**RIGA TECHNICAL UNIVERSITY**
Faculty of Materials Science and Applied Chemistry
Institute of Polymer Materials

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Candidate for a Doctor's degree in the study program "Chemical technology"

# IMPROVEMENT OF THE PROPERTIES OF DECIDUOUS WOOD BY THE THERMAL TREATMENT METHOD

**Summary of the Doctoral thesis**

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The Doctoral thesis is available at the library of the Riga Technical University, 10 Kipsalas Street, Riga, LV-1659 and the National Library of Latvia, 5 Anglikāņu Street, Riga, LV-1050.

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**CONFIRMATION**

Hereby I confirm that I have worked out the present Doctoral thesis, which I submitted for consideration at the Riga Technical University for acquisition of a Doctoral degree in engineering. The present Doctoral thesis is not submitted in other scientific institutions for acquisition of a scientific degree.

Vladimirs Biziks ………………

Date: 14.11.2011.

The Doctoral thesis is written in Latvian; it contains Introduction, References review (3 chapters), Experimental (8 chapters), Results and discussion (12 chapters), Conclusions and the used References list. The Doctoral thesis contains 148 pages, 52 figures, 48 tables, 37 equations, 9 attachments and 133 references.

## ACKNOWLEDGEMENTS

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## CONTENT



## ABRIDGEMENTS

HB – Brinell hardness
HTM – hydrothermally modified
CWU – capillary water uptake
EMC – equilibrium moisture content
MPa – mega Pascal
ASE – anti-swelling efficiency
RH – relative humidity
SEM – scanning electron microscopy
TM – thermal modification
TMW – thermally modified wood
IS – impact strength
$MOE_{dyn}$ – dynamic modulus of elasticity

## ESSENCE AND URGENCY OF THE PROBLEM

Recently, for economic and ecological reasons, the tendency of a wider application of the wood of local tree species has grown. This is determined by several factors: the accessibility of high-quality and tropical wood declines, the demand for renewable environmentally friendly materials for indoor and outdoor applications grows; from 2012, it is forbidden in the EU to import and use tropical wood of unknown origin. In the global aspect, the wider application of local deciduous wood spares tropical forests and favours the carbon sequestration, decreasing the global warming.

In Latvia, the local stock of deciduous trees is ~251 million $m^3$, of which about a half (56%) is birch, 22% is aspen, 20% is alder, and 2% is ash-tree. In the recent years, studies on the use of soft deciduous wood for producing products with a higher added value have been started also in Latvia. The practical application of soft deciduous wood in construction and civil engineering is limited by the low durability against biodegradation (the lowest durability class 5 conforming to the EN 350-2 classification) [9]. Upgrading the durability properties of this wood, in some fields, it is possible to replace with it the traditionally applied coniferous wood, leaving its resources to the fields, in which it is irreplaceable, for example, for bearing structures.

Thermal modification, in comparison with chemical modification or protection with biocides, is an ecological method to upgrade the physical and biodurability properties of wood without the human and environmentally hazardous compounds. In the treatment, new high-quality competitive construction materials with the properties that essentially differ from those of the raw material are obtained. Because modified wood does not contain hazardous compounds, it can be easily utilised at the end of the life cycle. The evaluation of the data on the life cycle, available in the literature, testifies that the modified wood, from the viewpoint of production, application and utilisation, is potentially a very environmentally friendly material. The thermally modified wood can be used in indoor (in dry and humid conditions) and outdoor conditions (CEN/TS 15679 [3]). The market of the thermally modified wood is expanding each year, besides EU, embracing Russia, North America, South America, Middle East, and Asian countries.

Originally, much attention was given to the modification of coniferous wood, but there is still comparatively scarce information on the modification of deciduous wood. The modification of soft deciduous wood in water vapour medium at elevated pressure is investigated little worldwide. There are not sufficient data on the hydrothermal treatment of alder, because this species has no industrial value in many regions. As the optimal regimes for thermal modification of deciduous wood (aspen, birch, ash-tree), temperatures from 180 to 220°C are recommended. In such a temperature range, the mechanical properties of wood as a material dramatically decline. Therefore, to forecast the properties of the modified material and its stability in the service conditions, it is important to elucidate not only the properties of the obtained product, but also to understand the processes that occur in wood as a result of the thermal action, carrying out for this purpose a multidisciplinary approach to the chemical composition of wood as well as the changes in the microstructure, and physical and mechanical properties. Such an approach is realised, working out this Doctor's thesis. Evaluating the service properties of the modified wood in compliance with CEN/TS 15679:2007, the corresponding standards of testing the unmodified wood products (sawn timber, parquet, edging, etc.), including evaluating biodurability, mechanical strength and surface properties, are applied.

**Urgency of the topic**

In recent years, interest in the possibility to obtain new products with a higher added value from local deciduous wood has considerably grown. The amount of the high-quality wood produced from both grey alder and aspen wood has increased, and the local woodworking enterprises (SIA "Ošukalns", SIA "4Pluss", SIA "Woodex", SIA LUK") show interest in the production of thermally modified wood in Latvia, promoting deeper processing of the local, less used tree species. As promising, we evaluate the thermal modification of deciduous wood (alder, aspen and birch) in the water vapour medium at elevated pressure for products used in humid indoor and outdoor conditions. As has been already mentioned, there are no data in the literature on the hydrothermal modification of grey alder wood, and there are only several publications, in which the results of the treatment of aspen and birch wood are considered, although the working cycle diagrams and parameters are kept in secret as „know-how". The next logical step after choosing the technology was to elucidate the hydrothermal (HT) process parameters, so that to produce products with the desired properties from the deciduous wood grown in Latvia.

**Aim of the Doctoral thesis**

To elucidate the optimal parameters of the hydrothermal treatment process to improve the form stability, as well as the hygroscopic and bio-durability properties of wood, extending its use in different construction objects in humid and outdoor conditions.

**To reach the aim, the following tasks are advanced in the Doctoral thesis:**

- based on the data available in the literature, the choice of a deciduous wood thermal treatment technology for further studies, with the aim to produce deciduous wood with improved service properties for use in high humidity and outdoor conditions;
- producing of samples (boards) of the corresponding size for modification treatment, their preparing for modification, characterisation of the properties (sizes, moisture, density);
- modification of wood samples in laboratory multifunctional experimental equipment in an exterior heating autoclave in water vapour medium at **five** **(three**

+ **additional two optimised)** regimes. Modification were carried out at temperatures of 140°C, 160°C, 170°C and 180°C, as well as varying the holding time at the maximum treatment temperature 160°C;

- determination of mass losses, as well as size and density changes, depending on the treatment regime;
- determination of the wood component composition (cellulose, lignin, extractives and hemicelluloses) and its changes after the thermal treatment (chemical analysis);
- determination of the chemical composition of the condensate after each treatment regime;
- changes in bending, modulus of elasticity, Brinell hardness, and dynamic impact strength properties for modified birch, aspen and grey alder wood samples depending on the treatment regime, in comparison with the case of untreated wood;
- durability of thermally treated wood against 3 rot fungi *(Coniophora puteana, Poria placenta, Coriolus versicolor* according to EN 113 [5], as well as after washing according to EN 84 [4]; comparison with untreated wood;
- evaluation of the biodurability, mechanical strength (MoE) and surface quality changes for grey alder samples (block test) exposed in outdoor conditions depending on the treatment. Surface changes for the wood obtained at optimised HTM regimes after exposure outdoors and in an accelerated ageing laboratory chamber;
- changes in the wood structure in the HTM process, using water vapour sorption and scanning electron microscopy methods;
- effect of the HTM regime on the hygroscopic properties of wood;
- determination of the optimal parameters of the HT treatment regime, based on the biodurability of wood against rot fungi, hydrophility and mechanical strength properties;
- evaluation of the realisation potentiality and products' applicability fields for the developed HTM wood production technology in Latvia.

**Scientific novelty of the Doctor's thesis:**

Optimal parameters for hydrothermal modification in an exterior heating autoclave, which extends the application potentialities of local deciduous wood (especially alder and aspen) in outdoor conditions with elevated humidity, are found and applied. It has been substantiated that, for soft deciduous wood, the treatment at lower temperatures (160°C or 170°C) is the optimal one, which contradicts the data available in the literature.

The obtained results on the changes of the impact strength properties of HTM deciduous wood in time, using dynamic impact strength equipment and obtaining destruction force-time curves not only demonstrate the effect of the treatment intensity on the material, but also allow to evaluate, how the material's load resistance changes. Such a study and the generalisation of the results on HTM wood has not been performed up to now.

A comparative scanning electron microscopy (SEM) method for the study of thermally modified wood has been developed, which makes it possible to obtain most precise quantitative characteristics of different anatomical elements of wood. The results on the deciduous tree libriform, vessel, wood rays cells' cross-sectional linear and area sizes' changes after thermal treatment make it possible to explain, with greater reason, the properties of modified wood, especially the reasons for mechanical strength changes.

**Practical significance of the work:**

1) The results of the work can be used for establishing environmentally friendly HTM deciduous wood productions, suitable for Latvia, which would enable the production of higher added value products from renewable resources.
2) The found HT treatment parameters essentially improve the properties (form stability, durability against rot fungi, hydrophobicity) of deciduous wood at relatively small mechanical strength losses.
3) Two optimal hydrothermal modification regimes are developed, which make it possible to obtain deciduous wood for the use in outdoor conditions, without the contact with soil (conforming to the use class 3 according to EN 335-1 [7]), with the durability classes 1 or 2 against rot fungi (according to EN 350-1 [8]), with improved form stability, lower equilibrium moisture and proportionate mechanical properties.

**Approbation of the results of the work**

The main scientific achievements and results of the Doctoral thesis are presented at 10 international scientific conferences. On the topic of the Doctoral thesis, there are 16 publications, including 12 papers in conferences' proceedings (2, 4, 7-16), 3 papers in books of abstracts (3, 5 and 6), 2 papers are submitted to the internationally cited journals: *Holzforschung* and *Wood Science and Technology*.


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# SUMMARY OF THE DOCTORAL THESIS

**Introduction** – the urgency of the Doctoral thesis is grounded, the aim and tasks are formulated, also the basic statements of the Doctoral thesis are outlined.

**The first chapter** is devoted to the literature review, in which the thermal modification of wood is considered as an alternative for biocides for upgrading wood biodurability. A small review of the development of wood thermal treatment is made. The main methods for thermal treatment of wood applied worldwide as well as their technological characterisations are described and compared. The volumes of thermally modified wood and tendencies in Europe and worldwide are shown. The choice of the products as well as the basic principles of their standardisation and classification are shown. The relationship between the changes in the physico-mechanical properties, biodurability and other properties of wood and the changes in the chemical composition is considered. The perspectives and limitations of deciduous wood production in Latvia are forecasted.

**The second chapter** is devoted to the experimental part, in which the choice of the wood samples is grounded and the preparation of samples is described, also the materials, methods and equipment used in the work are reflected. In the Doctoral thesis, for the development of new technological regimes, a scheme is developed, shown in Figure 1.

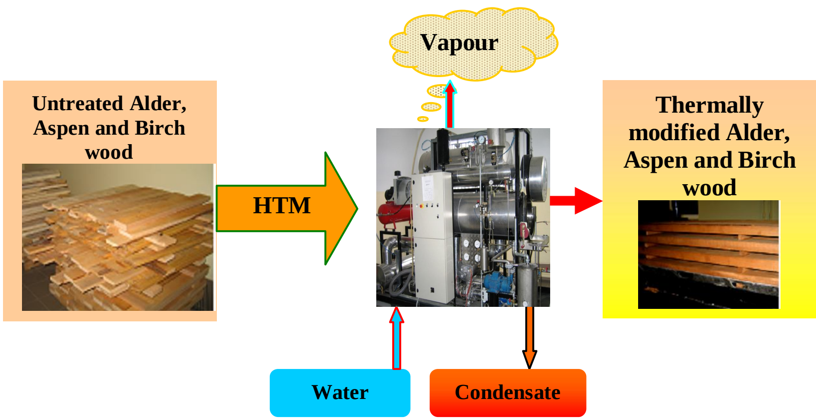



Figure 1. Producing of thermally modified deciduous wood

A characterisation of multifunctional wood modification equipment used for developing HTM regimes is presented. The initially investigated regimes for hydrothermal modification of deciduous wood (Table 1) are characterised and parameters for process optimisation are additionally developed (Table 2).

Table 1

Set parameters for investigating thermal modification process

| Treatment temperature, ˚C | Holding time at the maximum temperature, h | Temperature rise / cooling rate, degrees/min | Pressure, MPa |
|---|---|---|---|
| 140 | 1 | 0.24 – 0.28 / 0.11 – 0.14 | 0,44-0,45 |
| 160 | 1 | 0.24 – 0.28 / 0.11 – 0.14 | 0,62-0,63 |
| 180 | 1 | 0.24 – 0.28 / 0.11 – 0.14 | 0,94-0,96 |

Table 2

Additionally set regimes for optimising the modification process

| Treatment temperature, ˚C | Holding time at the maximum temperature, h | Temperature rise / cooling rate, degrees/min | Pressure, MPa |
|---|---|---|---|
| 160 | 3 | 0.24 – 0.28 / 0.11 – 0.14 | 0,63-0,65 |
| 170 | 1 | 0.24 – 0.28 / 0.11 – 0.14 | 0,78-0,80 |

As can be seen from the working cycle diagram shown in Fig. 2, the thermal modification process can be divided into three stages.

In the course of the process, as water evaporated, depending on the treatment temperature in the chamber, pressure grew, reaching 5-9 bars. Modification temperatures were 140°C, 160°C or 180°C. The treatment process had three technological stages, namely, (1) temperature increase up to the modification temperature; (2) holding at the modification temperature (1 h); and (3) cooling.

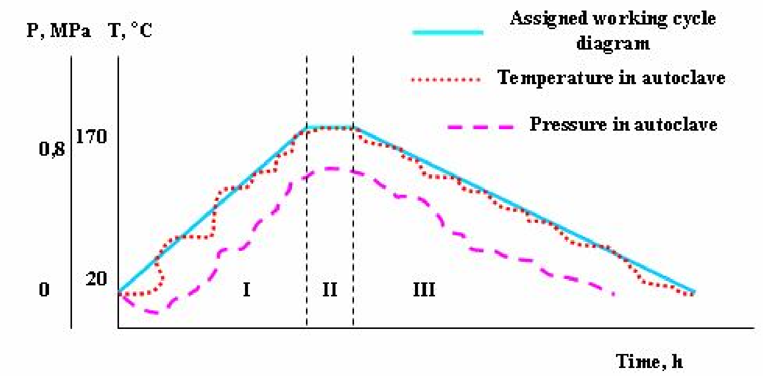


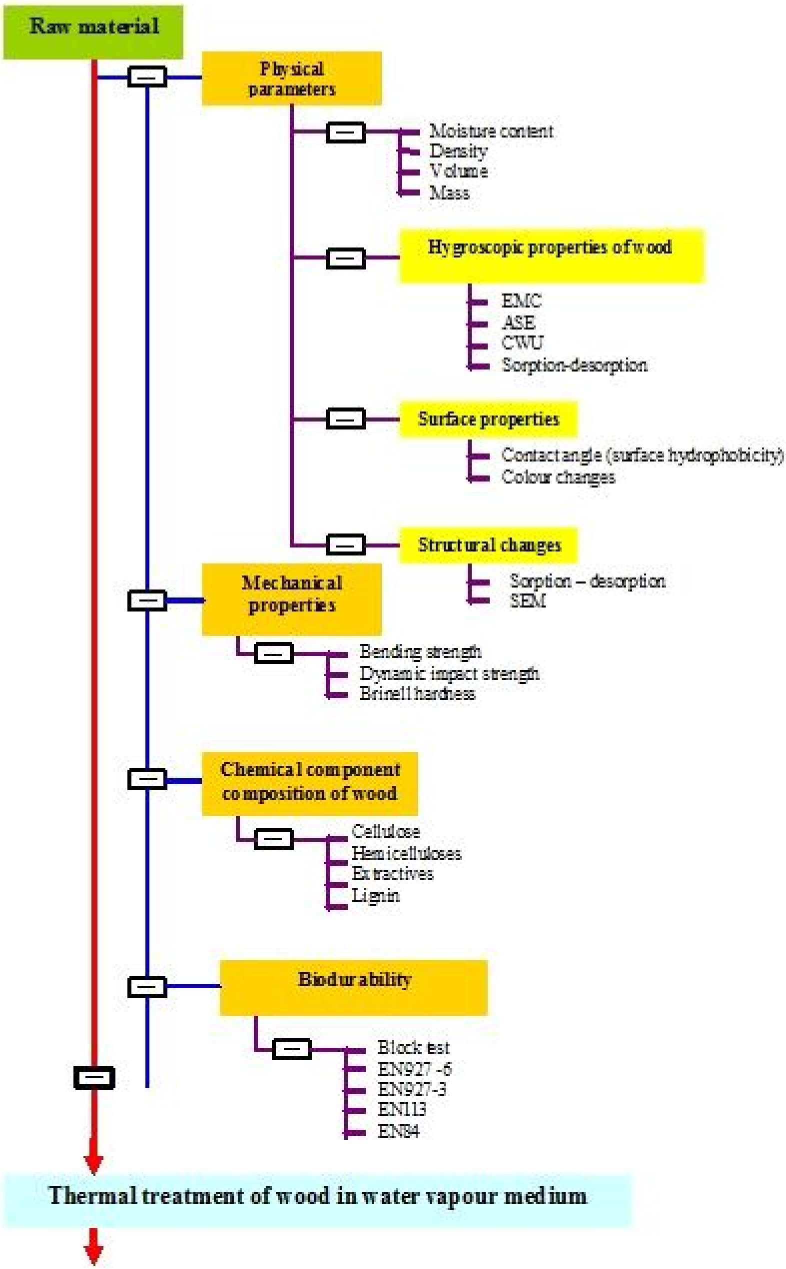
Figure 2. Diagram of the working cycle of thermal modification at 170 ˚C

The experimental course of the Doctoral thesis and the analysis methods applied are schematically shown in Fig. 3.

**The third chapter** – based on the information summarised in chapters 1 and 2, the results of the work are analysed and their evaluation is presented.

**Conclusions** – the achieved results of the work are formulated and the most essential statements are defined.

**References list** – the references used in the work are listed, based on which the directions of the study are determined, and the obtained results are compared.



Raw material
Physical parameters
Moisture content
Density
Volume
Mass
Hygroscopic properties of wood
EMC
ASE
CWU
Sorption-desorption
Surface properties
Contact angle (surface hydrophobicity)
Colour changes
Structural changes
Sorption – desorption
SEM
Mechanical properties
Bending strength
Dynamic impact strength
Brinell hardness
Chemical component composition of wood
Cellulose
Hemicelluloses
Extractives
Lignin
Biodurability
Block test
EN927 -6
EN927-3
EN113
EN84
Thermal treatment of wood in water vapour medium

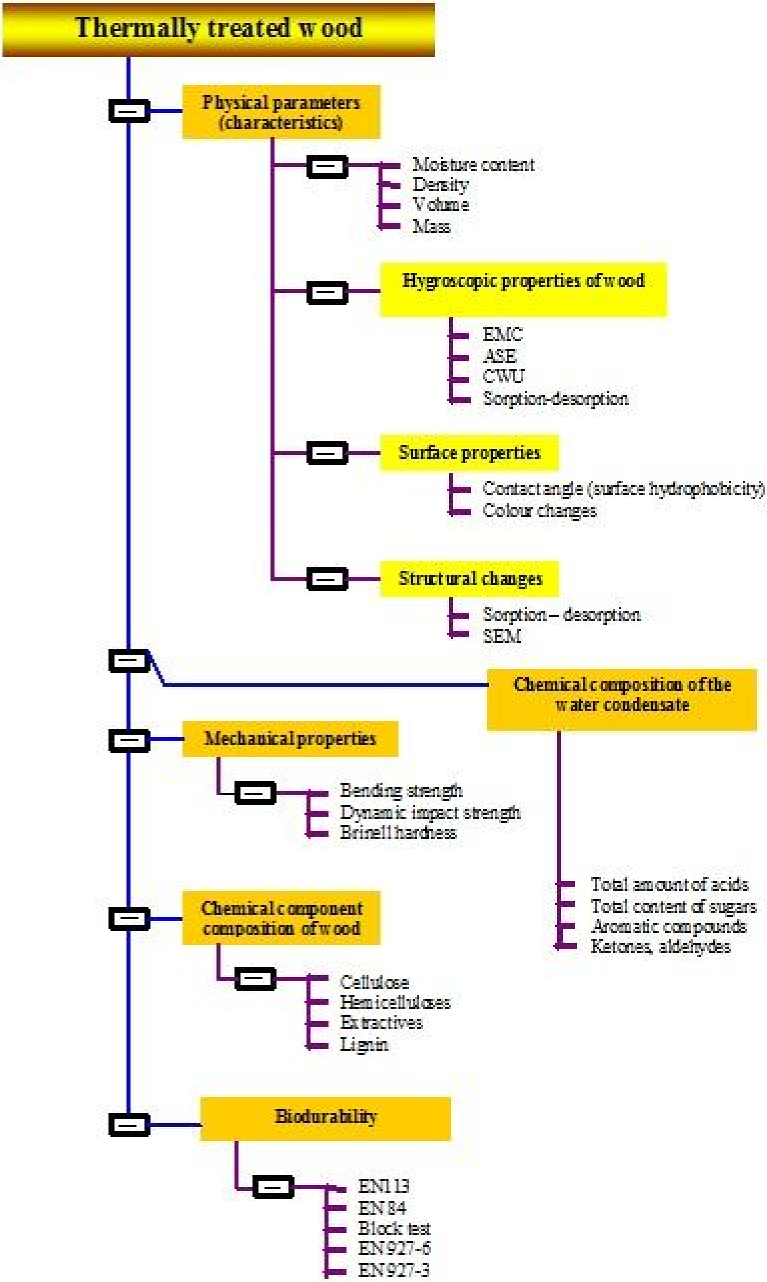



Figure 3. Course of the experiment and analysis methods applied

## RESULTS AND DISCUSSION

In the studies on the upgrading of deciduous wood properties, the thermal treatment method was chosen. Because multifunctional wood modification equipment, available at the LS Institute of Wood Chemistry, was chosen, the optimal modification conditions were adjusted to the exterior heating autoclave. We have concluded from the information summarised in the literature that thermal modification of wood in the water vapour - the so-called hydrothermal modification (HTM) is promising. To use in outdoor conditions the modified wood, obtained in an autoclave under elevated pressure in a HTM process, we have imposed the requirements corresponding to the technical specifications CEN/TS 15679 [3], and also introduced requirements for mechanical strength and form stability properties.

Beginning the optimisation of the HTM process regimes, we have developed and applied the first three hydrothermal modification regimes, for which we changed the maximum modification temperature. As the lowest temperature, 140°C was chosen; the temperature was increased by 20°C in each next regime. In the regimes chosen by us, not only temperature, but also pressure and water vapour (steam) concentration in the autoclave are varied (0.42 MPa at 140°C, 0.62 MPa at 160°C and 0.95 MPa at 180°C). These parameters vary depending on the process stage: at the temperature rise stage, pressure grows; at the holding stage, it practically does not change, and decreases to the atmospheric pressure at the cooling stage. With varying temperature and pressure, the wood autohydrolysis rate, the volumes of the volatile low-molecular products and other processes change. Hence, developing the first three regimes, we took into account only two variable parameters, namely, maximum temperature and pressure of the thermal treatment.

To characterise the HTM deciduous wood, we determined hydrophobicity, anti-swelling efficiency (characterises the form stability), mechanical strength properties, resistance against rot fungi in laboratory tests and outdoor conditions, (compact stacks, the so-called block test in the use class 3).

The treatment at a lower level at 140°C decreases the equilibrium moisture for all the three deciduous woods by 30% and 35%, at the air relative humidity (RH) 65% and 98%, respectively. With increasing treatment intensity up to 160°C, the equilibrium moisture continues to decrease, reaching the decrease by 50-55%, which is well seen from Figures 4a, 4b and 4c. The next modification regime at 180°C decreases the equilibrium moisture still by 4 and 10%, respectively, at the relative humidity 65% and 98%. (Error bars show a standard deviation, 10 replicates were used per treatment).

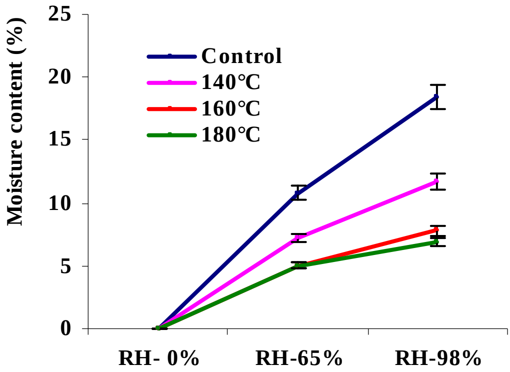



**Figure 4.a** Equilibrium moisture content of untreated and HTM **grey alder wood** under variable RH

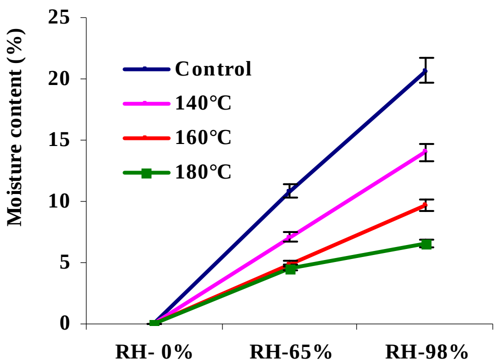



**Figure 4.b** Equilibrium moisture content of untreated and HTM **birch wood** under variable RH

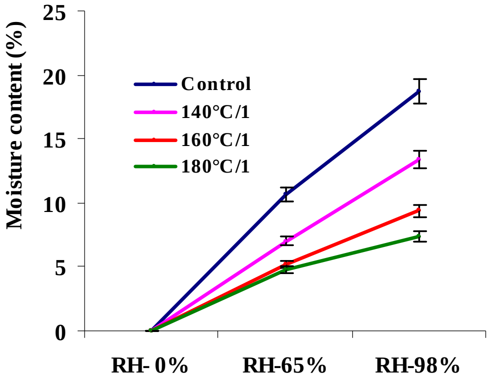



**Figure 4.c** Equilibrium moisture content of untreated and HTM **aspen wood** under variable RH

The form stability of all the HTM deciduous woods under study at different RM content of air is improved by 35-40%, already at the modification at 140°C, reaching the maximum improvement by 46 to 53% and 60 to 70%, respectively, at the RM 65 and 98%, at the treatment at 180°C. In such a treatment regime, the highest form stability is reached by birch wood, probably, owing to a higher density.

To predict the long-term anti-swelling efficiency (ASE) of HTM modified deciduous wood, several moistening-drying cycles were carried out. Figures 5a, 5b and 5c demonstrate that, in the first impregnation cycle, HTM wood shows similar form stability values, which we obtained holding the wood at the RH of air 98%. However, with each next cycle, the form stability worsened, especially dramatically for the samples modified at 140°C and 160°C. The treatment regime at 180°C practically for all deciduous woods ensured comparatively invariable form stability (decrease by 13-20%, comparing the results of the 1st and 5th cycles). (Error bars show a standard deviation, 10 replicates were used per treatment).

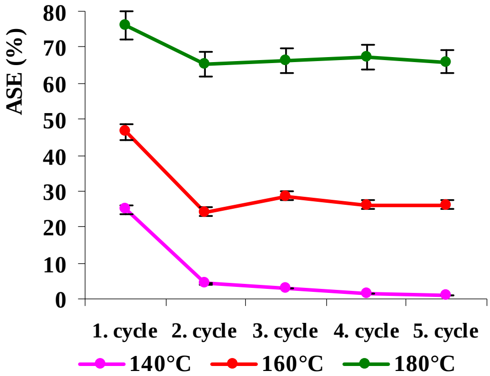



**Figure 5.a** Change of total anti-swelling efficiency (ASE) of HTM **birch** wood during 5 cycles of water saturation and oven drying

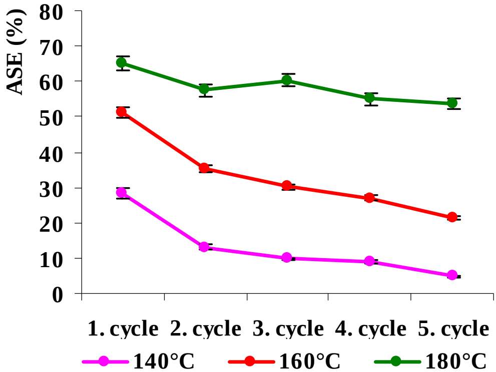



**Figure 5.b** Change of the total anti-swelling efficiency (ASE) of HTM **aspen** wood during 5 cycles of water saturation and oven drying

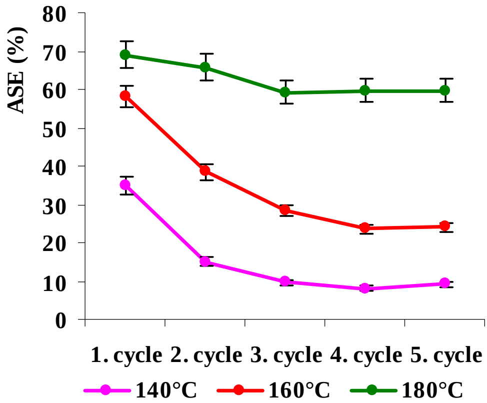



**Figure 5.c** Change of the total anti-swelling efficiency (ASE) of HTM **grey alder** wood during 5 cycles of water saturation and oven drying

The first results obtained on the effect of the HTM regime on the hygroscopic properties of deciduous wood have shown that the maximum improvement of the hygroscopic properties is achieved at the modification temperatures of 160°C and 180°C, but the stability of the hygroscopic properties is guaranteed only at the treatment at 180°C.

It is known from the literature that, with increasing thermal modification temperature, the mechanical strength of wood changes dramatically; therefore, it was important to know, to what extent our developed modification regimes will influence the mechanical properties of deciduous wood. Developing the optimal thermal modification regime/s, bending strength was chosen as one of the most essential criteria. We have advanced a demand that the decrease in the bending strength of HTM wood should not exceed 30%, comparing with the case of the initial wood. With increasing treatment temperature, bending strength changes, and there is a difference between the tree species. For birch, at the treatment at 140℃ and 160℃, and for aspen at 140℃, bending strength grows by 33 and 9% for birch and by 21% for aspen, respectively. For grey alder, bending strength for unmodified wood and that HT modified at 140℃ slightly differs. With increasing modification intensity (temperature and pressure), bending strength has decreased - at the HTM temperature 180℃, even by more than 50% for aspen and birch, but by 36% for alder, in comparison with the cases of the initial wood.

One of the indicators to characterise the wood strength against the dynamic force is impact strength. Its investigation was carried out in a dynamic regime, obtaining rupture strength - time curves shown in Figures 6a and 6b. These very well demonstrate the destruction character of each deciduous wood and the effect of the HTM regime on impact strength. The character of the rupture strength - time curves for untreated and HTM grey alder and birch wood is similar (see Figures 6a), which shows that wood, under the effect of a definite strength, is broken in a moment. With increasing modification temperature up to 180°C, the character of grey alder and birch wood breaking up remains constant, and only the work required for samples' breaking decreased by 61%.

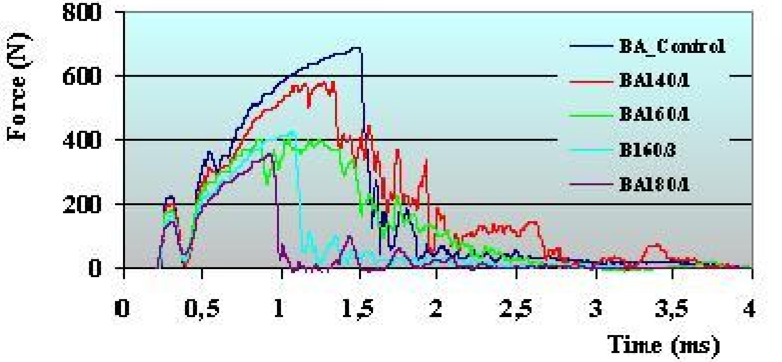



**Figure 6.a** Effect of HTM on the dynamic impact bending rupture-time curves of untreated and HTM **grey alder wood**

The strength-time curve for aspen wood is lower, although the work needed for breaking the sample is greater. It can be seen from Figure 6b that aspen is broken gradually. At the modification regimes 140°C and 160°C, impact strength decreases by 20 and 31%, respectively. The impact strength of aspen wood decreases gradually, decreasing dramatically and reaching 72% losses at the 180°C treatment. For this regime, it was not possible to take strength-time curves, because impact strength decreases dramatically. The results show that the HTM deciduous wood has become more brittle.

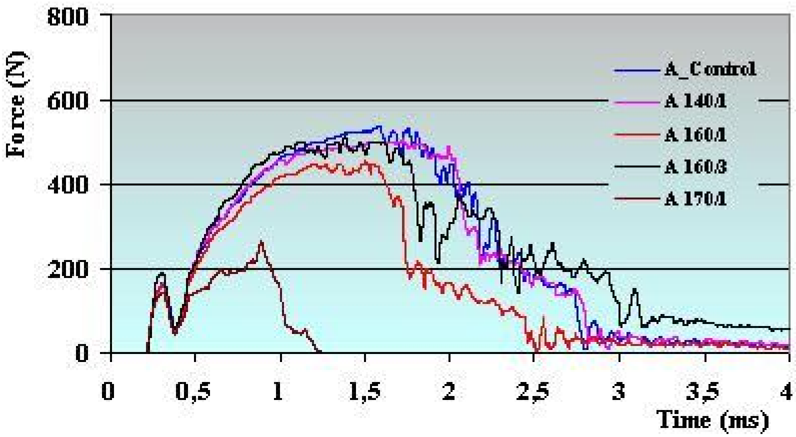



**Figure 6.b** Effect of HTM on dynamic impact bending rupture-time curves of untreated and HTM **aspen wood**

Summarising the changes in the hygroscopic and mechanical properties of HTM deciduous wood depending on the modification regime, it can be seen that the thermal treatment at 180°C improves the hygroscopic properties, but considerably worsens the mechanical properties. Because it is planned to use the HTM deciduous wood in outdoor conditions as well as in conditions of elevated humidity (use class 3), its biodurability against rot fungi is no less important.

The modification at 140°C does not improve the durability of the modified soft deciduous wood against the tested fungi. After the action of white (*Coriolus versicolor*) (see Figure 7a) and brown (*Coniophora puteana*, *Poria placenta*) (see Figures 7b and 7c) rot fungi, the mass losses are similar and even greater than for the control wood. Error bars indicated a 95% confidence interval (n = 3).

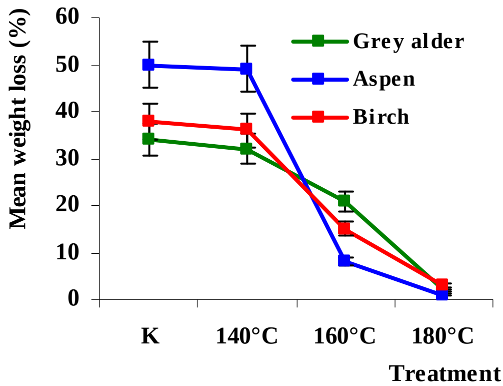



Figure 7.a Mean weight loss of untreated and HTM deciduous wood after 12 weeks inoculation to the **white rot fungus *Coriolus versicolor***

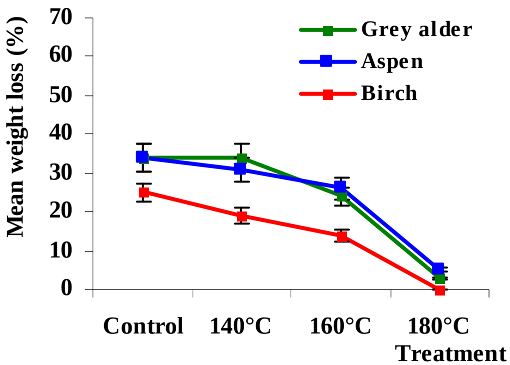



**Figure 7.b** Mean weight loss of untreated and HTM deciduous wood after 12 weeks inoculation to the **brown rot fungus *Poria placenta***

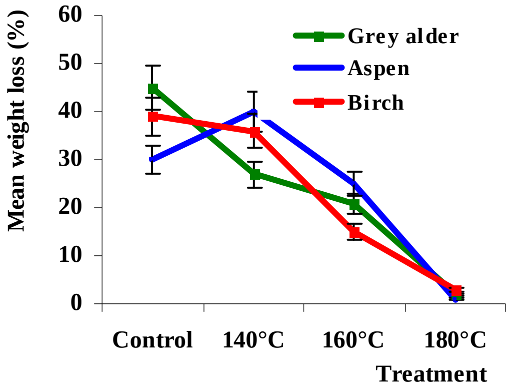



**Figure 7.c** Mean weight loss of untreated and HTM deciduous wood after 12 weeks inoculation to the **brown rot fungus** ***Coniophora puteana***

The treatment temperature 160°C improves the durability against the brown rot fungus *C. puteana* (decrease in the mass loss by 30-40%) and slightly also against the white rot fungus *C. versicolor* (decrease in the mass loss by 6-15%). The protection against the rot fungi is achieved, modifying the wood at 180°C; in this regime, deciduous wood with the durability class 1 or 2 is obtained.

Determining the biological resistance against the rot fungi in the laboratory, ideal conditions for growing the fungi are created artificially. To study the effect of the HTM regime on the long-term bio-durability of deciduous wood, the HTM alder wood samples were exposed in outdoor conditions (durability testing for use class 3: block test) for 3 years. During the study, two sample non-destructive test methods were applied. Bio-durability was determined according to the standard EN 252 [6], evaluating the visual changes of the sample by the point system. For mechanical strength determination, Grindo Sonic equipment was used, with the help of which, recording the ultrasound distribution rate in the sample, dynamic modulus of elasticity ($MOE_{dyn}$) was determined. After the first year, the $MOE_{dyn}$ value slightly decreased for beech (spacers in the stacks), untreated grey alder samples and those of grey alder, modified at 140°C, by 7, 3 and 5%, respectively. In contrast, the $MOE_{dyn}$ values for the grey alder samples modified at 160°C and 180°C practically have not changed (see Figure 8). The decrease in the $MOE_{dyn}$ value after the 2nd year of exposure in outdoor conditions was recorded for all the samples. The most dramatic decrease in the $MOE_{dyn}$ values was for beech, untreated grey alder and the alder wood modified at 140$^{o}$C (the decrease against the initial value was 40, 44 and 26%, respectively). After three years, the average $MOE_{dyn}$ value of the untreated grey alder wood decreased by 52%, in contrast to only 10% for that modified at 180°C, which shows well the efficiency of the treatment. The treatments at 140°C and 160°C do not guarantee the conformity of grey alder wood to the durablility class 3, because the samples' $MOE_{dyn}$ values within three years have changed by 40% and 27%, respectively. Error bars show a standard deviation, 20 replicates were used per treatment.

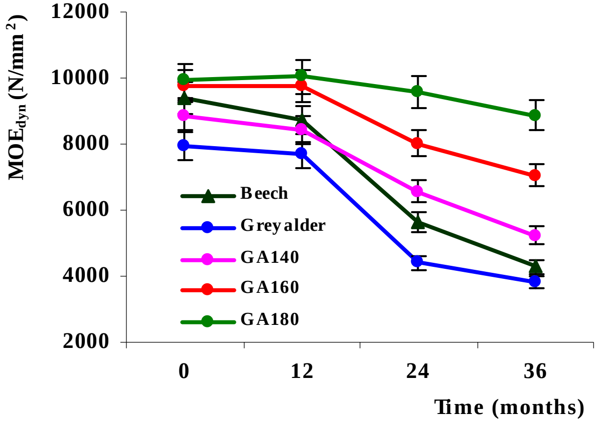



**Figure 8**. Average decrease in the dynamic modulus of elasticity for HTM **grey alder wood** until 3 years

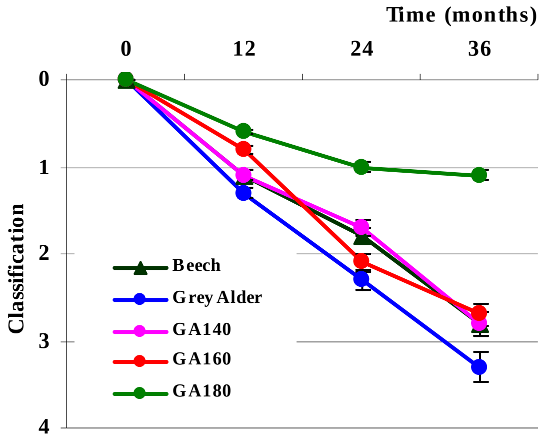



**Figure 9.** Average „pick-test" results of the untreated and HTM **grey alder** wood according to the EN 252 classification

During three years, the visual and surface state of all the treated and untreated beech and grey alder wood samples, except those thermomodified at 180°C, worsened from year to year (see Figure 9). After the first year, the samples' surfaces were partially covered with wood stain fungi, but within the next two years, fruiting bodies of the rot fungi were found on wood, which testifies the infection with different wood fungi. The results of this test also testify that the treatment at 180°C for grey wood ensures the use class 3, but the rest of the regimes are of little efficiency. The dramatic decrease in the $MOE_{dyn}$ values is explained by the invasion of rot fungi in wood, which is testified by the microscopic studies of the fungi samples taken from the block test. In wood, fruiting bodies of the white rot basidial fungi – corticia *Stereum hirsutum* and the stain *Aureobasidium pullulans* were found.

**Summarising and analysing the results on the properties of the HTM deciduous wood obtained in the first two regimes (140°C, 160°C, 180°C), we conclude that**

The hydrothermal modification at the highest treatment intensity improves the hygroscopic properties and bio-durability of deciduous wood. The HTM regime 140°C/1 h does not improve the bio-durability properties of the investigated wood; in its turn, the regime 180°C/1 h improves the hygroscopic and bio-durability properties, but considerably worsens the mechanical properties. The HTM regime 160°C/1 h is an intermediate point between the improved but insufficient biological durability and hydroscopic properties and the commensurable mechanical strength.

Therefore, **in addition, two modification regimes were developed**, which are shown in Table 2. The obtained HTM wood was subjected to identical tests. Summarising and analysing the properties of the HTM deciduous wood obtained in all experimental regimes, Table 3 was formed, from which we conclude that:

- ✓ The treatment at 160°C/3 h partially fulfils the advanced requirements, namely, partially improves the bio-durability of deciduous wood against the rot fungi and partially ensures the form stability, but the impact strength is considerably decreased. Besides, the wood of each tree species should be evaluated separately. The treatment 160/3 h ensures the requirements for deciduous wood for decreasing the bending strength and equilibrium moisture.
- ✓ The treatment 170°C/1 h practically meets all the quality requirements advanced for HTM deciduous wood, except those relative to impact strength. The treatment ensures the desired wood hydrophobicity, long-term form stability, considerably improves the durability against rot fungi, including after leaching - wood corresponding to the durability classes 1 or 2 is obtained.

Table 3

Deciduous wood properties depending on the maximum temperature of the HTM regime

| Tree species | T, °C | EMC decrease, % | | ASE improvement after, 5th cycle | CEN/TS 15083-1 durability class | Changes of the mechanical properties, % (-) decrease (+) increase | | |
|---|---|---|---|---|---|---|---|---|
| | | RH=65% | RH=98% | | | Bending | IS | HB |
| Grey alder | Control | 0 | 0 | 0 | 5 | 0 | 0 | 0 |
| | 140 | 33 | 36 | 9 | 5 – 4 | -3 | -19 | -18 |
| | 160 | 54 | 58 | 24 | 4 – 3 | -33 | -47 | -27 |
| | 160/3 | 51 | 57 | 37 | 2 – 1 | -21 | -64 | -38 |
| | 170 | 55 | 61 | 49 | 1 | -24 | -60 | -37 |
| | 180 | 54 | 63 | 60 | 1 | -36 | -61 | -47 |
| Aspen | Control | 0 | 0 | 0 | 5 | 0 | 0 | 0 |
| | 140 | 34 | 28 | 5 | 5 – 4 | +21 | -24 | -17 |
| | 160 | 51 | 50 | 21 | 4 – 3 | -14 | -31 | -25 |
| | 160/3 | 53 | 54 | 36 | 3 – 2 | -24 | -43 | -37 |
| | 170 | 57 | 58 | 43 | 2 – 1 | -23 | -60 | -36 |
| | 180 | 55 | 60 | 54 | 1 | -53 | -72 | -46 |
| Birch | Control | 0 | 0 | 0 | 5 | 0 | 0 | 0 |
| | 140 | 34 | 32 | 1 | 5 | +33 | -5 | -20 |
| | 160 | 54 | 58 | 26 | 4 – 2 | +9 | -19 | -28 |
| | 160/3 | 58 | 61 | 55 | 3 – 2 | -27 | -41 | -38 |
| | 170 | 59 | 65 | 57 | 2 – 1 | -41 | -38 | -38 |
| | 180 | 57 | 68 | 66 | 1 | -51 | -61 | -60 |
| Quality demands TMT according to CEN/TS 15679 | | ≥ 50 | ≥ 45 | ≥ 35 | ≤ 2 | ≤ 30 | ≤ 40 | ≤ 35 |

Comparing the optimised modification regimes 160°C/3 h and 170°C/ 1 h., from the viewpoint of the HTM deciduous wood production prime cost, the regime 170°C/1 h consumes by 11% less electricity (see Table 4) and decreases by 3.5% the prime cost of producing 1m$^3$ of HTM wood (see Table 5).

Table 5

Prime cost of producing 1m$^3$ of HTM deciduous wood, Ls

| Treatment | **Birch** | **Grey alder** | **Aspen** |
|---|---|---|---|
| **140** | 240 | 220 | 210 |
| **160** | 258 | 238 | 229 |
| **160/3** | 273 | 253 | 243 |
| **170** | 264 | 245 | 235 |
| **180** | 279 | 259 | 249 |

In our autoclave, using boards with the set sizes (thickness 25 mm, width 100 mm, length 1000 mm), 2 m$^2$ was treated in one treatment time. Performing recalculations, the prime cost of producing 1 m$^2$ of HTM deciduous wood depending on the species of the chosen deciduous tree and the HTM regime was obtained (see Table 6).

Table 6

Prime cost of producing 1m$^2$ of HTM deciduous wood, Ls

| **Treatment** | **Birch** | **Grey alder** | **Aspen** | **Possible market price, Ls** | | |
|---|---|---|---|---|---|---|
| | | | | **B*** | **GA*** | **A*** |
| **140** | 6.31 | 5.79 | 5.51 | - | - | - |
| **160** | 6.80 | 6.27 | 6.01 | - | - | - |
| **160/3** | **7.17** | **6.65** | **6.39** | **19.7** | **18.3** | **17.6** |
| **170** | **6.96** | **6.43** | **6.17** | **19.1** | **17.7** | **17.0** |
| **180** | 7,33 | 6,81 | 6,55 | - | - | - |

*: B - birch, A - aspen, GA - grey alder

The real market price is commonly 2.5-3 times higher than the production prime cost. At present, thermally modified birch, ash-tree and spruce wood, produced by the Finnish ThermoWood process, is available in Latvia. The market price of 1m$^2$ of the offered products is: ThermoWood Birch - 190°C – **22.44 Ls;** ThermoWood Spruce - 215°C – **14.78** Ls; ThermoWood Ash-tree - 215°C – **32.0** Ls [11]. Comparing the possible market price of 1 m$^2$ of the deciduous wood produced according to our regimes (160°C/3 h and 170°C/1 h), it can be seen that we can be compatible in price (see Table 6).

Table 4

Prime cost of producing **0.0525 m$^3$** of HTM deciduous wood depending on the treatment

| Wood species, assortment | Treatment | Price of the raw material, Ls | Electric energy consumption, kWh | Electric energy expenses, Ls | Expenses for water, Ls | Workforce, Ls | Amortisation Ls | Total, Ls |
|---|---|---|---|---|---|---|---|---|
| **Birch**, quality I dried, planed boards | 140 | 9.45 | 21 | 2.25 | 0.02 | 0.50 | 0.40 | 12.62 |
| | 160 | | 30 | 3.22 | | | | 13.59 |
| | 160/3 | | 37 | 3.97 | | | | 14.34 |
| | 170 | | 33 | 3.54 | | | | 13.91 |
| | 180 | | 40 | 4.29 | | | | 14.66 |
| **Grey alder** quality I dried, planed boards | 140 | 8.4 | 21 | 2.25 | 0.02 | 0.50 | 0.40 | 11.57 |
| | 160 | | 30 | 3.22 | | | | 12.54 |
| | 160/3 | | 37 | 3.97 | | | | 13.29 |
| | 170 | | 33 | 3.54 | | | | 12.86 |
| | 180 | | 40 | 4.29 | | | | 13.61 |
| **Aspen**, quality I dried, planed boards | 140 | 7.88 | 21 | 2.25 | 0.02 | 0.50 | 0.40 | 11.05 |
| | 160 | | 30 | 3.22 | | | | 12.02 |
| | 160/3 | | 37 | 3.97 | | | | 12.77 |
| | 170 | | 33 | 3.54 | | | | 12.34 |
| | 180 | | 40 | 4.29 | | | | 13.09 |

For calculating the prime cost of producing 1m$^3$ of HTM deciduous wood, for the basis, data were taken, obtained carrying out deciduous wood HTM in our autoclave. 0,0525 m$^3$ of the material was loaded in the autoclave per treatment, which was later recalculated to 1m$^3$.

## Changes in the morphological structure of birch wood during HTM

The changes in the morphological structure show that the widely accepted viewpoint on the destruction of the chemical components of wood is not the only argument that influences the changes of wood properties during thermal modification [1].

Wood is characterised by the heterogeneity of its chemical composition (cellulose, hemicelluloses, lignin, and extractives) and the uneven distribution of these components in wood cell walls. Besides, the wood microstructure is characterised by the diversity of the fibre elements. Hence, during the hydrothermal modification, changes in both the chemical reaction and chemical components' ratio as well as the structural changes occur in wood simultaneously.

For deciduous wood, the morphological structure is much more complicated than that for coniferous wood. Deciduous trees consist mainly of libriform fibres (36-70%), vessels (20-25%) and wood rays (6-20%) [2].

To gain insight into the behaviour of the deciduous wood structure as a result of the action of HTM, a task has been advanced to elucidate the changes in the morphological elements of birch wood depending on the treatment parameters.

Each morphological element of wood has a definite biological function. Depending on it, its elements' sizes, amounts and forms differ. Analysing the wood micrographs, we have calculated that most of our initial birch wood cross-sectional area (78.2%) is formed by libriform fibres, 1/5 part (19.9%) is occupied by vessels, and a small part by wood rays (1.9 %) (Figure 10). The average area of the libriform fibres is about 30 times smaller than the average vessel area, namely, 166 $\mu m^2$ and 5000 $\mu m^2$, respectively. At the same time, the number of the libriform fibres in wood is about 1000 – 1100 times greater than that of vessels.

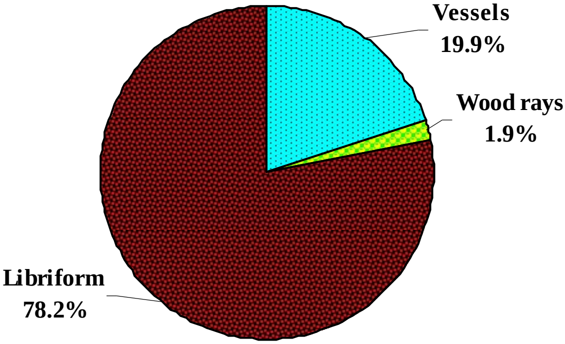



**Figure 10.** Amount of the morphological elements' area in birch wood

The mechanical functions in birch wood are played by compactly located, pronounced vertically extended narrow libriform fibres with comparatively thick walls (2-5 μm) (see Figure 11), which, as has been mentioned earlier, form the main wood bulk.

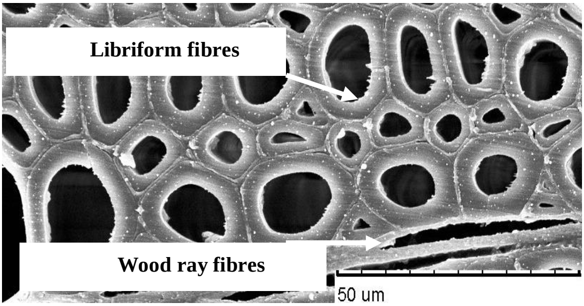



**Figure 11.** Libriform fibres and wood rays before the thermal treatment

The results of the quantitative measurements reflected in Table 7 demonstrate the effect of the thermal treatment on the change in the sizes of the birch wood libriform fibre cross-section.

The treatment at 140°C does not considerably change the sizes of the libriform fibre cross-section. However, increasing the treatment temperature every 20°C, in each next treatment, the fibre cross-section sizes (respectively, total area of fibres, wall area, wall thickness, etc.) considerably decrease (see Table 7). Their total area gradually decreases from 1% (140°C) to 20.7% (180°C), which is connected with the thermal destruction of the fibre wall forming thermally unstable components – hemicelluloses and extractives, the evaporation of bound water and other simultaneous processes. The thermal destruction of the wood substance and the diffusion of the destruction products occur across the diameter of the walls. The decrease in the fibre sizes and weight will depend on the ratio of the main components and their distribution in the wall. Hemicelluloses are known to be the most unstable wood forming components, and then their greatest amount in wood will cause the greatest changes in the fibre sizes during the thermal treatment.

With increasing thermal treatment intensity, birch wood becomes less dense. At 180°C, the intensity of the autohydrolysis process grows and the destruction products are released in the form of vapours.

Table 7

Average cross-section sizes of birch wood and their changes

| **Form of the element** | **Libriform fibres** | | | | |
|---|---|---|---|---|---|
| **Type of measurement** | **Total area, µm²** | **Wall area, µm²** | **Wall linear size, µm** | **Lumen area, µm²** | **Lumen linear size, µm** |
| **Initial sizes** | **166±63** | **103±38** | **2.58±0.55** | **62±28** | **8.65±2.28** |
| **Temperature, °C** | **Changes, %** | | | | |
| **140** | **-1.0** | **-5.2** | **-6.7** | **2.4** | **0.1** |
| **160** | **-10.5** | **-23.4** | **-21.8** | **14.0** | **6.1** |
| **180** | **-20.7** | **-37.3** | **-32.0** | **4.8** | **1.4** |

During the thermal treatment in the water vapour medium, the fibre wall is in a swelling state. In parallel with the decrease in the wall area at 180°C, the libriform fibre lumen cross-section form changes from multangular to roundish. The libriform fibre wall area decreases by 37.3 % and wall thickness by 32.0 %; they start to partly separate from each other (Figure 12). Libriform fibres form the bulk of wood, therefore, the decrease of their area, linear sizes and arrangement density are essentially affected by the mechanical and hygroscopic properties of wood.

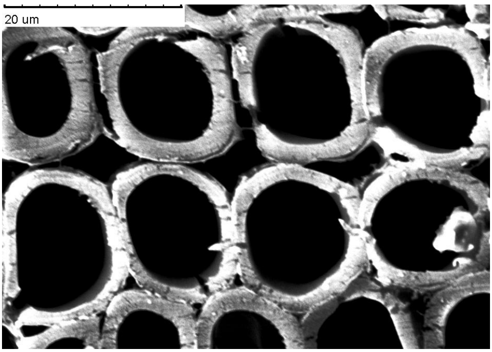



**Figure 12**. Libriform fibres after the 180°C treatment

In some places, middle lamella is cleaved from the libriform fibre wall, but has not disappeared. It is known that the middle lamella in wood consists mainly of lignin (70-90%), which is the most thermally stable wood component.

In our opinion, the wall area reflects the fibre changes most precisely from all types of measurements; it significantly correlates with the wall thickness.

The measurements of the vessel cross-section area sizes testify that those are the least changed morphological elements of birch wood as a result of the thermal treatment. These minor vessel area changes can be explained by the fact that vessel walls are twice thinner (1 µm) than the libriform fibre walls. Probably, the cell wall consists of thermally more stable lignin than it is in the libriform wall. It is known that vessels contain mainly guaiacyl structures, but libriform fibres and wood rays – mainly syringyl structures [10]. We have found that, with increasing treatment intensity, the decrease in the vessel lumen length in the radial direction is higher than in tangential one, namely, 2.9% and 2.3% against 0.5% and 0.7%, respectively.

In the experiment, we observed wood ray changes only in the wood cross-section. With increasing thermal treatment intensity, the wood ray walls decrease and begin to shrink. With increasing treatment temperature up to 180°C, also the effect of pressure and temperature on the wood ray fibres grows, and it is destructive. The lumens become deformed and form fully or partially open long and wide cracks. Crack width at 180°C increases by 250% on the average.

The results of our studies testify that, carrying out the modification of birch wood, the temperature of 180°C is too high, because the sizes of the morphological elements (libriform and wood rays) present in wood and their mutual arrangement density considerably change, the libriform fibres in separate regions move away from each other, and voids and cracks are formed between the fibres. In our opinion, such damaged arrangement of morphological elements in wood is one of most essential reasons for decreasing mechanical properties for HTM deciduous wood.

## CONCLUSIONS

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

1. With increasing HTM treatment temperature and time, the hydrophobic properties and bio-durability of deciduous wood improve. The HTM regime 140°C/1 h does not improve the bio-durability properties of wood; in its turn, the regime 180°C/1 h improves the hydrophobic and bio-durability properties, but considerably worsens the mechanical properties. The HTM regime 160°C/1 h is an intermediate point between the improved, but insufficient bio-durability and hydrophobicity, and the mechanical strength properties of wood.

2. **Two optimal hydrothermal modification regimes – 160/3 h and 170/1 h were developed**, in which deciduous wood for utilisation in the use class 3 was obtained, with the durability class 2 against fungi, by 50-65% better form stability, equilibrium moisture of wood 5-7%, as well as permitted strength losses no more than 30% for bending strength, 40% for impact strength and 40% for Brinell hardness.

3. The HTM regime at 170/1 h consumes by 11% less electric energy than the regime 160/3 h, which diminishes the prime cost of producing $1m^3$ of HTM deciduous wood.

4. The prime cost of $1m^2$ of HTM alder wood is by 7% and 32% lower than that of aspen and birch wood, respectively.

5. The changes (area, linear sizes) in the morphological elements of wood, observed on the cross-section of birch wood, significantly differ depending on the thermal treatment conditions and the type of the morphological elements.

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

7. Libriform fibres form the major part of wood, and affect to the greatest extent the total structural changes of wood after the treatment. After the treatment at 180°C, the cross-section area of the libriform fibres, the fibre wall area and the wall thickness are diminished, on the average, by 21%, 37%, and 32%, respectively.

8. After the treatment at 180°C, the integrity of wood morphological structure begins to break up. Voids and cracks are formed between morphological elements, which are one of the reasons for the decline in the mechanical properties of wood.

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