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Ecofriendly treatment to improving properties of beech wood

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Abstract
The objective of this study was to evaluate the effect of heat treatment on some conferred properties of beech wood namely durability, dimensional stability, elemental composition and color, allowing the heat treated wood to implement the exterior decoration of a building. Beech boards were heat treated by conduction at 230°C for three times to achieve three mass losses due to thermo-degradation. The results showed a significant increase in the carbon content associated with a decrease of the oxygen content, a change of color, an improvement of the durability and enhancement of the dimensional stability of wood treated according to the intensity of treatment (duration). These results clearly indicated that the chemical modifications of wood cell wall polymers are directly responsible for these properties. All these modifications of wood properties were proportional to treatment intensity which is function to temperature and duration of treatment.

Keywords: Beech wood, Heat treatment, Conduction, Elemental composition, Durability, colour, dimensional stability

1. Introduction
Wood is a natural composite material that has long been used in many applications. Despite its technical advantages (high tensile strength, high elastic modulus, low density, renewability, etc.), wood can undergo physical and chemical damage when used outdoors. Lignocellulosic polymers being responsible for most of the physical and chemical properties of wood degradation will result in an alteration of its properties. Several approaches for preservation and stabilization of wood based on the use of biocides are possible, but some of these products are subject to restrictions or prohibition. In this context, it appears new alternatives for protecting wood material such as heat treatment.

The heat treatment of wood has been reported as an attractive alternative to improve certain properties of low natural durability species, thus opening up new fields of applications that were previously restricted to tropical woods or wood treated with conventional preservatives.

During heat treatment, the wood cell wall polymers are more or less changed according to their chemical structure. The lignin is partially depolymerized, and subjected to thermal crosslinking reactions [1-3], the cellulose crystallinity is modified [4,5] and hemicelluloses are severely degraded [6,7]. All of these chemical modifications give the thermally treated wood a decrease in equilibrium moisture [8,9], better dimensional stability [10] and improved durability against rot fungi [11-12], while mechanical properties are reduced [13].

Heat treated wood is more resistant to natural weathering because some compounds of lignin degradation are less leachable compared to untreated wood. Thermal treatment improves the wood surface, particularly the roughness,

caused by the machining, which strongly decreases. In addition, it changes the natural wood color which becomes darker [14-16]. This change is often justified by the formation of colored products resulting from hemicelluloses degradation and extractives which seem to participate in the color of the heat treated wood. [17]. The formation of oxidation product such as quinones, is also given as the reason for the color change [18]. This color is uniform throughout the thermally treated wood. Heat treatments allow giving the appearance of a noble wood to a low-value species. In this context, interior and exterior architects are looking for wood materials which can be controlled in the mass shades of colors, for example to allow the partial replacement of wooden siding and roof in classified sites historical monuments, or that the material is based on the color shade of the implantation site.

The aim of this work is to apply a heat treatment to beech wood samples before carving and use them as decoration on the outside of buildings. For this purpose, beech boards were treated at 230°C under nitrogen for different times to achieve mass loss of about 5, 10 and 15%. The durability against brown rot fungi, dimensional stability, moisture content and colour parameters were measured and correlated with heat treatment intensity.

2. Materials and methods

2.1. Materials

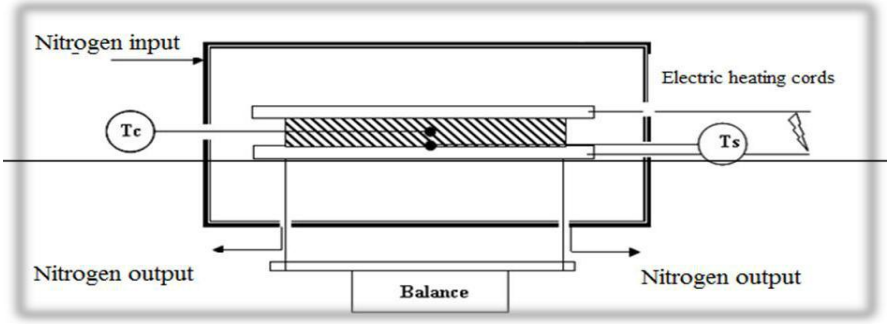
Heat treatment is performed on beech boards (*Fagus sylvatica*), dried in an oven at 103°C until stabilization of mass. The dimensions of these boards were 25 × 11 × 2.5 cm in longitudinal, tangential and radial directions respectively.

2.2. Heat treatment

Heat treatment of wood boards was performed at 230°C under nitrogen by conduction between two metallic heating plates placed on a precision balance allowing for recording the dynamic mass loss, as previously described (Figure 1) [19]. Thermally treatment is performed under different conditions of time to achieve the desired mass loss (5-10-15%). Mass of the sample was recorded automatically throughout the experiment, and mass loss (ML) due to thermo-degradation was calculated according the formula:

$$ML\ (\%) = 100 \times (m_0 - m_1)/m_0$$

Where m_0 is the initial oven-dried mass of the sample before heat treatment and m_1 is the oven-dried mass of the same sample after heat treatment.



Ts: Surface Temperature
Tc: Core Temperature

Figure 1: Experimental apparatus

2.3. Fungal durability

Wood blocks of 5 × 10 × 30 mm^3 were cut from heat treated boards for fungal durability evaluations. Petri dishes (9 cm diameter) were filled with sterile medium (20 ml) prepared from malt (40 g) and agar (20 g) in distilled

water (1L) and inoculated with a piece of mycelium of a freshly grown culture. Petri dishes were incubated at 22°C and 70% relative humidity (RH) until complete colonization of the medium surface by the mycelium. Two heat treated samples and one untreated sample were placed in each petri dish and exposed to the brown rot fungus (*Poria placenta*, *Coniophora puteana* and *Gleophylum trabeum*) for 16 weeks. Each experiment was duplicated. At the end of the test period, mycelia were removed and the blocks were dried at 103°C and weighed (m_2) to determine the weight loss (WL) due to fungal decay:

$$WL\% = 100 \times (m_{0\ or\ 1} - m_2) / m_{0\ or\ 1}$$

Were $m_{0\ or\ 1}$ are, respectively, the initial oven-dried mass of untreated or heat treated wood samples before fungal exposure and m_2 is the oven-dried mass after fungal attack.

2.4. Anti-swelling efficiency measurements

Anti-swelling efficiency (ASE) was determined by measuring the increase in volume after humidification of treated and untreated wood blocks (20×10×10 mm^3, L, R, T). The blocks (6 replicates) were immersed in distilled water in a beaker placed in a desiccator under vacuum (30 mbar for 30 min). Vacuum was then released and pressure was returned to atmospheric. A cycle of 5 water soaking was applied (changing the water every day) before determination of the water saturated volume. The blocks were then removed from water, wiped dry and measured. Volumetric swelling coefficients were calculated according to the formula:

$$S\ (\%) = ((V_2 - V_{0\ ou\ 1}) / V_{0\ ou\ 1}) \times 100$$

Where V_2 is the volume of water saturated wood and $V_{0\ or\ 1}$ is the volume of dry untreated or heat treated wood. The percentage of swelling was calculated from the wet and dry volumes of treated and untreated blocks according to the equation:

$$ASE\ (\%) = ((S_{control} - S_{heat\text{-}treated}) / S_{control}) \times 100$$

Where $S_{control}$ is the volumetric swelling coefficient of the unmodified samples and $S_{heat\text{-}treated}$ is volumetric swelling coefficient of modified samples.

2.5. Colour change

The surface color of all wood samples before and after heat treatment was measured with a BYK Gardner spectro-guide 45/0 gloss colorimeter. Percentage of reflectance was collected and converted into the CIELAB color system, where *L** describes the lightness, and *a** and *b** describe the chromatic coordinates on the red-green and blue-yellow axis, respectively. The parameters *L**, *a** and *b** were measured at three locations on each sample, and the average values were calculated. From the *L**, *a** and *b** values, the difference in the lightness (ΔL^*), chroma coordinates (Δa^* and Δb^*) and the total color change (ΔE^*) were evaluated. ΔE^* is defined as follows:

$$\Delta E^* = [(\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})]^{1/2}$$

2.6. Elemental analysis

Heat treated or untreated wood was ground to fine sawdust and sieved to obtain a particle size between 0.2 and 0.5 mm. The sawdust was dried in an oven at 103°C for 48 h and stored in closed bottle before analysis. Elemental analyses were performed on a Thermo Finnigan Flash EA1112 microanalyzer.

2.7. Equilibrium moisture content determination

Wood blocks (10×25×25 mm^3, the smallest dimension corresponding to the longitudinal direction, 6 replicates) were placed in a climatic chamber WTB Binder Type KBF 115 at 22°C and 65% relative humidity. The blocks

were kept for each RH% until stabilization of their mass. The equilibrium moisture content (EMC) was calculated according the formula:

$$EMC\ (\%) = 100 \times (m_h - m_d) / m_d$$

Where m_h is the humid mass of the sample at 65% RH% and m_d is the dry mass before experiment.

3. Results and discussion

Mass loss as a function of temperature and treatment time is shown in Figure 2. Thermo-degradation curve (black) is the average of 2 curves of mass loss (green and red).

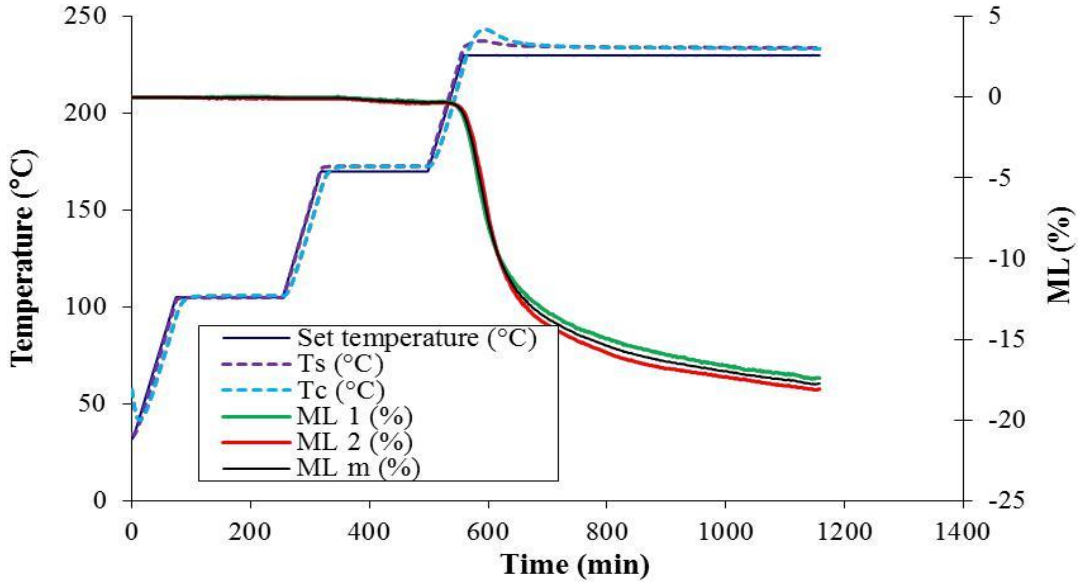


Figure 2: Thermo-degradation of beech wood treated at 230°C (1°C.min^{-1})
ML: mass loss due to thermo-gradation reactions, Ts: surface temperature, Tc: core temperature

Until 160°C, very low mass losses were observed corresponding to the vaporization of volatile extractives and of bound water retained in the cell walls. The thermo-degradation reactions begin at higher temperatures and are effective at 230°C.

The use of heat treatment apparatus designed to record a dynamic mass loss depending of time and temperature, allows to adapt treatment durations to achieve mass loss of about 5, 10 and 15% in order to check the impact of heat treatment intensity on the conferred properties. Evolution of elemental composition of beech wood according to heat treatment intensity is reported in Table 1.

Table 1: Elemental composition of beech wood as a function of treatment intensity

Treatment Time* (min)	Mass loss (%)	%C	%H	%O	O/C**
Untreated beech wood	0.0	48.8	6.0	45.2	0.69
580	5.5	51.4	6.1	42.4	0.62
610	10.9	52.9	6.0	41.0	0.58
765	15.2	54.1	5.9	40.0	0.56

** Total duration of treatment*
*** Atomic ratio*

Carbon content increases with the increase in mass loss, while oxygen content and O/C ratio decrease. The correlation between mass loss due to thermo-degradation reactions and O/C ratio is shown in Figure 3.

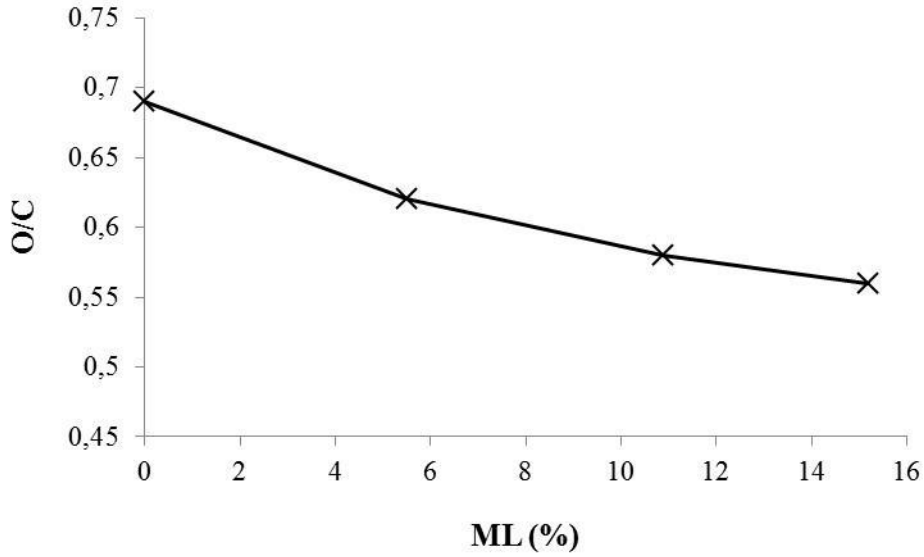


Figure 3: Correlation between mass loss (ML) due to heat treatment of beech wood at 230°C and O/C ratio

O/C ratio decreases linearly with the increase of mass loss indicating that O/C ratio could be a valuable marker of heat treatment intensity. This decrease can be mainly attributed to the thermal degradation of wood mentioned above and subsequent dehydration reactions explaining the decrease in oxygen content. Figure 4 shows the evolution of equilibrium moisture content according to treatment intensity.

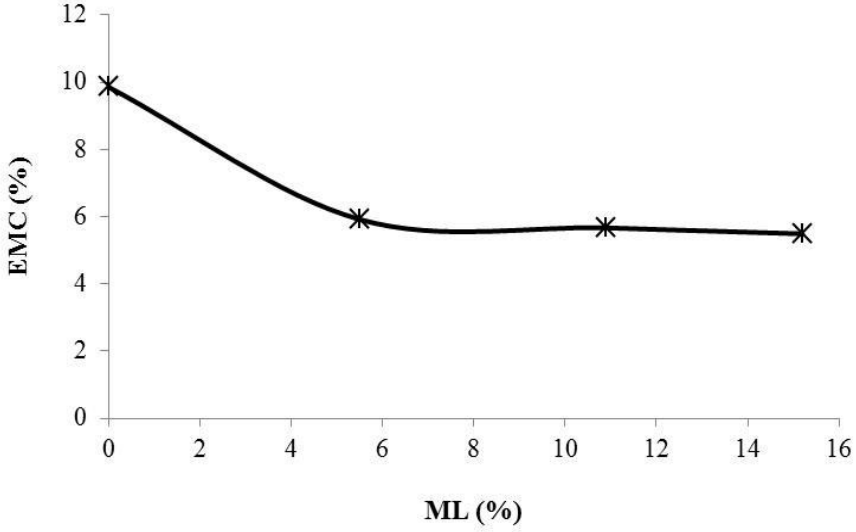


Figure 4: Relationship between mass loss (ML) due to thermal degradation of beech wood at 230°C and EMC

Heat treated beech wood loses a part of its hygroscopicity. This phenomenon is due to degradation of hemicelluloses which are the most hygroscopic components of wood cell wall. This degradation affects the hydroxyl groups of hemicelluloses involved in moisture uptake by forming H bonds with water molecules.

With the increase of mass loss, EMC was reduced. This can be explained by less moisture-accessible hydroxyl groups of thermally modified samples compared to untreated wood. However, the EMC remained stable beyond the limit value of 6% from the mass loss of 5%. The EMC limit value indicates a completion of the

decomposition of moisture-accessible hydroxyl groups by heat treatment, and therefore an extension of the treatment time does not significantly affect the EMC.

In parallel, dimensional stability (ASE) increases with the increase of mass loss (Figure 5). This can be explained by reactions occurring during heat treatment, involving mainly degradation of amorphous polysaccharides and reactions of depolymerization/recondensation of lignin. In general, a higher heat treatment intensity caused a higher ASE.

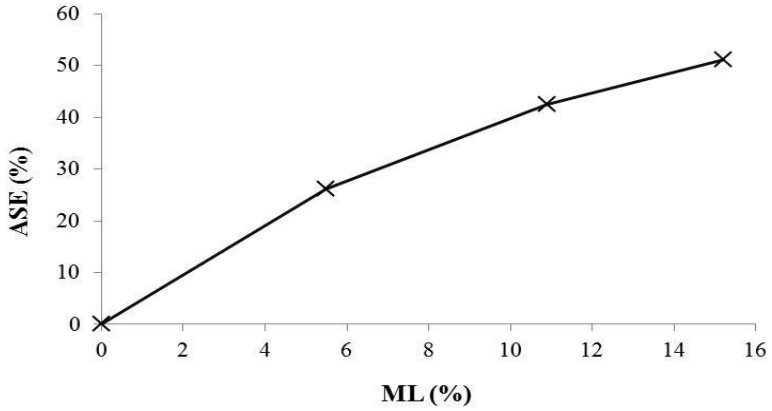


Figure 5: Evolution of ASE as a function of ML

At the same time, all these modifications, which are strongly interconnected, conferred to wood improved resistance against fungal decay (Figure 6).

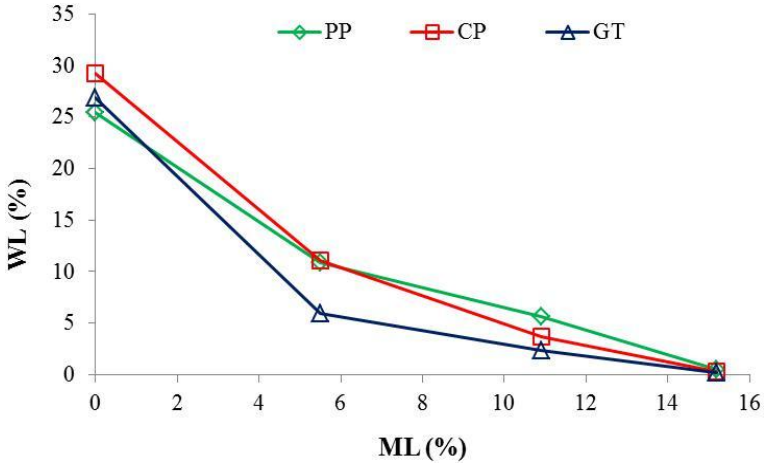


Figure 6: Correlation between mass loss due to thermo-degradation reactions (ML) and weight loss caused by *Poria placenta (PP), Coniophora puteana (CP)* and *Gloeophyllum trabeum (GT).*

Indeed, while control is severely degraded (WL above 25%), the heat treated samples present smaller weight losses. Improvement of durability is proportional to mass loss induced by heat treatment. The durability of the various samples becomes total for mass losses between 10 and 15%. These results are coherent with results from the literature showing that conferred durability of heat treated wood increases with treatment intensity.

Chemical modification of polymers of wood cell wall is the main reason for the improvement of the durability of heat treated wood [20].

Taking into account the thermal degradation reactions occurring during heat treatment of wood, involving the degradation of amorphous polysaccharides and reactions of depolymerization/recondensation of lignins [3], wood

properties are progressively modified during heat treatment leading on one hand to a decrease in oxygen content due to dehydration reactions explaining the decrease of EMC, and the other hand to increase the carbon content and dimensional stability associated with thermal crosslinking reactions. All these modifications, which are highly interconnected, conferred to wood improved durability. This improvement is the result of chemical changes caused by heat treatment which are a direct effect on EMC, ASE and elemental composition. Treatment intensity is the main parameter determining final properties of heat treated wood.

When we focused on colour parameters including *L**, *a** and *b**, we found that colour changes were drastic after heat treatment at 230°C. Figure 7 shows the changes in lightness and chromatic parameters according to treatment intensity of beech wood.

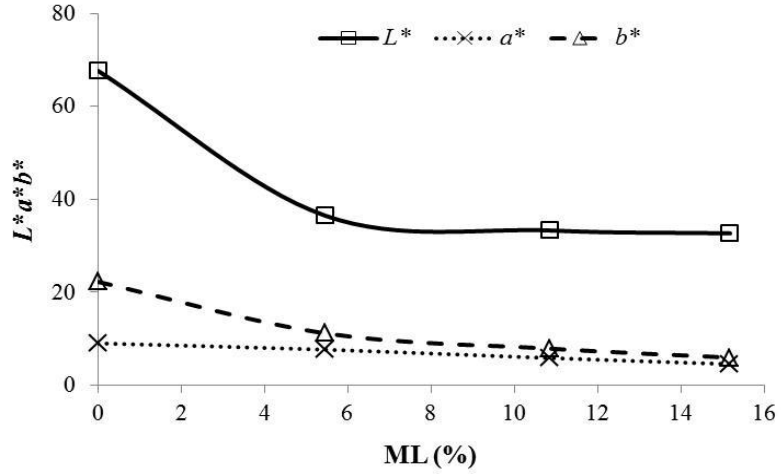


Figure 7: Effect of heat treatment of beech wood on the colour change at different ML

At low mass loss due to heat treatment of beech wood at 230°C, darkening increased. The darker tonality of heat treated wood is often attributed to the formation of coloured degradation products from hemicelluloses [21]. The greatest reduction of *L** was observed for a mass loss of 5%. At this condition, the loss of lightness was approximately 46%, reaching 36.5 units compared with 67.6 units for untreated wood. The *a** value remained stable. The *b* * value decreased slightly from 22.3 for untreated wood to 11.2 for a mass loss of 5%, and thereafter remained stable. The total colour difference (ΔE*) increased from 33% for a mass loss of 5% to 37% for a mass loss of 10% thereafter ΔE* remained almost stable (Fig. 8). Heat treatment induced extensive darkening of beech wood.

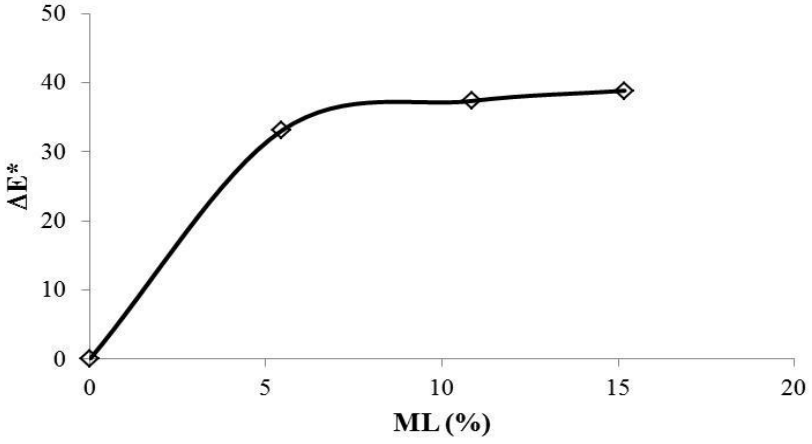


Figure 8: Effect of treatment intensity on the total colour difference (ΔE*) of beech wood

Heat treatment allows then to produce resistant wood when used for outdoors and undergoes physical and chemical weather damage. Treated wood will have a beneficial impact in industry for decorative purposes including facades, due to its durability, dimensional stability and ease of making; we were interested in its application as decoration of the front of the Academy of traditional Arts building (Figure 9). The use of this decoration opens an interesting perspective without resorting to demolition.

Conclusion

The results clearly show an increase in the durability of thermally treated wood against rot fungi. Improving the durability of the heat treated wood is strongly connected to treatment intensity and can be explained by the degradation of hemicellulose and reactions of depolymerization/recondensation of lignin. Beech wood can be considered as resistant to brown rot fungi for mass losses due to heat treatment between 10 and 15%. Oxygen content decrease progressively as the intensity of treatment increases, while carbon content increase reflecting the importance of chemical modifications of components of wood cell wall. Heat treatment has a direct effect on improving equilibrium moisture content, anti-swelling efficiency and the colour witch becomes darker. Treatment intensity is the main parameter which determines the final properties of heat treated wood.

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