ORIGINAL

Changes in the microstructure of birch wood after hydrothermal treatment

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Abstract Birch wood (*Betula pendula*) samples were treated in a thermal regime (140, 160, 180 °C) for 1 h and investigated by means of scanning electron microscopy (SEM). SEM microimages of the wood cross-section were taken from one and the same place before and after the thermal treatment (magnification $100-2,000\times$). The results of measurements of areas and linear sizes of the birch wood cells show significant changes, which depend on the thermal treatment conditions and the type of the cell: libriform, tracheid, vessel and ray. After the treatment at 180 °C, the integrity of wood morphological structure begins to break up. Voids and cracks are formed between fibres, thus leading to a decline in the mechanical properties of the wood.

Introduction

Wood is characterized by the nonuniformity of its chemical composition (cellulose, hemicelluloses, lignin and extractives) and the unequal distribution of the main components throughout the wood cell wall. In addition, the wood microstructure is characterized by the multiformity of its cell elements.

Birch (*Betula pendula*), a hardwood species, is the most significant deciduous tree species in Latvia. Around 28 % of the forest area in Latvia is covered with birch; its stock (153 million m^3) makes up 24 % of the total growing tree stock (www.zm.gov.lv). Birch wood is mainly used for plywood production and for indoor applications due to its low dimensional stability (Hill 2006) and the lack of

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natural durability with respect to wood fungi (EN350-2 1994). New fields of application could be found if these disadvantages were minimized or, even, overcome. Modification is one of the recent technologies used to improve the durability and dimensional stability of birch wood.

Industrial heat treatment processes have been successfully developed in Europe (Militz 2002). Thermal modification of hardwood has been intensively studied in recent years, and birch makes up about 7 % of thermally modified wood (Scheiding 2008). During the thermal treatment, changes in both the chemical composition and components' ratio, as well as wood microstructure changes, occur simultaneously. According to Boonstra et al. (2006a), thermal treatment affects the anatomical structure of the wood, but the effects depend on the wood species and the process used. Boonstra et al. (2006b) concluded that some hardwood species are sensitive with regard to the collapse of vessels and the degree of deformation of fibres near the vessels; numerous radial cracks near the rays have been observed after thermal treatment. The changes in the morphological structure elements of wood during thermal treatment have also been documented by Awoyemi and Jones (2010). They found that the thermal treatment of red cedar wood results in the destruction of tracheid walls, ray tissues and pith deaspiration.

Earlier results by the authors (Andersons et al. 2008) on the hydrothermal modification of hardwood have included efforts to find the optimum regimes of the process. The authors hoped to reach an acceptable compromise between the improved durability of wood against biodegradation agents (white and brown rot fungi) and form stability and still obtain acceptable mechanical strength properties. To gain insight into the behaviour of deciduous wood structure subsequent to hydrothermal action, it was attempted to elucidate the changes in the microstructure of birch wood depending on the treatment parameters. Scanning electron microscopy (SEM) was used as a suitable method for the studies. For more exact comparison of wood samples before and after the thermal treatment, it was necessary to develop a special methodology for quantity characteristics of various individual cells (libriform, tracheid, vessel and ray).

Materials and methods

Materials

Birch wood was used to study the anatomical changes of the various cell types in wood after hydrothermal treatment.

Sample preparation

A birch wood stick measuring $20 \times 20 \times 100 \text{ mm}^3$ was chosen for the assessment of the parallel annual ring, which was visible throughout the length of the stick, to reduce the inhomogeneity between the samples for microstructure investigation (Fig. 1). Three samples measuring $6 \times 7 \times 7 \text{ mm}^3$ were prepared from the stick and placed in deionized water for softening (4 days). The water was replaced every

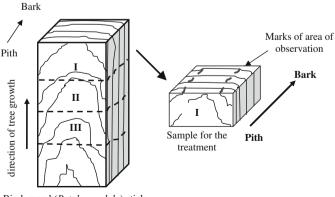
day with a new portion of deionized water to prevent the development of mould decay. After that, the samples' surfaces were smoothed with a blade and then dried. The drying phase consisted of two gradual drying steps to avoid crack formation. At the first step, the samples were dried at 20 °C for 5 days in atmospheric conditions. In the final step, 5-day drying at 40 °C under vacuum (50 hPa) was performed in a vacuum chamber, where nitrogen pentoxide was used as a water vapour absorber. Each sample was thermally treated according to a particular regime: sample (I) was treated at 140 °C for 1 h, sample (II) at 160 °C for 1 h and sample (III) at 180 °C for 1 h according to the procedure given in detail under the section "Treatment".

Treatment

The treatments were carried out in a multifunctional pilot scale device produced by wood thermal technology (WTT). Samples were placed in an autoclave, which ensured the thermal treatment in a water vapour medium at a chosen modification temperature, rising rate and holding time (Table 1). In the course of the process, as the water evaporated at a rate dependent on the treatment temperature in the chamber, the pressure increased, reaching 5–9 bars. Modification temperatures were stated as 140, 160 or 180 °C. The treatment process consisted of three technological stages: (1) temperature increase up to the modification temperature; (2) holding at the modification temperature (1 h); (3) cooling.

Obtaining and investigation of SEM images

The birch wood cross-section microimages were obtained according to the following procedure: between the final drying steps, the samples were coated with gold using a sputter coater Emitech K550X and then dried. After drying, the samples were directly placed in a SEM chamber, and cross-section microimages of untreated birch wood samples were obtained by using an SEM Vega Tescan 5136M. Then, untreated birch wood samples coated with gold were placed in an autoclave



Birch wood (Betula pendula) stick

Fig. 1 Schematic diagram of sample preparation

Table 1Characteristicparameters of the thermaltreatment	Temperature (°C)	Pressure (bar)	Time to reach the maximum pressure (h)
	140	4.4	10–11
	160	6.3	12–13
	180	9.6	13–14
	-		

Table 2 Types of birch wood morphological elements and number of measurements

Types of elements	Sites of measurements	SEM magnification (×)	Number of SEM images from the sample	meas elem	ber of sured ents in ole (n)	
				Ι	II	Ш
Annual ring	AYRW	100	3	50	50	50
Vessels	VLA	500	8	92	88	95
	VLL-R					
	VLL-Tg					
Libriform	OAC	2,000	5	311	345	265
fibres	LAC					
	ACW					
	LLC-R					
	LLC-Tg					
	CWT-R			622	690	530
	CWT-Tg			622	690	530
Rays	RW	2,000	3	70	70	-

VLA vessels' lumen area, VLL-R and VLL- T_g vessels' lumen linear size in radial and tangential direction, OAC outer area of cell, LAC lumen area of cell, ACW area of cell wall, LLC-R and LLC- T_g cavity linear size of cell in radial and tangential direction, CWT-R and CWT- T_g cell wall thickness in radial and tangential directions, AYRW width of annual year ring, RW ray width

and hydrothermally modified according to the treatment process; after the treatment, the surface in question was covered with gold again, and birch wood cross-section microimages were viewed at 100–2,000 magnification. To ensure precise evaluation, microimages were taken from one and the same site before and after treatment.

Table 2 shows the types of morphological elements and the number of SEM images taken from as well as the number of the measurements done on the sample. Calculations

Microimages of each sample were compared, and changes were calculated according to the formula:

$$\operatorname{SEM}(\%) = \left(\frac{\operatorname{SEM}_1(I) - \operatorname{SEM}_0(I)}{\operatorname{SEM}_0(I)}\right) \times 100 \tag{1}$$

where SEM (%) changes in the average values of morphological elements (%),

 $\text{SEM}_1(I)$ changes in the average values of the morphological elements for samples (*I*) after the treatment, and $\text{SEM}_0(I)$ changes in the average values of the morphological elements for samples (*I*) before the treatment.

Table 3 demonstrates the calculation method for obtaining the average value of the morphological elements before and after the treatment; for example, annual ring-50 measurements were made from three microimages per treatment.

Following hydrothermal treatment, the average morphological data of birch wood were calculated, namely libriform cross-section outer area (μ m²), libriform lumen area (μ m²), libriform wall area (μ m²), libriform lumen linear size in radial and tangential directions (μ m), vessel lumen area (μ m²), vessel lumen linear size in the radial and tangential direction (μ m) and ray width (μ m).

Results and discussion

Each cell type in wood has a specific biological function. Depending on the size of a given element, amounts and forms (shapes) differ. This can be visually established by viewing the initial untreated birch wood cross-section SEM images (Figs. 2, 3, 4, 5).

Calculations based on the authors' analyses of birch wood microimages show that a major part, that is, 78 % of the area of the cross-section, is formed by fibres with similar shape and size (libriform and tracheids), because a clear distinction between libriform and tracheids fibres in many cases is not very easy. Therefore, with this term, that is, fibres, it was decided to denote the type of wood fibres with similar shapes, namely with relatively thick walls and a small lumen size. A fifth of the area, that is, 20.0 %, is composed of vessels, and 2.0 % consists of rays (Fig. 6). The average area of a fibre is approximately $30 \times$ smaller than that of a single vessel, that is, 166 and 5,000 μ m², respectively. At the same time, the number of fibres in wood is approximately $1,000-1,100 \times$ greater than that of vessels.

Type of the element	Image no.	Annual ring width before the treatment throughout the images (μm)	Annual ring width after the treatment among all images (µm)
Annual ring	1	$l_{01} = \frac{\sum l}{15}$	$l_{11} = \frac{\sum l}{15}$
	2	$l_{02} = \frac{\sum l}{15}$	$l_{12} = \frac{\sum_{l} l}{15}$
	3	$l_{03} = \frac{\sum l}{20}$	$l_{13} = \frac{\sum l}{20}$
Average annu width for sa (µm)	-	$l_{0I} = \frac{\sum (l_{01} + l_{02} + l_{03})}{3}$	$l_{1I} = \frac{\sum_{(l_{11}+l_{12}+l_{13})}{3}}{3}$
Annual ring changes aft hydrotherm treatment (er al	$l = \left(\frac{l_{U}-l_{0I}}{l_{0I}}\right) imes 100$	

Table 3 Calculation method

15 and 20-amount of measurements from one picture

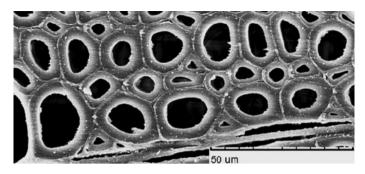


Fig. 2 Fibres and rays before thermal treatment

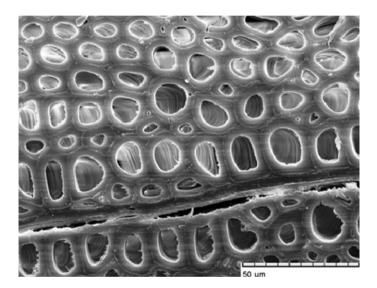


Fig. 3 Fibres and rays before thermal treatment

Annual rings

The annual ring represents the totality of the cells formed within a year. The annual ring width is closely connected to the amount and sizes of the elements incorporated within it. During thermal treatment, the cross-section sizes of the fibres change. The annual ring width decreases with increasing thermal treatment temperature. The most dramatic decline in the annual ring width (up to 22 ± 0.5 %) is observed when the wood is treated at 180 °C (Fig. 7). This dramatic decline in the annual ring width can be explained by the great mass loss in birch wood, even to 18%, at this treatment (Andersons et al. 2008) and the relaxation processes, which cause the corresponding changes in the sizes of the individual cells (see below).

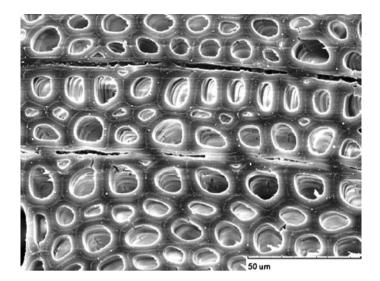


Fig. 4 Fibres and rays before thermal treatment

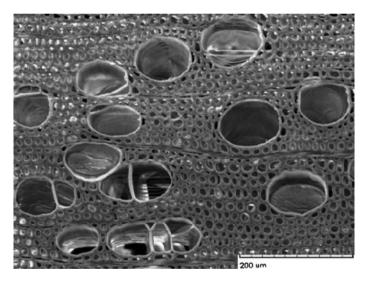
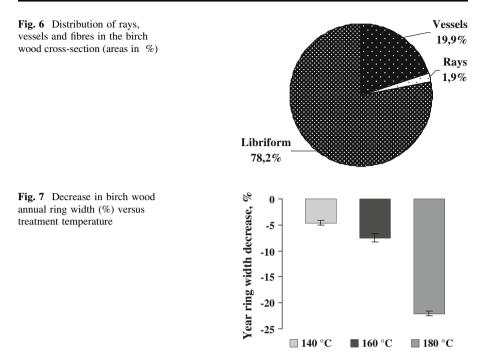


Fig. 5 Vessels before the treatment

Fibres

The narrow fibres with their relatively thick walls $(2-5 \,\mu\text{m})$ and compact arrangement (Figs. 2, 3, 4) fulfil the mechanical functions in birch wood. As mentioned above, this type of fibres forms the bulk of wood. In the wood cross-section, the sizes of the fibre lumen vary, because the fibres in wood are displaced throughout depending on the tree growth direction. The cross-section can intersect



the middle part of a fibre or its ends; therefore, these SEM images display fibres with different lumen sizes, that is, with smaller or greater lumen areas (Figs. 2, 3, 4). The differences in fibre wall thicknesses are insignificant.

In the authors' opinion, the changes established through a variety of measurements are reflected most precisely by the wall area. Wall area and wall thickness correlate with a considerable degree. The following measurements were made with regard to the fibre: total area, wall area, lumen area, wall thickness and lumen linear size (Fig. 8).

Similar measurements were conducted on vessels. The results of the measurements (Table 4) clearly demonstrate the effect of the thermal treatment on the change in the sizes of the birch wood fibre cross-section, and the effect on vessels and rays.

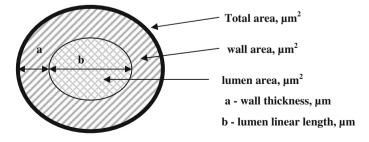


Fig. 8 Measurement positions of the fibres' cross-sections

The treatment at 140 °C has only a limited effect on the cross-section sizes of the fibres. However, when the temperature is increased to 160 and 180 °C, the fibre cross-section sizes (total area of fibres, wall area, wall thickness, etc.) decreases significantly. So, the total area gradually decreases from 1 % (140 °C) to 20.7 % (180 °C). This is connected with the evaporation of the bound water present in the thermally unstable components of fibre walls, that is, hemicelluloses and extractives, isolated as a result of thermal destruction and other accompanying processes, which has been reported by Sivonen et al. (2002). The thermal destruction of wood substance and the diffusion of the destruction products occur across the whole cross-section of the wall. The decrease in the fibre sizes and weight depends on the ratio of the main components and their distribution within the wall. It is known that hemicelluloses represent the most unstable wood-forming component. Hence, the greater the amount of hemicelluloses in wood species, the stronger the fibre sizes will be affected during thermal treatment.

With increasing temperature of the thermal treatment, birch wood becomes less dense (Andersons et al. 2008). At 180 °C, the intensity of the auto-hydrolysis process increases. The destruction products are released together with water vapour.

During the thermal action, the wood fibre wall is in a swollen state. The crosssection form of the fibre lumen changes simultaneously from multangular to more roundish with the decrease in the wall area at 180 °C. In the case here, the fibre wall area and wall thickness decrease to 37.3 and 32.0 %, respectively. Following the thermal treatment at 180 °C, birch wood displayed a decrease in the dense arrangement of wood fibres in some wood cross-section regions, in which the fibres had separated from each other, forming voids and cracks among the fibres (Fig. 9). Figure 9 demonstrates the breaking up of the layers adjacent to middle lamella. The relocating of the products of destruction towards the primary layer and into the intercellular spaces degrades the outer layers of the fibre and may isolate the middle lamella and disturb the density of the fibre packing in wood. In some places, middle lamella is stuffed off the fibre wall. The middle lamella, however, does not disappear because it consists mainly of lignin (70–90 %), which is a more thermally stable wood component.

Vessels

The unique feature that makes hardwoods differ from softwoods is the presence in the former of specialized conducting cells called vessel elements. The cells are stacked on top of one another to form vessels. Untreated birch wood vessels are clearly visible in SEM images as formations with large lumen (Fig. 5). Their function in wood is the transportation of water and nutrients from the roots to the stem. Vessels are located vertically in the direction of tree growth, and vessel length can reach several centimetres. The location of vessels in birch wood is evenly distributed across the whole annual ring. More vessels with greater lumen cross-section areas are observed in early wood; smaller lumen area cross-sections are found in latewood, which is explained by the more intensive transport of nutrients throughout the wood in spring (Rowell 2005).

Table 4 Average cross-section	ge cross-sec	tion sizes of	f the morpholo	gical elemen	sizes of the morphological elements and their changes for birch wood $(\%)$	inges for birch	(%) poom				
Type of elements	Fibres					Vessels					Rays
Type of measurements	Total area (μm ²)	Wall area (μm^2)	Wall area Wall linear Lumen (µm ²) size (µm) area (µm ²)	Lumen area (µm ²)	Lumen linear size (µm)	Total area (μm ²)	Wall area (μm ²)	Wall linear size (µm)	Wall area Wall linear Lumen area (μm^2) size (μm)	Lumen linear size (µm)	Wall width (µm)
Initial sizes	$166 \pm 63 103$		2.58 ± 0.55	62 ± 28	$\pm 38 2.58 \pm 0.55 62 \pm 28 8.65 \pm 2.28 5,000 \pm 669 313 \pm 98 1.24 \pm 0.23 4,695 \pm 592 79.8 \pm 26 1.13 \pm 0.23 4,695 \pm 592 79.8 \pm 26 1.13 \pm 0.23 1.24 \pm 0.24 1.24$	$5,000 \pm 669$	313 ± 98	1.24 ± 0.23	$4,695 \pm 592$	79.8 ± 26	1.13 ± 0.23
Temperature (°C)	-	Changes (%)				Changes (%)	\$ (%)				Changes (%)
140	Ī		-5.2 -	-6.7	2.4 0.1	-5.5	-6.3	-4.0	-5.5	-4.2	-8.8
160	Ī	-10.5 -	-23.4 -	-21.8	14.0 6.1	-3.4	-18.9	-17.7	-2.3	-1.7	-24.7
180		-20.7 -	-37.3 -	-32.0	4.8 1.4	-3.1	-16.3	-15.3	-2.3	-1.5	I

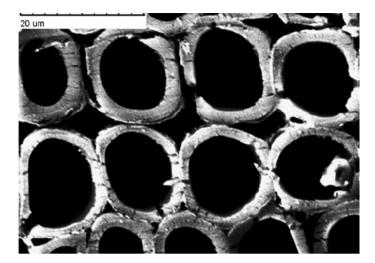


Fig. 9 Fibres after the treatment at 180 °C

Based on the measurements of the vessel cross-section area size, it can be concluded, as is show in (Fig. 10a, b), that birch vessels are the least changed as a result of thermal treatment. The vessel lumen area is decreased most intensively (5%) at 140 °C, but, with increasing treatment temperature, only a minor decrease in the total vessel area is observed (2.3%). These minor vessel area changes might be explained by the fact that the vessel walls are twice as thin (1 μ m) as the fibre walls. These vessel walls are assumedly comprised of lignin which is more stable than that in the libriform wall. Vessels are known to contain mainly guaiacyl structures, whereas libriform and wood rays contain syringyl structures (Gromov et al. 1977). It has been found that at 140 °C, the linear sizes of the lumen in both radial and tangential directions diminish in a similar fashion, that is, the wood sizes decrease similarly in both directions. With increasing treatment

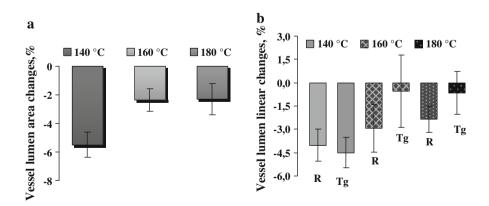


Fig. 10 a Changes in the vessel lumen cross-section area; b lumen linear size in radial and tangential directions with respect to treatment temperature

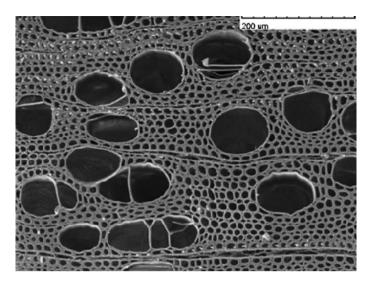


Fig. 11 Vessels after the thermal treatment at 180 °C

temperature, it has been found that the decrease in the lumen length in the radial direction is greater than that in the tangential direction at both 160 and 180 °C, that is, 2.9 and 2.3 % versus 0.5 and 0.7 %, respectively.

The effect of the treatment parameters on the vessel walls was relatively small (Fig. 11). The minor difference between the pressures in the lumen and the autoclave due to the good connectivity of vessels could have been one of the reasons.

Wood rays

Medullary rays in wood replenish the nutrient reserves. These elements have a thin horizontally stretched wall and a narrow long cavity, which extends throughout the wood from pith to bark.

Under the conditions in the experiment, rays' changes were only observed in the wood cross-section. There the rays are located on the surface and are only cut in the longitudinal direction due to their horizontal location in the wood (Fig. 12).

Rays in wood are very visible after treatment at 140 and 160 °C (Figs. 13, 14). This was also confirmed by the results of the quantitative measurements, that is, the wall width decreased by 8.8 %. With increasing thermal treatment temperature, the ray walls decreased to 25 % (160 °C) and began to shrink (Fig. 14). With increasing treatment temperature from 140 °C up to 180 °C, the effect of pressure and temperature on the rays increased and this was destructive (Fig. 15). The rays were most affected by these parameters. The lumen was deformed; its shape was transformed into a fully or partially open long-wide crack. The crack width grew, on average, to 250 % (180 °C). After the treatment at 180 °C, the walls became deformed and, due to the damage, they could not be measured precisely. As a result

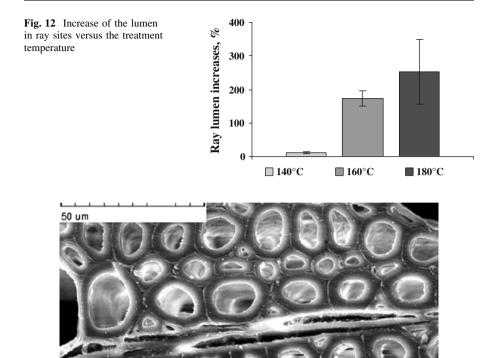


Fig. 13 Fibres and rays after the thermal treatment at 140 °C

of the thermochemical destruction and the diffusion of low-molecular destruction products, a partial destruction of the rays was observed.

It is mentioned that the observed changes relate only to the rays present on the surface of wood pieces. Even though the total content of the rays in birch wood is only 1.9 %, the formation of long micro-cracks on the surface could essentially affect the wood properties.

Discussion

Various authors (Zaman et al. 2000; Esteves et al. 2008; Alén et al. 2002) have established the effect of different factors of thermal treatment processes (temperature, pressure, medium and time) on wood. These are only a few factors, which can considerably change the wood and which are closely connected even at different size scales, that is, from log to wood fibre. Esteves et al. (2007) have reported a higher mass loss for hardwood than for softwood species under the same treatment conditions.

During the thermal treatment of this study, parameters, such as temperature, pressure and water vapour concentration in the autoclave, vary. As a result, the rate of auto-hydrolysis, the amount of the volatile low-molecular products and other

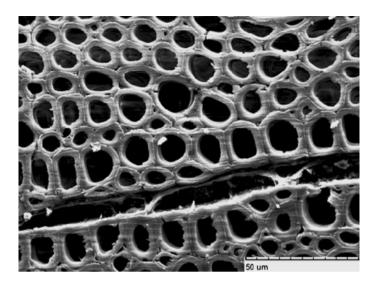


Fig. 14 Fibres and rays after the thermal treatment at 160 °C

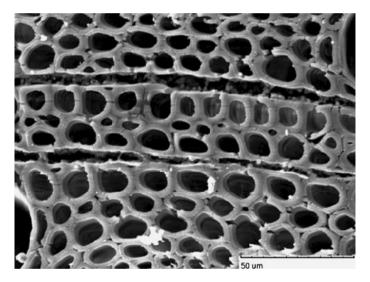


Fig. 15 Fibres and rays after the thermal treatment at 180 °C

functional circumstances also vary. In these comparative studies, the following variable thermal treatment parameters were taken into account: retention temperature within 1 h (140, 160 and 180 $^{\circ}$ C) and pressure.

As has already been mentioned, the peculiarity of the process under study is the presence of a number of stages within the technological thermal treatment process in the reactor, that is, a rise in temperature accompanied by simultaneous increase in pressure, a retention at a set temperature within an hour and a cooling phase

combined with a release of pressure at a given rate in an autoclave. Pressure, for example, during the treatment at 180 °C, reached 9.6 bar.

In the temperature range under study, hemicelluloses (HC) were subjected to the greatest destruction, leading to the formation of volatile and low-molecular compounds (acetic acid, acetone, formic acid, furfural, etc.), which has been reported (Dietrichs et al.1978; Bourgois and Guyonnet 1988; Tjeerdsma et al. 1998; Weiland and Guyonnet 2003; Wikberg and Maunu 2004).

In the authors' opinion, the most important reason for the appearance of these factors is the distribution of HC and lignin in the wood cell wall. The hemicelluloses content in birch wood is relatively high, approximately 30 %.

The authors made evaluative calculations of the comparative concentrations of the main components of wood in different layers of the birch wood cell wall (Table 5). Calculations of mass in cell wall layers (%) were based on the analysis of the SEM images of the fibres in the initial birch wood and the data presented by (Dudkin et al. 1991). It was observed that the concentration of HC in the M + P layer and in the outer layers of the secondary wall S1 and S3 is higher and exceeds that in the S2 layer by five- to tenfold. The highest concentration of lignin was found in the ML + P and S1 layers.

Thus, the S1 and S3 layers must have been subjected to the greatest changes during modification. It was observed that on one side of the fibre, the region adjacent to the fibre cavity was destroyed, and on the other side had the region adjacent to the intercellular space. The S2 layer is the thickest (75–85 %) of the total thickness of the cell wall (Fengel and Wegener 1989). Therefore, the S2 layer is most stable, because it has a relatively lower concentration of hemicelluloses that could turn into destructible components. As a result, it is definitely the S2 layer that determines the stability of the wood subjected to thermal treatment.

In the thermal treatment process, the chemical processes that occur in wood components are accompanied by diffusion processes, which in turn are connected to the aqueous removal of destructive products from the fibre walls.

It was originally thought that in the initial period, the high pressure in the reactor would squeeze (compress) the wood and hamper the isolation of the destruction products from the fibre walls. During the cooling stage, with a gradual decreasing of temperature and pressure, the intensity of the thermal destruction in the fibre wall diminished. The cell wall was in a plastic state due to the effect of high temperature, water vapours and low-molecular products. It was assumed that a variety of large and small pores were formed in the walls, filled with water vapours and destruction products. Pressure release in general provides conditions for the fast removal of destructive volatile and low-molecular products via water vapours from the walls through the newly formed loose regions. As a result, the removal process of destructive products is greatly intensified.

With the temperature decrease below the phase transition temperature of cellulose (60-62 °C) (Sang et al. 1999), the inner structure of fibres is gradually densified, because the cellulose framework remains almost unchanged chemically. The fibrillar structures of cellulose once again form the inner and outer dense structures through the formation of new hydrogen bonds and through the rearrangement of hydrogen bonds as the content of water and destruction products in the walls continue to

all	Mass			Lignin		Cellulose	
layers	(%)	Distribution $\left(\%\right)^{a}$	Concentration (relative units)	Distribution (%) ^a	Distribution Concentration (relative $(\%)^{a}$ units)	Distribution (%) ^a	Distribution Concentration (relative $(\%)^a$ units)
ML + P	7.2	20	2.8	70	9.7	10	1.4
SI	9.0	19	2.1	57	6.3	24	2.7
S2	76.7	31	0.4	17	0.2	52	0.7
S3	7.1	37	5.2	11	1.5	52	7.3

ants in the hirch wood libriform cell wall **Table 5** Distribution of the main *cc* decrease. The cellulose framework shrinks, giving the fibres in the cross-section an oval form. As a result, a decrease in the wall sizes was observed in fibres, vessels and rays throughout the above temperature range (Figs. 13, 14, 15). However, with increasing temperature, all processes proceeded more intensively, and therefore, all the changes in the wall sizes were greater (Table 4). So, in the temperature range 140–180 °C, the loss of the wall area of fibre gradually increased by 5.2–37.3 %; and for vessels by 6.3-16.3 % (160 °C—17.7 %).

In our opinion, the isolation of destructive products and water from the fibre wall at decreasing temperature and pressure throw-off in the reactor could be crucial for the fast removal of the destruction products and, consequently, the change in the sizes of the elements.

The results of the measurements of the fibre and vessel lumen after the thermal treatment of wood have shown that the observed changes are nonlinear with respect to the temperature. So, the fibre lumen area first increased by 2.4 % (140 °C), then by 14 % (160 °C) and finally by 4.8 % (180 °C), with a simultaneous decrease of the total fibre wall area to 37.3 %.

Thus, the unequality of the distribution of hemicelluloses in the wood cell wall, and the unevenness of the related destruction, diffusive and relaxation processes caused nonlinear changes in the sizes of the fibre cavities, but also created additional porosity in the intercellular space, weakening the mechanical properties of the wood. The results of these studies indicate that, with the modification process used, a temperature of 180 $^{\circ}$ C is probably too high for the modification of birch wood, because the types of cells present in wood (e.g. libriform, tracheids and rays) are affected with regard to their sizes and their mutual arrangement density in wood.

Conclusion

From the above studies, the following conclusions can be drawn:

The changes (area, linear sizes) in the different cell types of wood, observed with regard to the cross-section of birch wood, differ significantly depending on the thermal treatment conditions and the type of the cell.

Analysis of the SEM images of the initial birch wood cross-section showed that the area occupied by the morphological elements is approximately 78, 20 and 2 % for fibres, vessels and rays, respectively.

Minor changes in the sizes of all morphological elements (libriform, vessels, rays, annual rings) were found after the treatment at 140 °C. A considerable decrease in sizes was observed after the treatment at 160 °C, but the greatest changes were established after the treatment at 180 °C.

Fibres form the major part of wood and have the greatest effect on the total structural changes of wood following the treatment. After the treatment at 180 °C, the cross-section area of the fibres, the fibre wall area and the wall thickness were reduced, on average, by 21, 37 and 32 %, respectively.

Vessels are the least affected morphological elements of birch wood; that is, irrespective of the treatment intensity, their total area was diminished within the

range of 2.3–5.4 %. Following the treatment at 140 °C, linear sizes of the lumen decreased similarly in both radial and tangential directions. With increasing treatment temperature, the decrease in the linear sizes of the lumen in the radial direction (2.9 and 2.3 % at 160 and 180 °C, respectively) was greater than that in the tangential direction (0.5 and 0.7 % at 160 and 180 °C, respectively).

The treatment at 180 $^{\circ}$ C essentially changed the shape and size of the wood (medullary) rays located in the wood structure (cross-section); however, this did not appreciably affect the wood properties due to their low level of occurrence in that area.

Following the treatment at 180 °C, the integrity of wood morphological structure begins to break up. Voids and cracks are formed between fibres. Such a formation is one of the reasons for the decline in the mechanical properties of the wood.

A comparative SEM-method was developed as a means of studying the properties of thermally treated wood. This tool makes it possible to precisely ascertain the quantitative characteristics of a variety of fibres of wood there and to provide a more well-founded explanation for the changes in the physical parameters as well as for the changes in wood properties, including the changes in mechanical properties.

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