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ВЛИЯНИЕ ТЕРМИЧЕСКОЙ ОБРАБОТКИ НА СТРОЕНИЕ ДРЕВЕСИНЫ ГЕВЕИ БРАЗИЛЬСКОЙ (*HEVEA BRASILIENSIS* MÜLL. ARG.)

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Представлены результаты исследования влияния температуры и продолжительности термической обработки (при 180 и 220 °С в течение 15, 25 и 35 ч) на строение древесины гевеи бразильской (*Hevea brasiliensis* Müll. Arg.). Использован метод сканирующей электронной микроскопии для получения электронных микрофотографий в целях измерения толщины двойной клеточной стенки и размера люмена. Рассчитаны соотношения люмен/стенка для волокон и паренхимных клеток в радиальном и тангенциальном направлениях. Результаты работы показали, что термообработка при 180 °С не влияла или оказывала незначительное влияние на строение древесины гевеи бразильской в течение любой продолжительности обработки. Термическая обработка при 220 °С в большей мере повлияла на структуру растения: уменьшилась толщина двойной клеточной стенки, но диаметр люмена не изменился, поэтому значение отношения люмен/клеточная стенка увеличилось. Сделан вывод о том, что длительность термообработки оказывает лишь незначительное влияние на изменения в строении древесины гевеи бразильской.

Ключевые слова: термическая обработка, анатомия древесины, сканирующая электронная микроскопия (СЭМ), волокно, паренхима, двойная клеточная стенка, люмен клетки, соотношение люмен/стенка

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EFFECT OF HEAT TREATMENT ON SOME CELLULAR PROPERTIES OF RUBBERWOOD (*HEVEA BRASILIENSIS* MÜLL. ARG.)

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The effect of different treatment temperatures and different treatment durations on the certain anatomical properties of the rubberwood (*Hevea brasiliensis* Müll. Arg.) was examined. Rubberwood samples were treated at 180 ° and 220 °C for 15, 25 and 35 hours. SEM pictures were taken to measure double cell walls, cell lumens; and the lumen/wall ratio was calculated for fiber and parenchyma cells, in radial and tangential directions. Treatment at 180 °C had no or only a slight effect on the anatomy of rubberwood for any treatment duration. The treatment at 220 °C has an effect on the anatomy of rubberwood: the double cell wall size decreased, the lumen diameter did not change, so the lumen/cell wall ratio increased. The treatment duration has only a slight effect on the changes.
Keywords: heat treatment, wood anatomy, SEM, fiber, parenchyma, double cell wall, cell lumen, lumen/wall ratio

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Numerous studies deal with the anatomy of different tree species in many respects, for example: [1, 2]. Some studies focused on the dimensions of wood cells and their changes in heat treatment. Hietala *et al.* [3] used liquid state NMR for measurements and did not find significant differences in the pore size of Scots pine (*Pinus sylvestris* L.) before and after heat treatment at 180 ° C and 230 °C for 4 h. Ahmed *et al.* [4] studied the preservative uptake of thermally modified aspen (*Populus tremula* L.) and birch (*Betula pendula* Roth.). Gunduz *et al.* [5] used an environmental scanning electron microscope (ESEM) to compare the anatomy of the healthy and infected (Chestnut Blight Diseased) chestnut tree (*Castanea sativa* Mill.) and described several changes in the anatomy of the cambium and the wood.

Kocaefe *et al.* [6] examined jack pine (*Pinus banksiana* Lamb.) samples by several methods, including scanning electron microscopy (SEM). They found only slight effects: micro-cracks were formed on cell walls during the heat treatment at 210 °C. Anderson *et al.* [7] found similar results: thermal modification did not change the anatomy of the Scots pine (*Pinus sylvestris* L.) at the micrometer level, but they found that the surface of the pores increased because of the cracks in the cell wall observed. Heat treatment of red cedar (*Thuja plicata* Donn ex D. Don) at 200 °C for 1 and 2 h destroyed tracheid cell walls, caused despiration of the pits, which resulted more openings in the wood [8]. Similar changes were found in aspen and birch wood by Ahmed *et al.* [4]. Batista *et al.* [9] investigated the anatomical changes of *Eucalyptus grandis* W. No significant changes in fibers, vessels and parenchyma cell were noted. Bakar *et al.* [10] treated red oak (*Quercus rubra* L.), Eastern red cedar (*Juniperus virginiana* L.) and rubberwood (*Hevea brasiliensis* Müll.Arg.) at 120 °C and 190 °C for 2 and 8 h and used SEM to investigate the anatomical changes. They found that most of the cells in all heat treated samples had some deformation, such as collapse. They thought that high temperature softened the components of the cell wall, causing closing of the cell lumens and loss of wood strength. Boonstra [11] and Boonstra *et al.* [12, 13] examined the effects of heat treatment on soft and hardwood species. Radial and tangential cracks were observed in different amounts. Cracks near the rays were often observed and collapse and deformation of vessels and libriform fibers was found in hardwoods. Ling *et al.* [14] treated *Populus cathayaha* Rehder at 180 °C, 200 °C and 220 °C for 4 h. The anatomical structure of the wood was examined, and an increase in the number of the distorted and cracked cell walls was observed. The average wall thickness to lumen diameter ratios of the fibers was also calculated and it was found that fiber walls thickened, while the lumen of the fibers shrank. Biziks *et al.* [15] measured the total area, wall area, lumen area, wall thickness and lumen linear size of fibers and vessels of birch (*Betula pendula* Roth.), before and after heat treatment (140 °C, 160 °C and 180 °C, 1 h). With the increasing treatment temperature, the fiber cross-section sizes decreased significantly. Parallel to this, cracks appeared in the middle lamella and the shape of the fibers became more round. The effect of the treatment parameters on the vessel walls was relatively small. The rays also became more visible after the treatments. Bernabei and Salvatici [16] made real time observations with ESEM, during heat treatment of spruce wood (*Picea abies* Karst.). Up to 100 °C the swelling of the cell wall thickness was observed. Subsequently

up to 200 °C no changes were observed, but over 200 °C a great reduction of the cell wall thickness was seen. At the end of the observations, the cell lumen decreased by 10 % too.

Most of the studies used a shorter treatment duration. Based on the above, it appears that short-term, lower-temperature treatments have barely detectable results, while the effects of longer or higher temperatures (above 180 °C) changes can be detected.

The aim of this research was to examine the effect of different treatment temperatures 180 °C and 220 °C and different treatment durations 15, 25 and 35 hours on the certain cellular properties of rubberwood (*Hevea brasiliensis* Müll.Arg.).

Materials and methods

Rubberwood samples derived from the region of Nakhon Si Thammrat province in Thailand. The samples were cut from the same board with the dimensions of 10×10×10 mm (Fig. 1). The samples were prepared before the treatment, later no other changes were made.

According to the heating schedule, the rubberwood samples were heated from room temperature to 90 °C in five hours, from 90 °C to 130 °C in another 5 hours, and the top (180 °C and 220 °C) temperature was achieved two in hours. Three different treatment durations (on constant 180 °C and 220 °C) were used which last 15, 25 and 35 hours. For the soft cooling, the thermal inertia of the chamber was used and the whole system cooled down to room temperature after about 15 hours. For the treatment a custom-made laboratory chamber was used. The system is an open (not air tight) and dry system. As the system is open, the water content of the small samples is completely removed during the heat treatment. This affected all samples equally, since this stage of treatment was the same in all cases. After heat treatment, the samples were placed in a climate chamber (20 °C, 65 %), which was followed by SEM examination. Because of the conditions inside the electron microscope, the samples again lost most of their water content. This water loss does not cause any further cracks. Consequently, the differences were caused by peak temperature treatments.

The samples were examined with a Hitachi-3400N scanning electron microscope, 10 kV acceleration potential was used and the working distance was around 10 mms. The samples were coated with gold-palladium. The cross-section of the rubberwood was taken for the measurement at 100–1,000 magnification. The measurements were done with Image-Pro Plus 7.0 software (Media Cybernetics Inc.).

The following properties were measured and calculated (see Fig. 2):

– fiber double cell wall thickness (FW) [μm];

– fiber lumen diameter (FL) [μm];

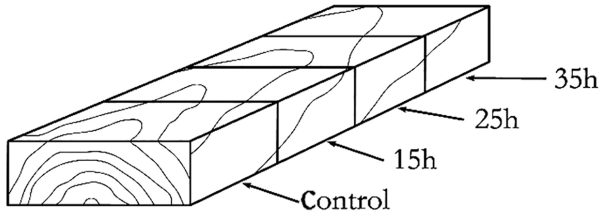


Fig. 1. Sampling method: The specimens came from the same board and followed each other in the longitudinal direction

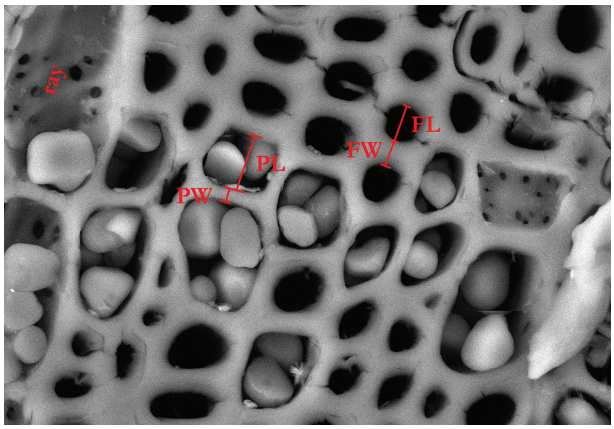


Fig. 2. Measured cell properties in radial direction. Cell wall thickness (PW) and lumen diameter (PL) of parenchyma and of fiber (FW and FL respectively) were measured

– fiber lumen/double cell wall ratio (FLW) [dimensionless];

– parenchyma double cell wall thickness (PW) [μm];

– parenchyma lumen diameter (PL) [μm];

– parenchyma lumen/double cell wall ratio (PLW) [dimensionless].

At least 100 measurements were made from each type. The parameters of the cells were separately measured in radial and tangential directions.

In addition to the above, pictures were taken of cracks and changes in cell walls. Longitudinal images were also made for this purpose.

Results and discussion

It is noteworthy that in many cases, the cell wall thickness of parenchyma cells is less in the tangential direction than radial. The reason for this is that the parenchyma cells are arranged in tangential lines and bands besides the rays. Thus, in many cases in a tangential direction, a thin-walled parenchyma is also a neighbour of a parenchyma cell, while radially most often there is a thick-walled fiber.

In many cases, the latewood fibers are flattened radially, so the average size of the lumens of the fiber cells higher in the tangential than in the radial direction.

Fig. 3 and Table shows the changes of cell wall thicknesses and lumen diameters of the fibers and parenchyma cells of heat treated rubberwood compared to control samples.

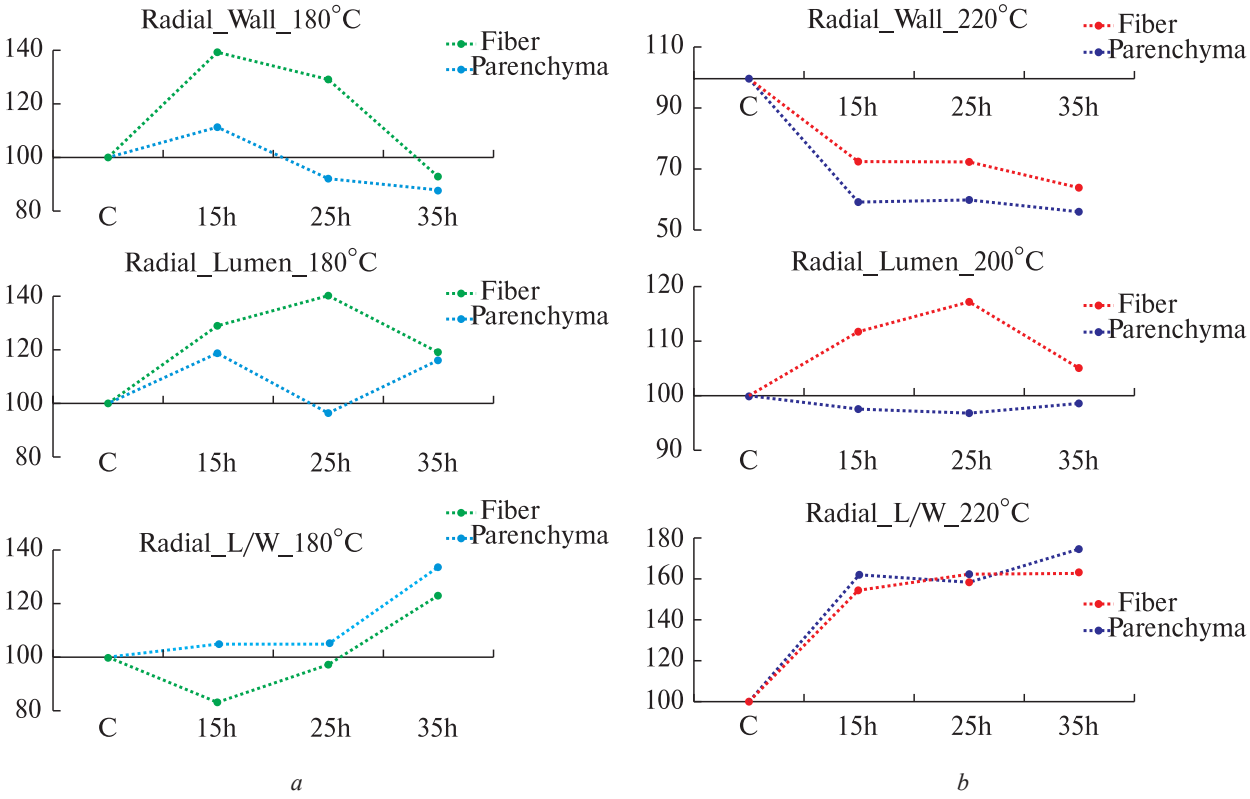


Fig. 3. Changes of average cell wall thickness, lumen diameter and lumen/wall ratio for fibers and parenchyma of rubberwood, at 180 °C (*a*) and 220 °C (*b*) treatment for 15, 25 and 35 hours, and for radial direction (C = control) on the base of control (100 %)

Changes (%) of average cell wall thickness, lumen diameter and lumen/wall ratio of fibers and parenchyma cells of rubberwood, at 180 °C and 220 °C treatment for 15, 25 and 35 hours (C = control) on the base of control (100 %)

Temperature, °C	Cell type*	Radial											
		Double cell wall				Cell lumen				Lumen/Wall ratio			
		C [μm]	15 h [%]	25 h [%]	35 h [%]	C [μm]	15 h [%]	25 h [%]	35 h [%]	C [μm]	15 h [%]	25 h [%]	35 h [%]
180	F	**6,4 (100%)**	110,9	92,2	88,6	**9,9 (100 %)**	117,8	96,8	115,4	**1,6 (100 %)**	104,7	104,6	131,9
180	P	**2,8 (100 %)**	138,4	128,2	92,7	**17,2 (100 %)**	127,4	138,1	117,9	**7,3 (100 %)**	84,3	97,4	121,9
220	F	**8,1 (100 %)**	72,2	72,6	64,0	**9,4 (100 %)**	111,5	117,0	105,1	**1,3 (100 %)**	153,4	160,6	161,0
220	P	**3,9 (100 %)**	59,6	59,9	56,4	**21,5 (100 %)**	97,8	96,8	98,6	**6,4 (100 %)**	160,6	156,7	173,0
Tangential													
180	F	**6,8 (100 %)**	90,5	91,7	92,4	**11,6 (100 %)**	103,5	97,8	135,6	**1,8 (100 %)**	110,3	103,9	145,4
180	P	**2,4 (100 %)**	120,5	126,2	109,5	**13,9 (100 %)**	135,5	123,1	145,2	**6,3 (100 %)**	112,4	96,1	139,8
220	F	**6,2 (100 %)**	84,7	83,6	71,7	**13,2 (100 %)**	94,0	99,0	82,8	**2,2 (100 %)**	112,6	122,9	119,2
220	P	**2,9 (100 %)**	59,6	67,2	57,0	**19,2 (100 %)**	93,0	88,1	84,7	**7,2 (100 %)**	153,1	129,2	148,2
*Cell type: F — fiber; P — parenchyma.													

Treatment at 180 °C

No significant difference was observed in the fibers or parenchyma cells compared to control samples. In the 15-hour treatment, there is a slight increase in the cross-section of the cell walls as in the parenchyma and in the fiber cells, which disappears during longer treatments. Changes are similar in the cell lumen as well. However, the rate of decrease is less at 35 hours of treatment, so the average size of the cell cavities is greater than the baseline (control) value. This slightly larger average size can be explained by the larger amounts of earlywood in these samples. As a result, the cell lumen / cell wall ratio hardly changes during the 15 and 25 hour treatments, while the 35 hour treatment shows a marked increase.

In contrast, Ling *et al.* [14] was able to detect changes in the cell lumen/cell wall ratio at 180 °C for 4 hours in poplar. Some authors observed in short-term 180 °C or lower temperature hydrothermal treatment, the decrease of the cell wall area and the increase of the lumen area and linear size [15]. On the other hand, Bernabei and Salvatici [16] found that at approximately between 100 °C and 200 °C, the cell wall dimensions remained almost constant; only a slight decrease could be detected. Due to the time-elongation of the process and the increase in temperature, the degradation of hemicelluloses was observed during hydrothermal treatment.

The treatments in our study were much longer and the treatment occurred in the presence of oxygen but with the exclusion of steam, which may have led to the disappearance of the initial cell wall swelling as the treatment progressed.

Treatment at 220 °C

In the radial direction, the cell wall of the parenchyma cells the median and the average of control group is the largest, and for all treatments the median and average values are lower. In contrast, cell lumens do not differ in size from the control group, but for fibers a slight increase can be observed, but this could be from the fluctuation of the measurement. Hence the ratio of lumen to cell wall is higher for treated samples. The same can be said for the tangential direction and for fiber cells in both directions (Table). As a result of the 220 °C heat treatment, all the treatment times had an effect on the anatomical structure: the average cell wall thickness decreased, the size of the cell lumen remained unchanged, while the lumen/cell wall ratio increased. However, there was no detectable difference between treatment durations; only minor fluctuations were detected due to different rates of earlywood and latewood.

The reduction of cell wall thickness was observed above 180 °C (even more above 200 °C) in other studies too [15–17], which is mostly explained by the

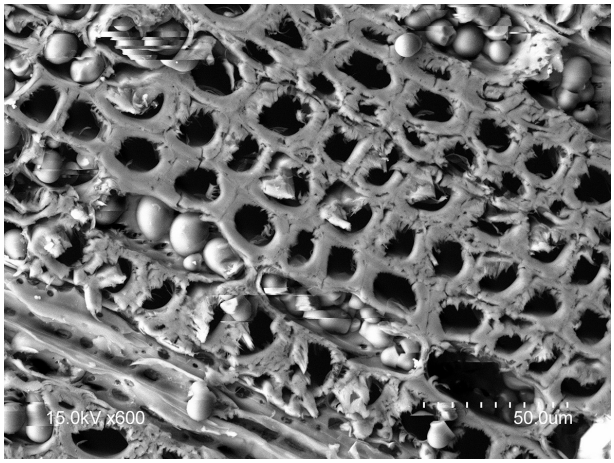


Fig. 4. Separation of adjacent cells of rubberwood treated at 180 °C, for 15h

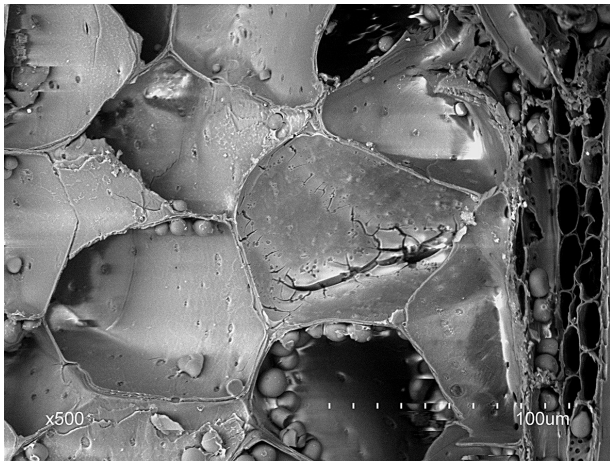
decomposition of hemicelluloses. The decomposition of hemicelluloses in an inert atmosphere begins at about 200 °C [18–20], and under this temperature, mainly hydrolysis can be detected in the presence of vapor. The degradation of hemicelluloses occurs in the same temperature range in the presence of oxygen [21]. It can be explained with the onset of the decomposition of hemicelluloses that the treatment at 180 °C had no detectable effect, but decomposition had already begun at 220 °C (above 200 °C), resulting in a reduction in the cross-sectional size of the cell walls. As there was no significant change in the size of the cell lumens, the lumen/cell wall ratio also changed with the decreasing cell wall size.

Other observations. Samples exposed to 180 °C heat treatment have already seen a change in the middle lamella. The pattern of the middle lamella becomes more visible on samples treated for 15 hours, while in the longer-term treatments the partial or complete separation of the adjacent cells are seen (Fig. 4) (180 °C; 15 h).

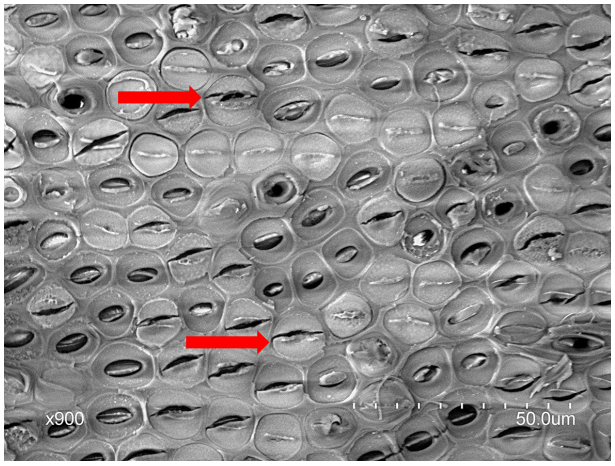
There are more changes in samples treated at 220 °C. A number of cracks were found on the samples when scanning specimens (Fig. 5, *a–f*). Although no numerical survey was performed, the number of cracks is likely to increase as the duration of the heat treatment increased.

Vessels. Changes were found in vessels mainly on intervessel bordered pits (Fig. 5, *b*): In most cases, the cracks start from the "corner" of the aperture of the pits and often run along the entire length of the pit; (see arrow). In some cases, the direction of the crack does not follow the direction of the aperture. A crack can run around the edge of the pit too; (see arrow). Fissures were also observed on the tyloses, especially where the adjacent cell walls meet (Fig. 5, *a*).

Axial and ray parenchima. Cracks appeared around the axial parenchymal cells too. Parenchyma cells separate from surrounding fibers (Fig. 5, *c*).



a



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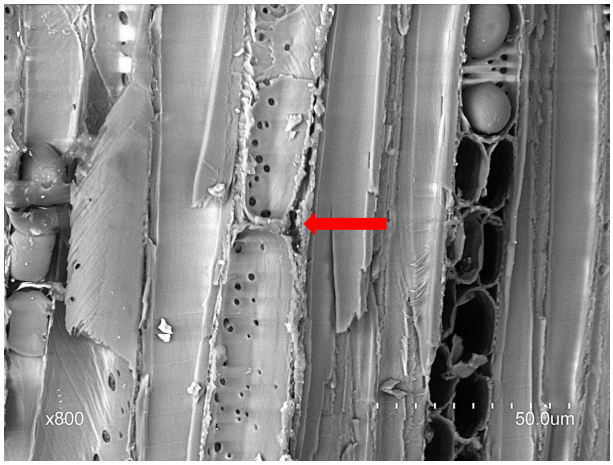

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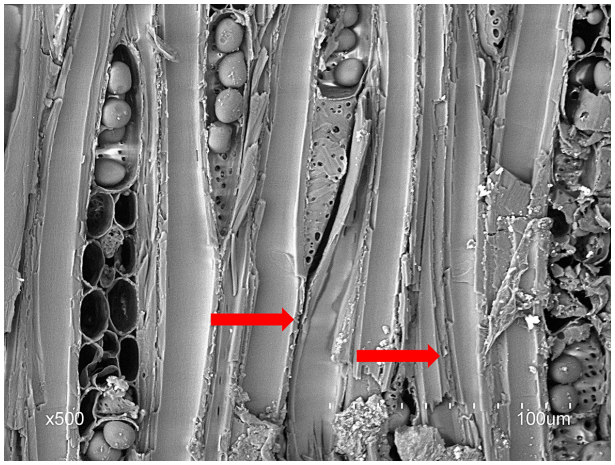
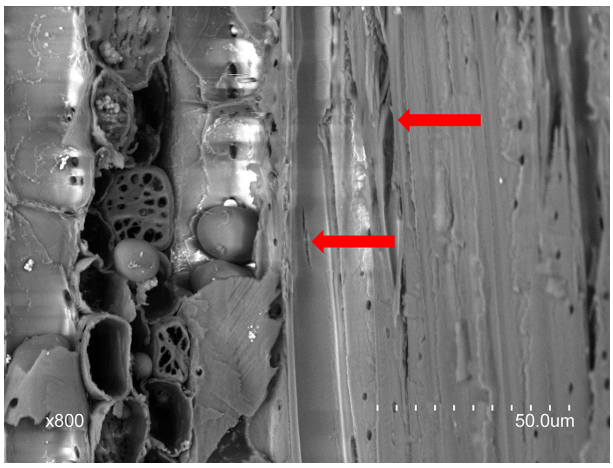
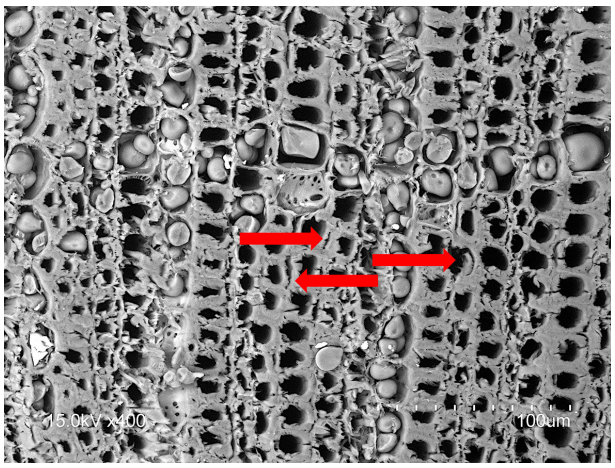


d



e



f

Fig. 5. Images of typical degradation during heat treatment of rubberwood (220 °C)

Cracks often developed around the rays, which are conspicuous in cross sections as well and it seems like the ray has opened (Fig. 5, *d*). On the other hand, like Biziks and his colleagues [15], the deformation and opening of the rays were observed. This was mainly found when the cross-section of the marginal cell of the rays was formed. As the rays of the rubberwood are 1–4-seriate and are characterized by larger, upright marginal cells [22], the cracks start mainly from these marginal cells. Gilani *et al.* [17] also found, that the microcracks appear mainly in the multiseriate rays of the beech.

Fibers. Smaller cracks can occur in the wall of the fibers. In many cases, the micro-cracks start from the pits. These cracks follow the directions of cellulose fibrils; they are only rarely perpendicular to it (Fig. 5 *e*,*f*). Sometimes these cracks are linked to each other and create a network. Similar micro-cracks have been found by Kocaefe *et al.* [6] on jack pine, heated to 190–210 °C. Isolated fibers or separated cell wall layers often can be seen (Fig. 5, *f*). The separation of the cells usually occurred at the middle lamella. Similar delaminations were also observed by Kocaefe *et al.* [6], Biziks *et al.* [15] and Ling *et al.* [14].

Conclusions

Treatment at 180 °C had no or only a slight effect on the anatomy of rubberwood at any treatment durations. The average size of both the cell wall and the cell cavity increased as a result of the treatment, so the lumen/wall ratio did not change. The fluctuations of the measured properties may result from the different ratios of earlywood and latewood.

SEM pictures show that treatment at 180 °C had only a slight effect on rubberwood: changes of the middle lamella were observed, followed by the separation of neighboring cells.

The treatment at 220 °C has an effect on the anatomy of the rubberwood: the double cell wall size decreased, the lumen diameter did not change, so the lumen/cell wall ratio increased.

The treatment duration at 220 °C had no effect on the anatomy of rubberwood. The changes were made during the shortest 15-hour treatment; the longer treatment did not cause any further detectable change.

There are more anatomical changes at 220 °C treatments. SEM images show changes in all cell types, and many new cracks appear in the wood and in the cell walls.

Acknowledgement

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