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EFFECT OF THERMAL TREATMENT ON COLOUR AND HYGROSCOPIC PROPERTIES OF POPLAR WOOD

Vasiliki Kamperidou, Ph.D. Candidate
Ioannis Barboutis, Associate Professor
Vasileios Vasileiou, Professor

Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment, Laboratory of Wood Products and Furniture Technology, Thessaloniki

Greece

vkamperi@for.auth.gr

ABSTRACT

Thermal modification of wood is one of the most popular and eco-friendly ways to enhance some of its properties, like its hygroscopic behavior and its resistance against fungi and microorganisms attacks, as well. At the same time, though, intensifying the treatment final temperature and duration, some of the mechanical properties of wood seem to degrade and the colour of wood tends to darken, because of the modification of the chemical composition of wood mass. The present research was carried out with poplar wood (*Populous* sp.) of Greek origin, in order to investigate its hygroscopic behavior after its thermal treatment at the temperature of 200 ^{o}C, for five different durations of 2, 4, 6, 8 and 10 hours in a drying oven in the presence of air. The colour change of the treated specimens was examined taking measurements before and after each treatment, in order to draw some conclusions of great significance about the rhythm of chemical degradation of wood owing to heat treatment, the potential decrease of its mechanical strength and also, the change of the appearance and aesthetics of the treated material.

Key words: colour, hygroscopic properties, modification, poplar, thermal treatment

1. INTRODUCTION

Wood is characterized by many valuable material properties, such as a good strength to weight ratio, aesthetic appearance, acoustic and thermal properties etc. that enable its use in a wide range of applications. It is well known, though, that this versatile material demonstrates some drawbacks, most of which are associated with its hygroscopic properties in combination with anisotropic swelling and shrinkage due to changes in moisture content (MC). Except the dimensional instability, there are also some other moisture - related problems, such as the susceptibility to microorganism (fungi, bacteria and insects) attacks, which are dependent on the presence of water for their survival (Johansson, 2005).

Many studies have been done so far, in order to enhance some of these problematic wood properties. Aiming at this quality improvement of wood, many different "wood modification methods" have been proposed. "Heat treatment" is the oldest, easiest, cheapest and most eco-friendly wood modification method among them (Ates *et al*., 2010) and has been developed in Europe during the last decade using mainly low natural durability wood species (Mburu *et al*., 2008). Based on previous research on this field, the whole procedure of thermal modification creates a physically, chemically and mechanically "new" material, as it is regarded by many scientists, characterized by improved dimensional stability, increased biological resistance against fungi and microorganisms attacks, lower Equilibrium Moisture Content (EMC) and density, increased wettability, reduced electrical resistance, reduced emissions of volatile organic compounds, improved resistance to natural weathering, enhancement of colour uniformity and stability (Awoyemi *et al*., 2009).

During thermal treatment, the colour of the specimens tends to darken, a phenomenon caused mainly by the considerable changes in the chemical composition of wood, such as the degradation of the amorphous carbohydrates. Hemicelluloses start to decompose first among the wood polymers, due to the low molecular weight that makes them more reactive. Additionally, lignin softens, cellulose and hydrophilic groups modify. As a result of this process, treated wood at high temperatures loses its reabsorbing water capacity, acquiring a more hydrophobic behaviour compared to untreated wood. Unfortunately, thermal treatment begets as well a deterioration of some of the mechanical properties, such as bending strength, impact bending strength, tensile strength etc. and increase of brittleness of the treated wood. Therefore, it is extremely crucial to choose carefully the thermal treatment conditions and intensity, referring specifically to maximum temperature, treatment duration, temperature rate, boards' dimensions, kiln atmosphere etc., in order to achieve the enhancement of some properties, limiting the mechanical strength loss of the material.

There is a high interest in the research of several thermally modified wood species and extensive research has been done so far evaluating the influence of thermal treatment on the colour and the hygroscopic properties of wood. Some of the most current and significant researches referring to colour change and hygroscopicity of thermally treated wood are summarised hereupon. Momohara *et al***.** (2003) studied the effect of high-temperature treatment of air-dried Japanese cedar wood, on colour and durability against the brown-rot fungus, Fomitopsis palustris and the termite Coptotermes formosanus. During the treatment, the wood was exposed to a saturated vapour at 105 °C to 150 °C, for 6 to 72 hours. Thompson *et al.* (2004), applied on red alder veneer thermal modification at 30, 50, 70 and 90 °C, for 8, 24, 36, 48 and 72 hours and recorded the variation in colour of red alder wood and the optimal thermal treatment (temperature and time) that can impart the tan colour to red alder wood. Furthermore, Tjeerdsma and Militz (2005) examined the hygroscopicity and the chemical changes in hydrothermal treated and dry heat-treated beech and Scots pine wood, using FT-IR analysis. Zivkovic *et al.* (2008) determined the dimensional stability (hygroscopic range and equilibrium moisture content, shrinking) of heat treated wood floorings, constructed with ash and beech wood. The treatment was applied at 190 ^{o} and 210 °C in water vapour atmosphere. Olek and Bonarski (2008) estimated the hygroscopic behaviour, the microfibril angle (MFA) and the texture changes of thermally modified poplar and beech wood. Additionally, Johansson (2008) studied the strength properties, the hydroscopic properties (EMC, wettability), density loss and colour response of birch, spruce and pine wood, heat treated using ThermoWood method and studied also, the possibility of using colour for predicting mechanical strength. Gunduz et al (2009) assessed the effect of thermal treatment (160, 180 and 200 ^{o}C, for 3, 5 and 7 hours) on the mechanical properties of wild Pear wood and also, the changes in physical properties, such as density, EMC, swelling and colour change. Tuong and Li (2010) checked the effect of heat treatment, applied under nitrogen at 210, 215, 220 and 230 ^{o}C, for 2, 4 and 6 hours, on the colour and dimensional stability of Acacia hybrid wood. Oliveira *et al.* (2010) estimated the wettability, shrinkage and colour changes of Araucaria angustifolia wood, after its heat treatment at 120, 140, 160, 180 and 200 °C, for 8 hours. In an experiment of Metsa and Kortelainen (2010), Scots pine and Norway spruce timber have been treated at 180 and 230 ^{o}C for 2-3 hours, under air pressure steam and the following properties were tested: change in colour, biological durability, swelling, EMC and changes of properties after 6 months of weathering.

The aim of the current research is to determine the influence of thermal treatment of poplar wood at 200 °C for 2, 4, 6, 8 and 10 hours, on its dimensional stability (absorption and swelling), EMC and the colour of wood surface. Thus, the study will facilitate a considerable knowledge of the rhythm of chemical degradation of wood owing to heat treatment, the potential change of the hygroscopic behavior of wood and also, the gradual change of the appearance of the treated material.

2. MATERIAL AND METHODS

Experiment was carried out with poplar (*Populous* sp.) wood, of Greek origin and naturally desiccated for one and a half years. Poplar wood was obtained from a local wood industry of Thessaloniki. The boards were cut parallel to grain and the dimensions of the boards, intended for thermal treatment, were of 25mm thickness x 50mm width x 120mm length. Prior treatment, the boards were placed into a conditioned room at 20 °C temperature and 65 % relative humidity and were allowed there to reach a nominal equilibrium moisture content (EMC) of 10.32 %, a limited enough moisture content that contributes to the protection of wood from stress generation and its resultant splitting and distortion. The mean density (oven-dry mass/volume measured at 10.32 % moisture content) of the poplar wood before the treatment was measured as 0.395 g/cm^3.

For the thermal treatment of the boards, a laboratory heating unit of 80 cm x 50 cm x 60 cm dimensions, with two different thermometers was used, a conventional zinc one incorporated in the unit and also, a thermometer of digital indication with temperature sensor inside the drying oven, and therefore, the unit was capable of controlling the temperature within a range of ±1 °C. The temperature applied during the thermal treatment was constantly 200 °C, while the treatment was implemented under atmospheric pressure, in the presence of air. The boards placed in the kiln, were of 10.32 % moisture content, as mentioned before, and the interior of the kiln had already reached the chosen temperature of 200 °C. Five different time durations of 2, 4, 6, 8 and 10 hours were determined to be used for the thermal treatment of the boards and six boards were used for each treatment.

At the end of the treatment, samples were cooled and stored in desiccators before weighing. The weight loss (WL) after heat treatment was estimated according to the following equation (Eq.1):

$$WL\ \% = (m_o - m_{ht}) / m_o * 100 \qquad (1)$$

where m_o is the air-dried weight of the specimen before thermal treatment and m_{ht} is the weight of the specimen measured directly after the thermal treatment. Weight measurements of the specimens were also made after 7, 14 and 21 days after thermal treatment, in order to detect the rhythm of reconditioning progress and the gradual increase of specimen weight during the conditioning process at 20 ± 2 °C temperature and 60 ± 5 % relative humidity. Afterwards, EMC and density of the specimens were measured and the boards were cut in final cross section dimensions for the measurement of hygroscopic properties and colour, according to the respective standards. For each test 10 specimens were prepared.

The swelling (in radial and tangential direction) and the absorption percentage measurements were conducted after the immersion of the samples in water of 20 ± 3 °C temperature, for 1 hour, 3 hours, 6 hours, 1 day and 3 days.

Surface colour of the specimens was measured using a Minolta Colorimenter, in order to evaluate the color change owing to heat modification. The three colour coordinates, L^*, a^*, and b^* of the CIELAB system were recorded before and after each thermal treatment and these parameters were used to calculate the total colour change (ΔE) and the Saturation index (ΔC*).

The equations used for the determination of these parameters are the following (Eq. 2,3,4,5,6,7):

$$\Delta L^* = L^*_{ht} - L^*_{ut} \quad (2)$$
$$\Delta a^* = a^*_{ht} - a^*_{ut} \quad (3)$$
$$\Delta b^* = b^*_{ht} - b^*_{ut} \quad (4)$$
$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (5)$$
$$C^* = (a^{*2} + b^{*2})^{1/2} \quad (6)$$
$$\Delta C^* = C^*_{ht} - C^*_{ut} \quad (7)$$

where L^*_{ht}, a^*_{ht}, b^*_{ht}, and C^*_{ht} refer to the corresponding values of heat treated specimens, while L^*_{ut}, a^*_{ut}, b^*_{ut}, and C^*_{ut} correspond to the values of untreated specimens (control).

3. RESULTS AND DISCUSSION

According to the results, the weight loss of the specimens, measured directly after each thermal treatment was proved to increase relatively with the intensity of the treatment (duration), which means that the treatment causes not only the release of the whole moisture content of the specimen, but also, the loss of wood mass, including the volatile extractives and a part of chemical constituents, such as hemicelluloses (Table 1). During the conditioning period the specimens tend to regain moisture from the atmosphere of the control climate room and this gradual progress of conditioning process is depicted in Table 1.

Table 1. Weight loss (%) caused by thermal treatment and Weight increase during conditioning period

Duration of treatment at 200°C	Mean Weight Loss directly after thermal treatment %	Mean Weight increase in 7 day %	Mean Weight increase in 14 days %	Mean Weight increase in 21 days %
2h	13.48	3.82	4.36	4.51
4h	16.77	3.92	4.32	4.44
6h	19.12	3.98	4.37	4.50
8h	21.77	4.25	4.63	4.75
10h	23.40	4.44	4.84	4.96

As it is obvious, during the conditioning period, the specimens treated at mild conditions, displayed lower weight increase owing to moisture absorption, compared to the specimens that were exposed to more intense treatment, whereas the differences were not statistically significant. In an attempt to explain and interpret this research finding, our attention is caught in the fact that the percentage of weight increase of the specimens from 7 to 21 days is quite different for the specimens of each treatment. Specifically, the mild treated specimens absorb, indeed, a low amount of moisture in 7 days, but they seem to continue moisture absorbing in a quite high rate, till 21 days, whereas the specimens intense treated, behaved differently. They demonstrated a high moisture absorbance in 7 days and subsequently they recorded a higher decrease in the moisture absorbance rate till 21 days. The mean weight loss (%) and the mean mass loss (%) of the specimens of each treatment were measured directly after the thermal treatments and are presented in Figure 1.

Table 2. Mean values of equilibrium moisture content (EMC) and density of the control and treated specimens

Duration of Treatment	EMC %	Density
0h	9.39	0.395
2h	4.45	0.368
4h	4.30	0.346
6h	4.27	0.334
8h	4.25	0.311
10h	4.24	0.307

The equilibrium moisture content of all heat-treated samples decreased in relation to the initial untreated wood, even for the less intensive treatment of 2 hours at 200 ^{o}C. More specifically, the EMC of poplar wood specimens was 9.39 %, while after the thermal treatment and a conditioning period of four weeks the EMC of 2 hours treated specimens was found to be 4.45 %, 4.3 % for 4 hours treated specimens, 4.27 % for 6 hours treatment, 4.25 % for 8 hours treatment and 4.24 % for 10 hours treated specimens. This EMC reduction clearly suggests that thermal treatment affects in great extent the dimensional stability and water absorbing capacity of wood (Table 2).

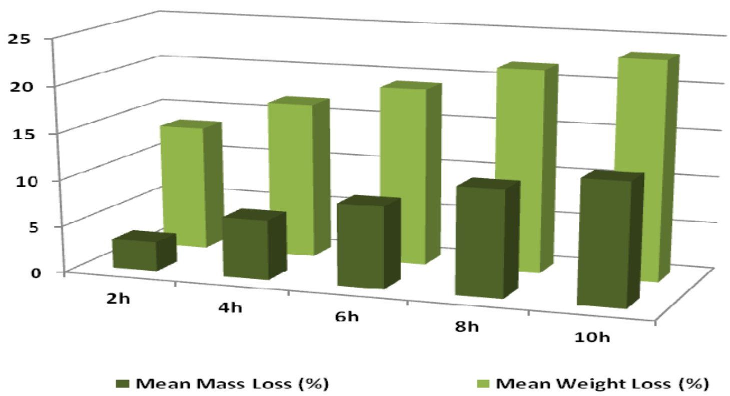

Figure 1. Configuration of the mean Weight Loss (%) and the mean Mass Loss (%) of the specimens, measured directly after the thermal treatments. The value of Weight Loss is equal with the sum of Mass loss and the Moisture Content of each specimen

Referring to hygroscopic properties, all the thermal treatments used in the specific experiment seem to enhance the hygroscopic behavior of the poplar wood specimens (Figure 2). Swelling in tangential direction proved to be quite higher than the swelling in radial direction. Neverthelless, thermal treatment proved to enhance more efficient the tangential swelling, than radial swelling. As the duration of thermal treatment was intensifying, tangential swelling tend to decrease and this tendency is similar in the case of swelling in radial direction, with the difference that when the treatment duration exceeded the six hours, the swelling values slightly increased, of course without approaching the swelling levels of the control specimens.

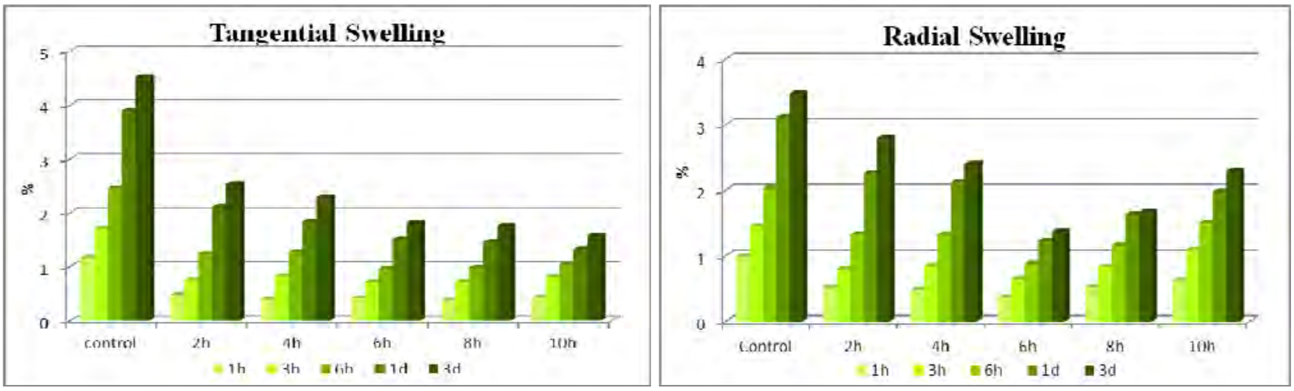

Figure 2. Percentage of tangential (left) and radial (right) swelling of the specimens, measured after an immersion of 1, 3, 6 hours and 1, 3 days in water

The absorption of the treated specimens demonstrated a satisfing enhancement, compared to the corresponding value of the control specimens (Figure 3). Increasing the treatment duration was proved to be beneficial to the limitation of absorption, while exceeding the six hours of treatment absorption percentage values record a slight increase. This fact could be explained by the potential permanent changes that usually occurr during thermal treatments of long durations like 8 and 10 hours, mainly in the chemical composition of the material and the physical properties, referring to mass loss, density loss, the thermal degradation of the polysaccharides and lignin etc. Therefore, in a case of thermal treaatment like that, being used in this study (dimensions, temperature etc.), the duration should not exceed six hours, especially when the hygroscopic properties of the material are of great importance.

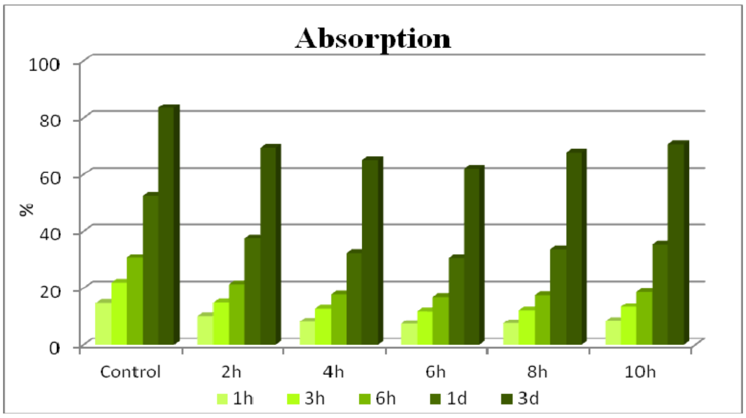

Figure 3. Percentage of absorption of the specimens, measured after an immersion of 1,3,6 hours and 1,3 days in water

In order to estimate the colour change of the specimens caused by thermal treatment of its mass, the three colour coordinates, L^*, a^*, and b^* of the CIELAB system were recorded before and after each treatment. According to the results, the quite high L* parameter of the untreated poplar wood, tends to decrease as the duration and hence, the intensity of the treatment increases. That decrease represents the loss of "lightness" and therefore, the darkening of wood surface (Figure 4). The a* parameter which positions the colour in a scale of green to red, appears to be of quite low value in untreated specimens, while it increases abruptly for heat treated specimens and subsequently, tends to decrease slowly, with further increasing of treatment duration. Finally, b* parameter, which depicts the colour coordination in a scale of yellow to blue, demonstrates a similar progress with that of a* parameter, differing only in the fact that the b* parameter value of the 10 hours treatment was measured to be lower than the corresponding value of the control specimens. All these remarks about the colour parameters of poplar wood samples, apply and refer to all of the three directions of each specimen (tangential, radial, longitudinal) (Table 3).

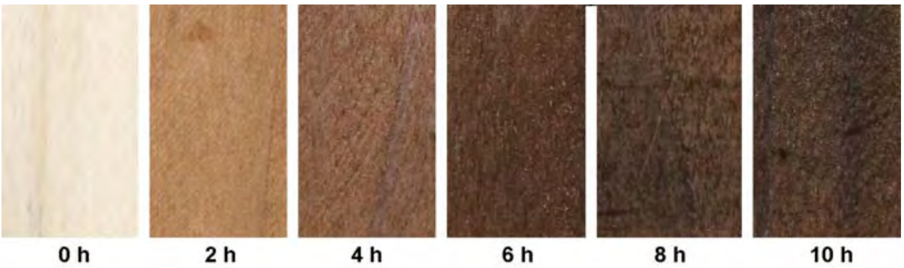

Figure 4. Configuration of the gradual colour change of the poplar wood specimens, from the untreated poplar wood (0h) to 10 hours thermal treated wood

These colour coordinates, L*, a* and b* were used to calculate the total colour change (ΔE) and the Saturation index (ΔC*) (Table 3). A low value of ΔE amounts to a low colour change for the treated sample, compared to control specimen.

According to Table 3, ΔE value of the specimens seem to increase, while ΔC* value seem to decrease, as the duration of the thermal treatment increases, which means that the colour change (darkening) of wood surface is proportional to the thermal treatment intensity.

The phenomenon of darkening of thermal treated wood could attach additional aesthetic value to the material and expand the range of applications being used. Generally, it can be mentioned that nowadays the consumers seem to favour wood species of darker colours, due to their resemblance to tropical species of high mechanical properties and durability. So, the method of heat treatment could be used, in order to modify a variety of wood species of low natural durability and dimensional stability, to a “new” more stable and durable material with enhanced properties and more uniform desirable colour and wood surface appearance.

Table 3. The CIE Lab system parameters measured for the determination of Total Colour Difference (ΔE) and Saturation index (ΔC*) for each treatment and each direction (tangential, radial, longitudinal) of the specimens

Duration of the treatment	Direction	L*	a*	b*	Total Colour Difference (ΔE)	Saturation (ΔC*)
0h	**Tang.**	87.23	1.24	15.46	-	-
2h		54.40	8.00	22.89	34.38	8.73
4h		43.46	7.92	19.04	44.47	5.10
6h		38.90	7.13	16.02	48.71	2.01
8h		37.82	6.98	14.85	49.77	0.88
10h		32.43	5.90	10.94	55.19	-3.10
0h	**Rad.**	87.20	1.14	15.65	-	-
2h		54.57	8.05	23.74	34.35	9.37
4h		39.40	7.61	16.81	47.94	2.75
6h		39.69	7.44	16.37	48.30	2.27
8h		34.49	6.44	12.87	53.08	-1.32
10h		32.73	5.98	11.49	54.85	-2.76
0h	**Longit.**	75.85	3.82	19.19	-	-
2h		46.36	6.67	19.92	29.65	1.43
4h		30.79	5.96	14.14	44.43	-4.4
6h		28.15	5.41	12.87	45.39	-5.62
8h		31.79	5.94	14.11	48.15	-4.7
10h		19.51	4.00	9.23	57.22	-9.52

4. CONCLUSIONS

Current research has facilitated a considerable knowledge of the colour change and the hygroscopic properties of poplar wood, thermally treated at 200 ^{o}C for 2-10 hours in the presence of air. According to the results, as the intensity of the treatment increases, the weight loss of the specimens, owing to heat treatment, seems to increase, while their EMC decreases. The EMC and density decrease abruptly for the milder treatments, while as the duration increases this decrease tends to be slower and more gradual, which indicates that treatment duration of 2 hours seem to be quite enough in order to improve the water reabsorbing capacity and hygroscopic properties of poplar wood. Indeed, thermal treatment causes swelling and absorption percentage decrease of the specimens in great extent, but also it was proved that in a case of treatment like this (dimensions, temperature etc.), the duration should not exceed six hours, because the radial swelling and the absorption percentage value start to increase slightly after the six hours. Additionally, the colour coordinates L*, a* and b* of the thermal treated specimens changed and these changes depicted the tendency of wood surface to darken, proportionally to treatment intensity, approximating more desirable colour tones of high aesthetics and colour uniformity for the final material. Consequently, as heat treatment technology of wood evolves and offers the opportunity to enhance some of its most crucial for the field of construction properties, like the colour and the hygroscopic properties, a fast-grown species of low quality, like poplar wood is able to be used in a wide variety of applications, competing other more valuable species.

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