Effect of combined modification processes on the physical properties of wood

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ABSTRACT

The investigated treatment in this study was a heat treatment in paraffin wax at 180°C or 200°C with 5 hours duration. Impregnation was accomplished by the cooling of the material in the paraffin after heat treatment step, from the treatment temperature to 100°C. During this cooling step, wood material was saturated by the melted paraffin wax. After taking the samples from the hot paraffin, wax was hardened in the cell lumen. In addition, samples were heat treated without impregnation (normal heat treatment) to determine the effect of additional impregnations to the effect of heat treatment. Samples of beech wood were used for the tests. Mass loss, colour change, swelling, bending strength and compression strength was investigated. Colour changed remarkably in case of both heat treatment and combined treatment. However, colour change as a result of the combined treatment was larger. Slight increase of red- and yellow hue, and large decrease of the lightness was observed in case of normal treatment. Contrarily, in case of combined treatments, red hue decreased slightly, while a significant decrease of yellow hue was observed. Mass loss was slightly higher in case of combined treatment, which result can be explained by the different heating medium. Heat transfer is more intensive in case of the paraffin wax treatment medium. Interesting result was found, that paraffin uptake was higher in case of treatment at 180°C compared to the treatment at 200°C. Bending strength decreased by 25-40% depending on the treatment. Compression strength increased by 30% as a result of normal heat treatment, while it remained unchanged in case of the combined treatment. Swelling properties were decreased as a result of heat treatment, but in case of the combined treatment swelling was remarkably lower compared to the simple heat treatment.

INTRODUCTION

The thermal treatment of wood was a research topic long ago, and the processes were permanently optimized in different countries. The first trials by Tiemann (1920) showed already, that high temperature drying increases the dimensional stability. But targeted investigations only began later (Stamm and Hansen 1937). During time, different processes have been widely used in Europe (Sandberg *et al.* 2017). The advantage of paraffin wax is its water repellent and nontoxic nature.

Waxes are natural or synthetic substances depending on their origin. Waxes are used for wood surface finishing and coating from the ancient times. Since biocides, due to EU regulations are increasingly restricted, waxes and wax emulsions are becoming one of the most important solutions for non-biocidal wood protection in outdoor applications to improve durability, dimensional stability and sorption properties. Furthermore, treatments with waxes slow the photodegradation process of wood as well (Lesar *et al.* 2011). With the impregnation method, the cell lumens can be filled with wax. An advantage of wax impregnation of wood is the improvement in wood's mechanical properties. For example, the hardness can be increased in beech wood up to 86 to 189% in the longitudinal and lateral directions, respectively. Other mechanical properties of wood, like compression, bending or impact-bending strength can be also improve as the effect of impregnation with different waxes (Scholz *et al.* 2010). Both the heat treatment and the impregnation with waxes provide some dimensional stability for wood, but only limited. The mode of action of these treatments is different. In case of heat treatment the effect is mainly the removal of hydroxyl groups through the thermal degradation of the cell wall polymers. In case of impregnation, the effect is the clogging of the cell lumens and cell wall pores by waxes, thus the occlusion of water from the cell wall. With the combination of these treatments, it is possible to improve the dimensional stability of wood, as the effects of different mode of actions are added.

EXPERIMENTAL

Modification processes

Beech (*Fagus sylvatica*) wood was heat-treated in paraffin wax. The dimensions of the treated samples (laths) were 30×50×320 mm^3 (TxRxL). Macrocrystalline paraffin wax was used as heat treatment medium. The paraffin bath ensured the deficiency of oxygen during the treatment as the samples were immersed in the paraffin. The investigated treatment was a heat treatment in paraffin wax (combined treatment) at 180°C or 200°C with 5 hours duration. Impregnation was accomplished by the cooling of the material in the paraffin after heat treatment step, from the treatment temperature to 100°C. During this cooling step, wood material was saturated by the melted paraffin wax. After taking the samples from the hot paraffin, wax was hardened in the cell lumen. Six samples (laths) with an initial moisture content of 12% were used for each schedule. The samples were placed directly in the warm paraffin bath having temperature of 80°C without preheating. At the end of the process the samples were removed from the paraffin bath at 100°C, to avoid hardening of the wax before the removal of the samples. Samples were stored after treatment under standard conditions (T=20°C, φ=65%). Untreated and air-dried laths having the same dimensions served as the control. To be able to show the additional effect of paraffin impregnation to the heat treatment, samples with the same dimensions and parameters were heat treated (HT) together in the chamber with the paraffin treated samples. The heat treatment medium in this case was atmospheric air. Thus, all samples (both HT and combined treatment) were heat treated under the same conditions at the same time, excepting treatment medium. Untreated samples were used as control. Samples of beech (*Fagus sylvatica*) were used for the tests.

Colour measurement

Colour analysis was carried out according to the CIELab colour system. The moisture content of air-dried, conditioned (T=20°C; φ=65%) samples was measured. For colour measurements, a Konica-Minolta CM – 2600d model spectrophotometer was used. Colour of the initial laths was measured before and after the heat treatment at five points on each lath.

Mechanical properties

Bending strength (MOR) and compression strength parallel to the grain was determined on normal heat treated and heat treated/impregnated samples. Untreated samples served as control. To determine the MOR, a standard three-point bending method was used, based on the standard MSZ 6786-5/1984. To determine the bending strength, a method based on the standard MSZ 6786-8/1984 was used. 20 samples were tested for both treated and untreated material.

Swelling

To determine swelling, 20×20×30 mm (radial × tangential × longitudinal) samples were cut from the heat-treated laths; there were 20 pieces from all treatment types investigated. 20 pieces of untreated samples served as the control. The samples were dried at 105°C until a constant mass and then the dimensions were measured in the radial, tangential and longitudinal directions. Thereafter, the samples were stored under water for 7 days and finally the dimensions were measured again.

Swelling was determined according to Eq. (1):

$$S_{r,t} = \frac{l_u - l_0}{l_0} \cdot 100 \ (\%) \qquad (1)$$

Where, $S_{r,t}$: swelling, radial, tangential or longitudinal (%)
l_u : dimension after wetting (mm)
l_0 : dimension in dry state (mm)

RESULTS AND DISCUSSION

Mass loss was significantly higher in case of combined treatment, which result can be explained by the different heating medium. Heat transfer is more intensive in case of the paraffin wax treatment medium. Interesting result was found, that paraffin uptake was higher in case of treatment at 180°C (278,37 kg/m^3) compared to the treatment at 200°C (144,29 kg/m^3), however, treatment duration was the same in both cases (Table 1).

Table 1: Mass loss and paraffin uptake as a result of different modification methods

Treatment	**Mass loss (%)**	**Paraffin uptake (kg/m^3)**
Combined 180°C	4,91 %	278,37
Combined 200°C	12,10 %	144,29
HT 180°C	2,41 %	-
HT 200°C	4,11 %	-

Colour changed remarkably in case of both heat treatment and combined treatment, but colour change as a result of the combined treatment was larger. However, there was a difference between normal and combined treatments (Figure 1). Slight increase of red- and yellow hue, and large decrease of the lightness was observed in case of normal treatment. Contrarily, in case of combined treatments, red hue decreased slightly, while a significant decrease of yellow hue was observed. Additionally, the decrease of lightness was more intensive compared to the normal treatment. The reason for the different colour change as a result of the two investigated treatment types treatments is the different treatment medium. The liquid treatment medium ensured a more intensive heat transfer that led to more intensive colour change. Additionally, the paraffin became dark coloured as a result of the treatment as well. This can be explained by the precipitation of the heat degradation products from wood material. The presence of this discoloured paraffin led to a darker colour of the material after heat treatment as well.

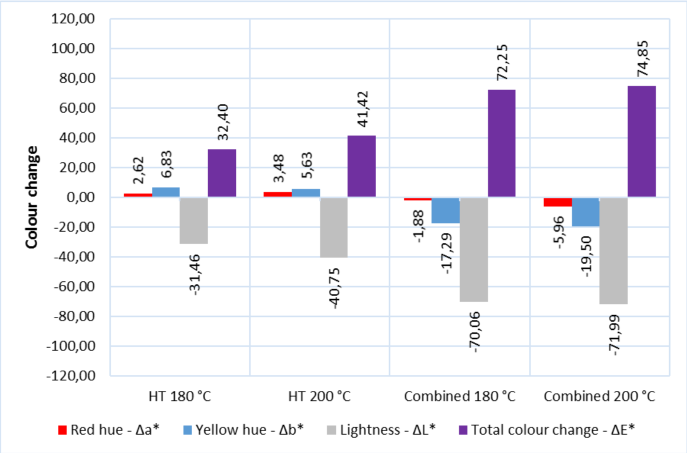

Figure 1: Change of colour properties as a result of different modification methods

Bending strength decreased in case of all investigated treatments significantly, compared to the control (Figure 2). Bending strength decreased by 25-40% depending on the treatment type. Comparing the results of normal and combined treatments at identical treatment temperatures, there was no significant difference between them. These results indicate an intensive heat degradation of the cell wall structure. However, our hypothesis was that the presence of solid paraffin in cell lumens will result in higher bending strength compared to the normal heat treated material, this was not supported by our results. The explanation for this might be in this case as well, that the more intensive heat transfer in the paraffin medium resulted in somewhat higher heat degradation that compensated the strengthening effect of the presence of paraffin in the cell lumens of wood.

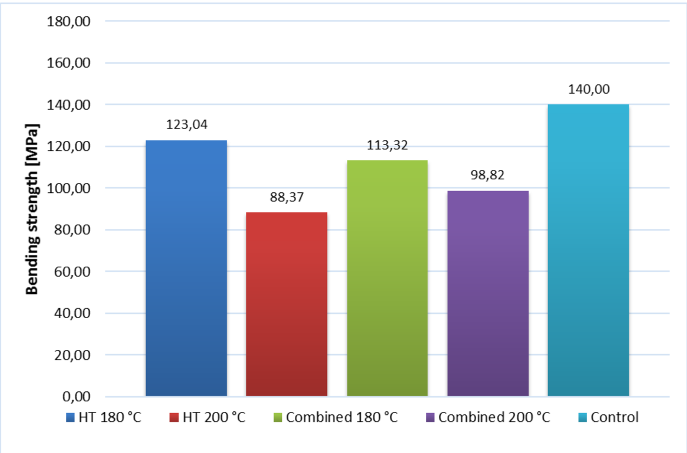

Figure 2: Change of bending strength as a result of different modification methods

Compression strength increased in case of normal treatments significantly, compared to the control, while in case of combined treatment, there was no significant difference

(Figure 3). Compression strength increased by 30% as a result of heat treatment, while it remained unchanged in case of the combined treatment. Higher treatment temperatures resulted in slightly lower compression strength values. These results support again the theory, that the heat degradation of the cell wall components is increased when using a liquid (paraffin) as heat treatment medium, because of the more intensive heat transfer. The result of this phenomena is the lower mechanical properties in case of wood material heat treated in paraffin. This result is even more interesting, if we take into account that bending strength is more depending on the properties of cellulose and hemicelluloses (tensile properties), while compression strength is more dependent on the condition of lignin. The lower compression strength values of combined treatments compared to the normal heat treated material indicates a higher degradation of lignin, using a more intensive heat transfer during the heat treatment.

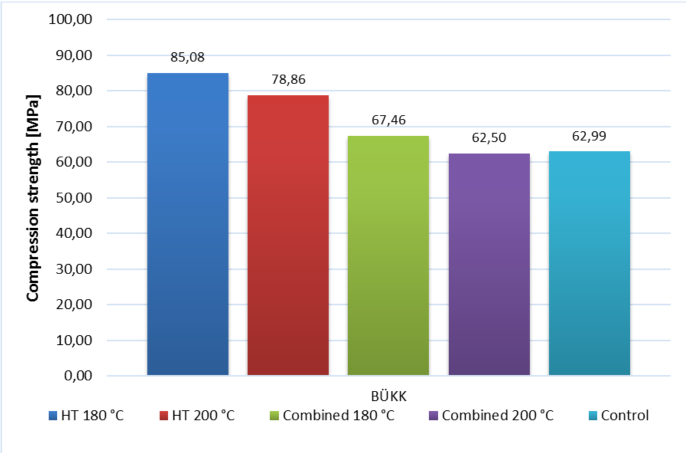

Figure 3: Change of compression strength as a result of different modification methods

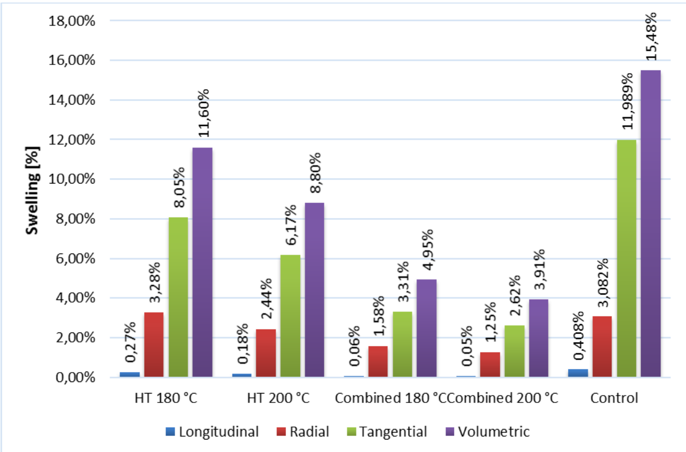

Figure 4: Swelling properties of beech wood before (control) after normal (HT) and combined heat treatments

Swelling properties were decreased as a result of all investigated treatments, but in case of the combined treatment swelling was remarkably lower compared to the normal heat treatment (Figure 4.). Dimensional stability increased in proportion with the treatment temperature, as higher treatment temperature resulted in lower swelling values in case of both combined and normal treatments. Promising result was found that combined treatments resulted in much lower swelling compared to the normal treatments. This can be explained from one hand again with the higher heat degradation because of more intensive heat transfer, resulting in higher efficiency against moisture uptake. From the other hand, this result can be explained by the occlusion of the cell lumens and the water repellent effect of paraffin wax.

CONCLUSIONS

The goal of this study was to investigate the effect of a combined wood modification process, consisting of a heat treatment step in paraffin wax, followed by an impregnation step with the paraffin by the cooling of the samples in the paraffin. Results showed significant differences compared to a normal heat treatment process regarding the mass loss, colour, mechanical and swelling properties as well. Mass loss and darkening of the material increased as a result of combined treatment compared to the normal process. No significant difference could be found in case of bending strength between the effect of different treatment processes, while there was a decrease in case of combined process when using a combined treatment. These results are explained by the more intensive heat transfer in case of a combined treatment compared to a normal process. However, dimensional stability increased remarkably in case of combined treatment, because of the favourable effect of paraffin impregnation additionally to the effect of heat treatment.

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