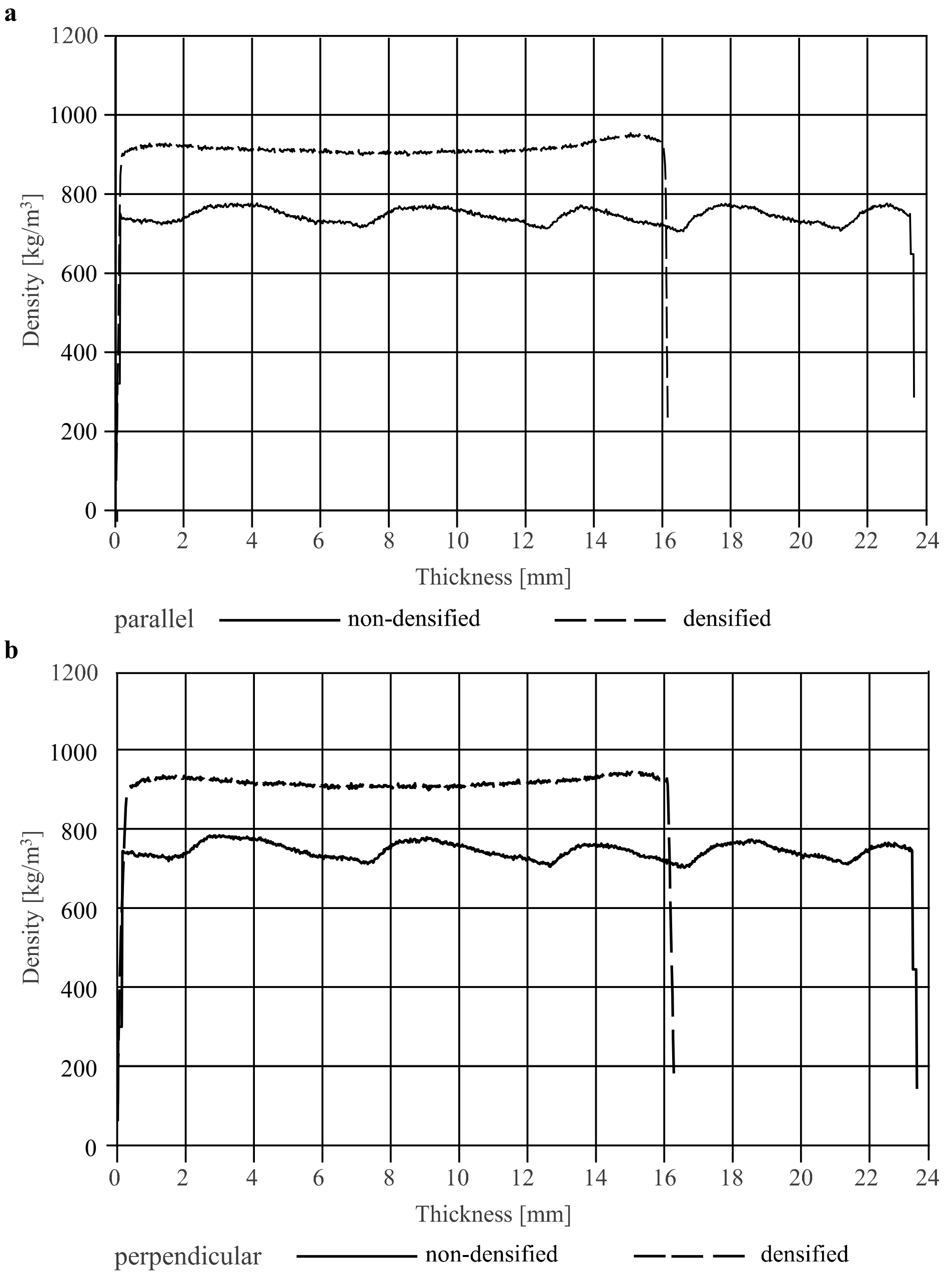


**Fig. 2. Directions of wood density profile measurement (a) and wood density profile parameters tested (b)**

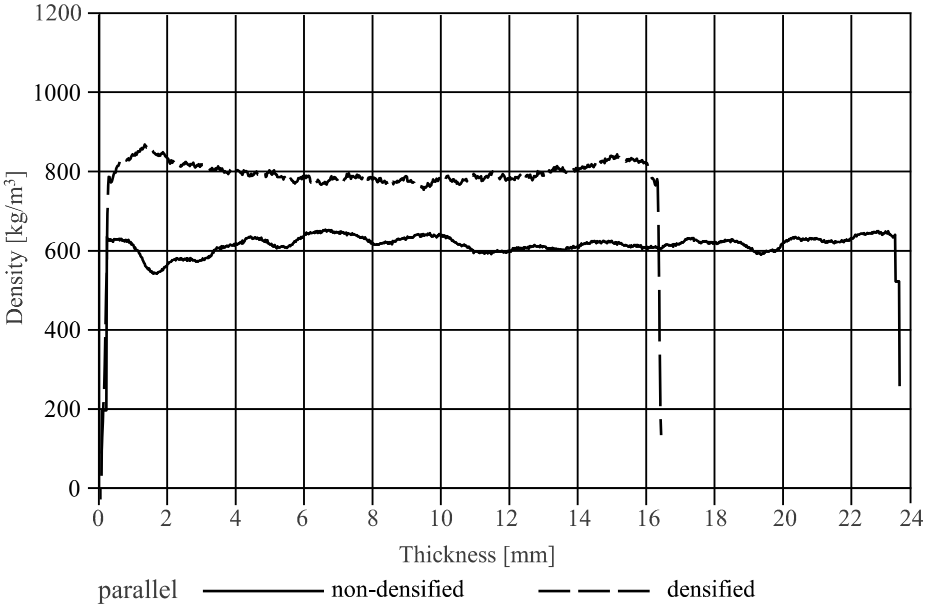
universal testing machine 3000LDB manufactured by C.V. Instruments Ltd. (Sheffield, United Kingdom). The machine was equipped with an indenter with a diameter of 10 mm. The maximum load was 1 kN. The wood hardness was determined for 20 samples of each variant of non-densified and densified wood. Statistical analysis was performed using STATISTICA version-12 software of StatSoft, Inc. (Tulsa, USA). The statistical analysis of the results was carried out at a significance level of 0.05.

## Results and discussion

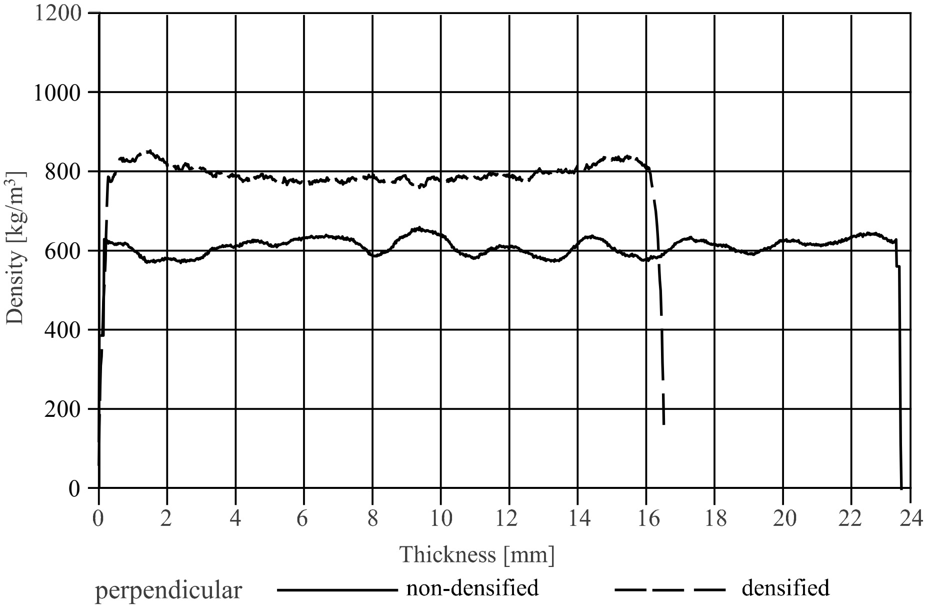
Density profiles of particular wood species before and after thermo-mechanical modification have been presented in Figure 3. No differences in wood density



**c**


1200
1000
800
600
400
200
0
Density [kg/m³]
0
2
4
6
8
10
12
14
16
18
20
22
24
Thickness [mm]
parallel
non-densified
densified

**d**


1200
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Density [kg/m³]
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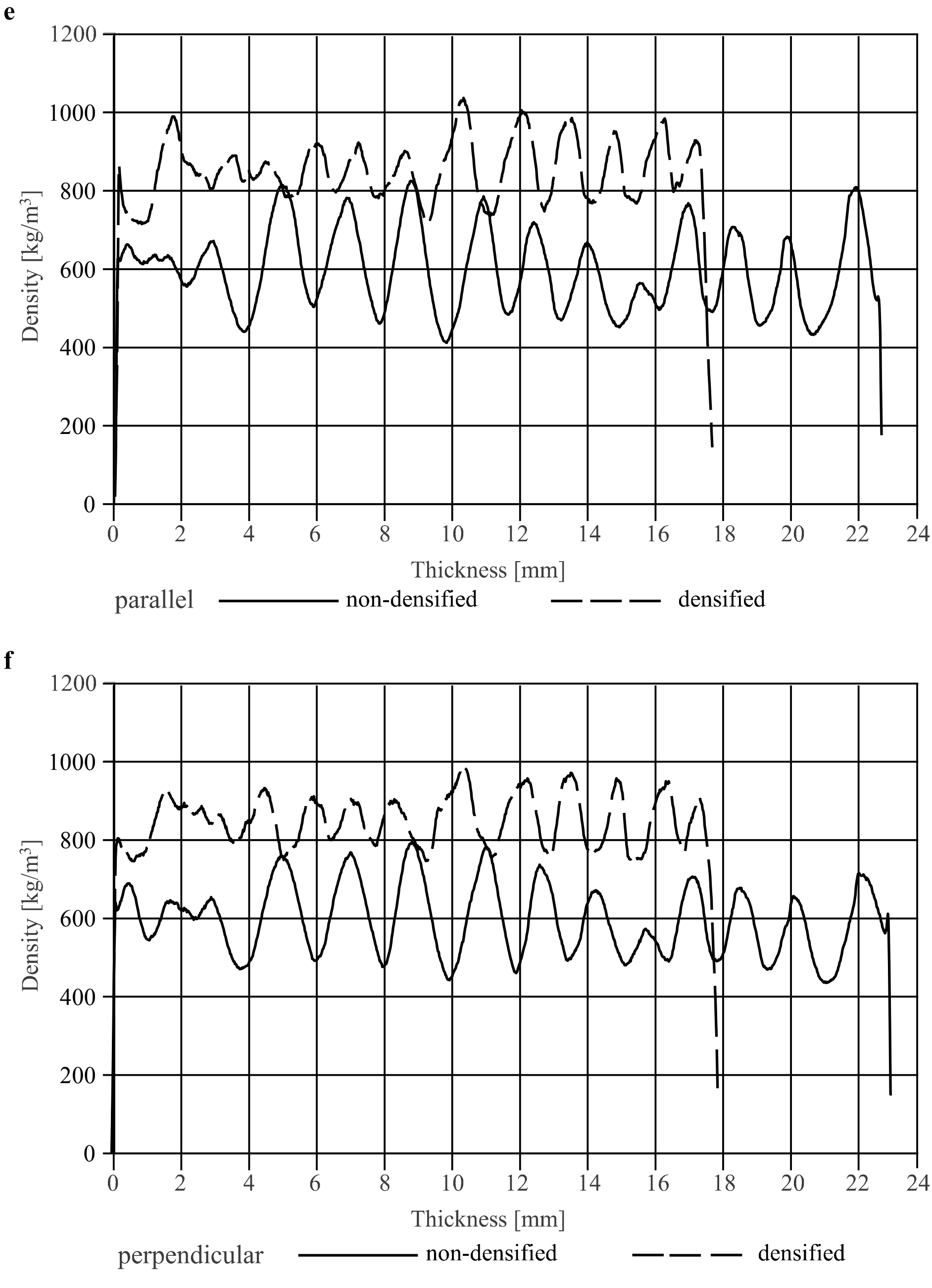

**Fig. 3. Density profile of investigated wood species**

distribution measured parallel or perpendicular to the grain were noted (Fig. 3). This concerned both non-densified and densified wood. As the research showed, the thermo-mechanical modification resulted in homogenizing the structure of beech (Fig. 3a, b) and oak (Fig. 3c, d) wood. No differences in density between earlywood and latewood were noted. In pine wood, however, the differences between earlywood and latewood were observable after the densification as well (Fig. 3e, f). Earlywood underwent densification to a higher degree than latewood. The difference in susceptibility to densification between earlywood and latewood of coniferous species had been pointed out by other authors as well. Dogu et al. [2010] and Laine et al. [2016] observed differences in the densification of early and late pine wood. Schrepfer and Schweingruber [1998], Navi and Girardet [2000] noted differences between earlywood and latewood structure in densified spruce wood.

The mean values of the compression ratio (CR) for beech, oak and pine wood were 30%, 28%, and 23%, respectively (Table 1). This showed that volume-densified pine wood was characterized by considerably lower susceptibility to densification than beech or oak wood, and these differences were statistically significant (Dunnett test, $p < 0.05$). This resulted from the wood's anatomical structure. Earlywood, built of large-diameter thin-walled cells, has an average density of 340-360 kg/m$^3$, and latewood, with thick-walled cells, has an average density of 810-900 kg/m$^3$, i.e. 2.5 to 3 times greater [Kollmann and Cöte 1984]. The results of the research have justified a general statement that the densification of pine wood brought about a 60% increase in earlywood density, whereas the latewood density after densification was ca. 30% higher than that of non-densified latewood (Fig. 3e, f).

The density (DMean) of non-densified beech and oak wood amounted to 739 kg/m$^3$ (±5 kg/m$^3$) and 604 kg/m$^3$ (±8 kg/m$^3$), respectively, whereas the density of non-densified pine wood amounted to 587 kg/m$^3$ (±16 kg/m$^3$). According to Wagenführ [2007], beech wood (*Fagus sylvatica* L.) density in an air-dry condition ranges from 540 kg/m$^3$ to 910 kg/m$^3$, with the mean value 720 kg/m$^3$, whereas oak wood (*Quercus robur* L.) density in an air-dry condition ranges from 430 kg/m$^3$ to 960 kg/m$^3$, with the mean value 690 kg/m$^3$. Pine wood with average width of growth rings ca. 1.5 mm attains its maximum density, which in an air-dry condition amounts to ca. 570 kg/m$^3$ [Kollmann 1951; Trendelenburg and Mayer-Wegelin 1955; Kollmann and Cöte 1984]. The densification of beech, oak and pine wood increased their density by 25%, 26%, and 41%, respectively, in comparison to non-densified wood of beech, oak and pine. The highest density after the densification characterized the beech wood (921 ±7 kg/m$^3$). High increase in pine wood density resulted from the fact that its density before densification had been the lowest among the wood species tested. The increase in density contrasted with low initial density resulted in high percentage of density increase. The tested pine wood, at 23% compression ratio,

**Table 1. Density profile parameters of beech, oak and pine wood (standard deviation in parentheses)**

| Wood species | Modification | Direction | Properties | | | |
|---|---|---|---|---|---|---|
| | | | thickness [mm] | CR [%] | DMean [kg/m$^3$] | DMin/DMean [%] |
| Beech | non-densified | | 23.58 (0.02) | | 739 (5) | |
| | | parallel | | | | 95 (1) |
| | | perpendicular | | | | 95 (1) |
| | densified | | 16.61 (0.20) | 30 | 921 (7) | |
| | | parallel | | | | 98 (1) |
| | | perpendicular | | | | 98 (1) |
| Oak | non-densified | | 23.67 (0.02) | | 604 (8) | |
| | | parallel | | | | 90 (3) |
| | | perpendicular | | | | 89 (2) |
| | densified | | 17.05 (0.60) | 28 | 762 (36) | |
| | | parallel | | | | 95 (1) |
| | | perpendicular | | | | 96 (2) |
| Pine | non-densified | | 22.98 (0.07) | | 587 (16) | |
| | | parallel | | | | 68 (9) |
| | | perpendicular | | | | 76 (6) |
| | densified | | 17.63 (0.22) | 23 | 827 (33) | |
| | | parallel | | | | 89 (5) |
| | | perpendicular | | | | 90 (3) |

attained 827 ±33 kg/m$^3$ density (increase by 41%). Laine et al. [2016] densified pine wood (*Pinus sylvestris* L.) at 150°C for 1 h. These authors, at 39% densification ratio, obtained pine wood density 757±2.6 kg/m$^3$ (increase by 47%). The differences in pine wood density after densification resulted from different initial densities of the wood.

As a result of densification, the wood structure became homogenized, which was reflected in higher minimum to mean density ratios (DMin/DMean) for densified then for non-densified wood. The greatest changes in DMin/DMean were observed in pine wood. The change in DMin/DMean amounted to 14 and 21 percentage points in density profiles measured perpendicular and parallel to

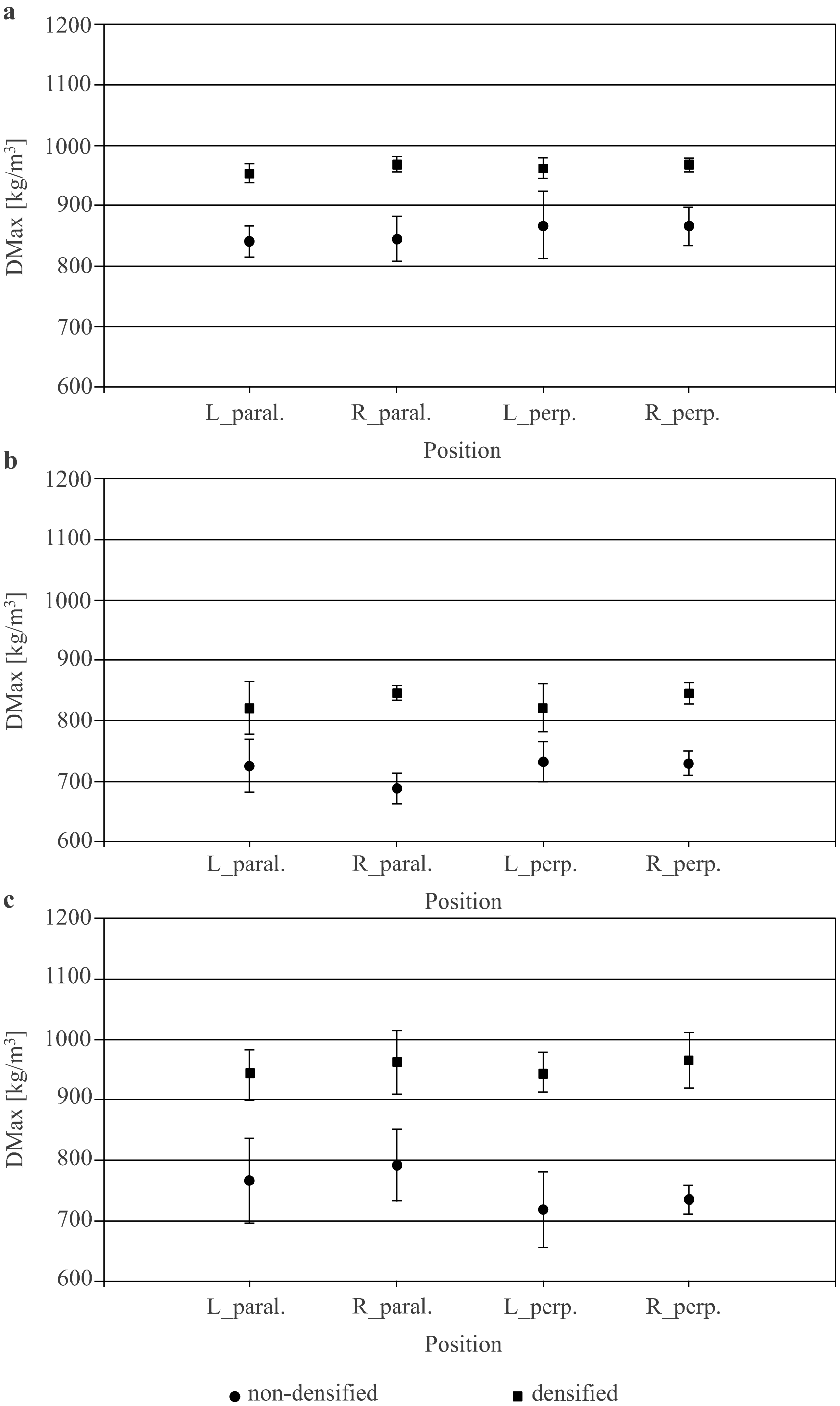

**Fig. 4. Maximum density (DMax) of investigated wood species**

**Table 2. Distance between the maximum density area and the wood surface (ADMax), (standard deviation in parentheses)**

| Wood species | Position | | Modification | | | |
|---|---|---|---|---|---|---|
| | | | non-densified | | densified | |
| | | | mean [mm] | std. dev. [mm] | mean [mm] | std. dev. [mm] |
| Beech | parallel | L | 0.10 | 0.02 | 0.78 | 0.19 |
| | | R | 0.05 | 0.01 | 1.09 | 0.23 |
| | perpendicular | L | 0.09 | 0.01 | 0.82 | 0.20 |
| | | R | 0.06 | 0.01 | 1.43 | 0.23 |
| Oak | parallel | L | 0.11 | 0.03 | 1.60 | 0.30 |
| | | R | 0.05 | 0.02 | 1.04 | 0.22 |
| | perpendicular | L | 0.10 | 0.01 | 1.71 | 0.24 |
| | | R | 0.04 | 0.01 | 0.89 | 0.16 |
| Pine | parallel | L | 5.20 | 0.51 | 2.02 | 1.30 |
| | | R | 0.95 | 0.21 | 6.18 | 1.72 |
| | perpendicular | L | 5.02 | 0.40 | 1.93 | 1.00 |
| | | R | 1.00 | 0.20 | 6.96 | 0.89 |

the grain, respectively. The smallest changes in DMin/DMean were observed in beech wood. They amounted to 3 and 4 percentage points in the density profiles measured parallel and perpendicular to the grain, respectively.

No significant differences were observed in maximum density of wood (DMax) measured parallel or perpendicular to the grain. This concerned the tested species of wood both before and after thermo-mechanical modification. Densified beech, oak and pine wood was characterized by considerably higher maximum density than before the densification (Fig. 4a, b, c). The greatest differences were observed in pine wood (increase in DMax by ca. 25%). The changes observed in beech and oak were comparable (increase in DMax by ca. 15%). Similarly, no significant differences were observed in DMax measured on the left- or right-hand side. The lack of significant differences in this respect was observed in both non-densified and densified wood. This demonstrated the low variability in wood density, in this case in the density of latewood areas. It is namely the density of latewood areas which chiefly determines the DMax value. After the densification, the highest maximum density was that of beech wood, 968±12 kg/m$^3$, and the smallest - of oak wood, i.e. 821 ±11 kg/m$^3$.

No significant differences were observed in the distance between the maximum density area and the wood surface (ADMax) measured parallel or perpendicular to the grain in the wood species tested (Table 2). After the densification, ADMax was situated further from the sample's surface. The exception was pine wood in the cases when the measurements were taken parallel and perpendicular the grain on the left side. The research results showed that beech and oak wood became densified to a higher degree than pine wood in near-surface areas. This was demonstrated by the low value of ADMax which in the case of beech wood amounted to ca. 0.5 mm on the left side, and ca. 1.5 mm on the right. ADMax of oak wood amounted to ca. 1.7 mm on the left, and ca. 1.0 mm on the right. In pine wood, however, the DMax areas after densification were situated at the distance of ca. 7.0 mm on the left, and 1.0 mm on the right. The impact of temperature on wood surface caused the wood tissue to plasticize and the maximum density DMax area to "shift" deeper into the sample.

Brinell hardness of densified beech, oak and pine wood was twice as high as before the densification (Fig. 5a). The smallest hardness after densification characterized the pine wood (49.60 ±13.11 N/mm$^2$), whereas the greatest hardness was that of the beech wood (78.60 ±10.56 N/mm$^2$). Significant changes in wood hardness were likewise observed after different densification processes [Inoue et al. 1993; Navi and Heger 2004; Kamke 2006]. It ought to be pointed out that different values of the compression ratio (CR) were obtained for beech, oak and pine wood as a result of the densification (Table 1). The same increase in hardness, namely its doubling, was obtained at different wood compression ratios. Rautkari et al. [2013] also stated that the degree of compression did not significantly affect the Brinell hardness. The crucial factor for the hardness of wood is its density, in particular the density of the near-surface layers. The authors surface-densified pine wood (*Pinus sylvestris* L.) and obtained over 90% increase in Brinell hardness. Ülker et al. [2012], after having densified pine wood (*Pinus sylvestris* L.) at temperature 120°C, observed the density to increase by 83% and the hardness to multiply 2.3 times. Laine et al. [2016], after having densified Scots pine sapwood at 40%, 50% and 60%, obtained increase in wood hardness from 15.5 N/mm$^2$ to 19.0 N/mm$^2$ (by 23%), 29.3 N/mm$^2$ (by 89%), and 23.9 N/mm$^2$ (by 54%). Fang et al. [2012] densified wood veneers made of aspen (*Populus tremuloides*) and hybrid poplar clone 15303 (*Populus maximowiczii* × *Populus balsamifera*) using heat, steam, and pressure. Authors stated that the Brinell hardness of densified veneers was about two or three times that of control for both aspen and hybrid poplar. Gašparík et al. [2016] obtained considerably lower values of beech wood hardness after densification. Depending on the treatment parameters, the hardness of beech wood was from 1% to ca. 10% higher after the densification. Minor differences resulted from the fact that the wood was cold-pressed without prior plasticizing. Linear dependences between the density of non-densified or densified wood and its

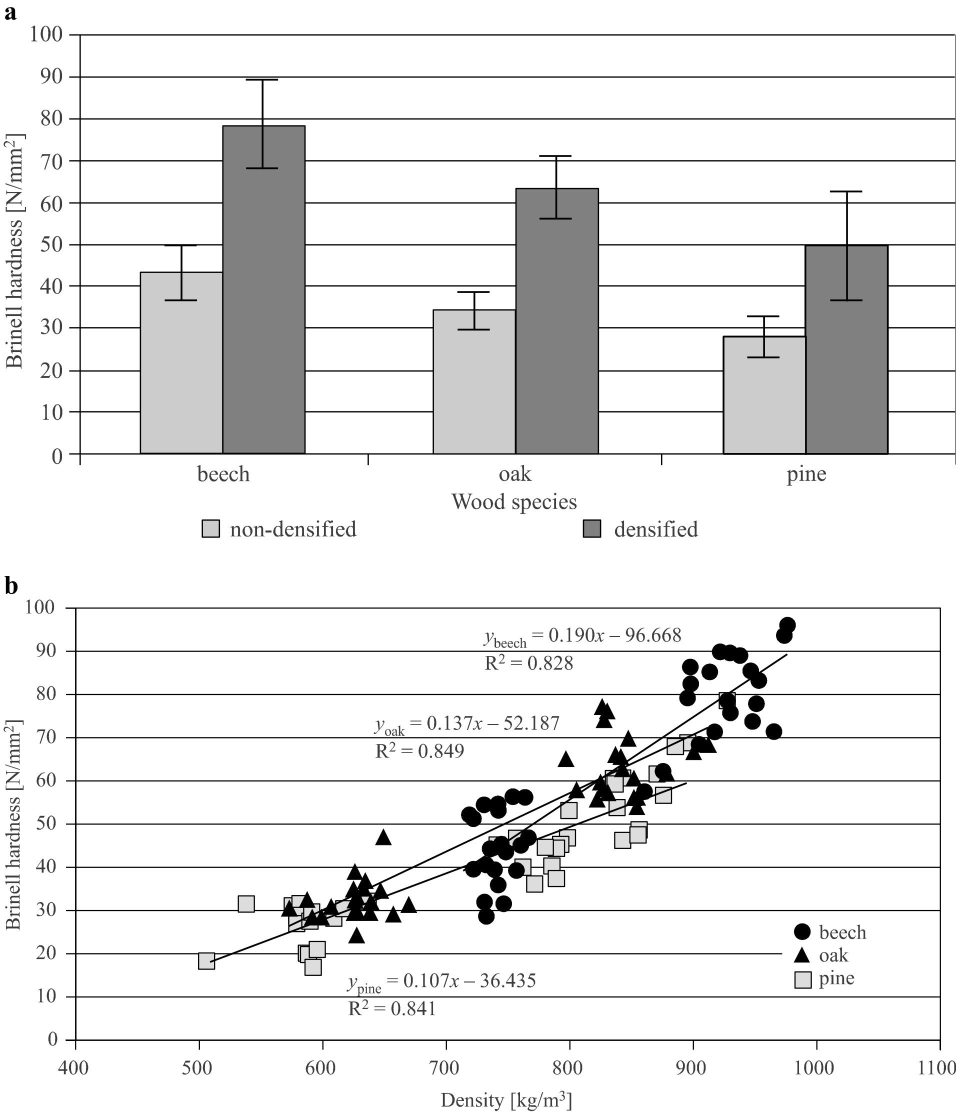

**Fig. 5. Brinell hardness (a) and relationship between mean density and Brinell hardness (b) of investigated wood species**

hardness may be described by means of equations detailed in Figure 5b. The hardness of non-densified and densified wood shows high degree of correlation with wood density, which has been confirmed by the research.

## Conclusions

Thermo-mechanical modification resulted in homogenizing the structure of beech and oak wood. However, in the case of pine wood, the differences between earlywood and latewood were noticeable after the thermo-mechanical modification as well. No differences in density distribution measured parallel or perpendicular to the grain were identified. Oak and beech wood were characterized by comparable compression ratios (28-30%), whereas the compression ratio of pine wood was the lowest and amounted to 23%. As a result of densification, mean density of beech, oak and pine wood increased by 25%, 26%, and 41%, respectively, in comparison with non-densified beech, oak and pine wood. The highest density after densification was observed in beech wood. The maximum density of densified beech, oak and pine wood was considerably higher than before the densification. As a result of the densification, the hardness of the tested wood species doubled. The greatest hardness was observed in beech wood, the smallest in pine wood.

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*Submission date: 15.09.2017*

*Online publication date: 15.05.2020*