

# Effect of thermal treatment on some properties of lime wood

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**Abstract** Thermal treatment of lime wood was performed in a drying oven at two temperature levels (180 and 200 °C) and for four durations (1, 2, 3 and 4 h). Mass loss, color change, swelling and hygroscopicity were investigated. The dimensional stabilization reached up to 66.4 % and the hygroscopicity reduction up to 33 %, both maximum values being attained at 200 °C/4 h, associated with a mass loss of 9.3 %. The results will be realized in the manufacturing of solid wood panels made of heat-treated lime wood lamellas for outdoor uses.

## 1 Introduction

Thermal treatment is known as the most environment-friendly method of dimensional stabilization which can be applied to wood. It is able to make wood suitable for exterior uses by reducing its hygroscopicity. This is possible due to the modifications which occur in the chemical composition of wood under the effect of high temperatures (above 160 °C). The treating temperature must be chosen so as to produce those modifications of the chemical structure which favor only the incipient decomposition of the hemicelluloses, but do not affect the cellulose structure. This way, wood becomes more stable to moisture, without losing its mechanical strengths.

Over time, numerous experimental researches regarding the effect of high temperatures upon wood properties of different wood species have been performed (with spruce: Bekhta and Niemz 2003; Borrega and Kärenlampi 2010;

pine: Esteves et al. 2011; oak: Korkut et al. 2010; Miklecic et al. 2012—to name only a few of the numerous publications in this field).

Lime (*Tilia cordata* Mill.) is a soft, diffuse-porous, broad-leaved species with low density (oven-dry density around 390 kg/m<sup>3</sup>), with rather limited use: solid wood is used for pencils, icons, carving blocks, and lime chips are used for wood-based composites. Its natural color is light and quite dull, with no specific grain. However, when being exposed to high temperature, the color darkens, thus increasing its aesthetic value and making lime wood interesting for furniture as well.

Preliminary studies regarding the structure, chemical composition and properties of lime wood show great potential to increase its aesthetic value, durability and dimensional stability through heat treatment. Until now, only Barboutis et al. (2011) have researched this potential by heat-treating lime samples at 180, 200 and 220 °C for 15 and 30 min. The general conclusion of this study was that only the heat treatment with longer duration (30 min) caused significant dimensional stabilization. The most severe heat treatment applied (220 °C/30 min) led to a mass loss of 8.7 %, a swelling decrease by 57 % and a decrease of bending strength, compression strength and hardness by 22, 9 and 15 %, respectively.

The present research focused on employing similar temperatures (180 and 200 °C) but longer durations (1–4 h) in order to obtain better results in terms of color change, dimensional stabilization and hygroscopicity reduction without affecting so much the mechanical strengths.

## 2 Materials and methods

The wooden material used within the experiments consisted of eighteen 620 × 85 × 30 mm<sup>3</sup> pith-free lime

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boards, cut from three 40 years old trees ( $\Phi = 17\text{--}19$  cm at 1.3 m above ground), originating from the same forest area: Stroesti–Arges, Romania ( $45^{\circ}8'0''$  North,  $24^{\circ}47'0''$  East). The average oven-dry density of the tested material was  $428 \pm \text{kg/m}^3$ .

The boards were first air-dried to 12 % moisture content and then conditioned for 2 weeks at  $20^{\circ}\text{C}$  and 65 % RH in a FEUTRON KPK200 climate chamber. Hereinafter, one-hundred-and-eighty  $20 \times 20 \times 30$  mm<sup>3</sup> strictly radial-tangential samples were cut for the determination of color, swelling coefficients and mass loss, and ninety  $20 \times 20 \times 40$  mm<sup>3</sup> samples were cut for the hygroscopicity test.

Heat treatment was carried out in a BINDER drying oven at two different temperatures: 180 and  $200^{\circ}\text{C}$ , combined with four treatment durations: 1, 2, 3 and 4 h. For each heat treatment condition applied, as well as for the controls (oven-dry untreated wood), a set of ten samples was employed. After each hour of treatment, a set of samples was evacuated from the oven and cooled down in a desiccator.

The mass and color for all three dimensions of each sample were measured before and after the heat-treatment. A KERN electronic scale with 0.001 g accuracy, a MUS-TEK 1248UB scanner with a resolution of  $600 \times 1,200$  dpi, and an electronic sliding gauge with 0.01 mm precision were used for this purpose.

The mass loss ( $ML$ ), color change ( $\Delta E^*$ ), anti-swelling efficiency after water immersion ( $ASE$ ) and hygroscopicity reduction ( $HR_{20^{\circ}\text{C}, 65\%}$ ) due to heat treatment were determined.

### 3 Results and discussion

Table 1 presents the most important physical properties of heat-treated lime wood as a function of the applied heat treatment conditions.

The most important indicator of the heat treatment efficiency is the mass loss, because it reflects the degradation

severity of the chemical composition of wood. The higher the mass loss, the better the dimensional stabilization attained, but at the same time, the wood becomes weaker. According to Viitaniemi et al. (1997), the mass loss should not exceed 5 % (with possible extension up to 6 % for some species) in order to keep the strength losses at an acceptable level.

In spite of the longer duration than that used by Barboutis et al. (2011), the mass loss values obtained at  $180^{\circ}\text{C}$  are still low ( $<2.5\%$ ), hinting towards a low efficiency of the heat treatment. However, lime wood is extremely sensitive to temperature: the mass loss values obtained at  $200^{\circ}\text{C}$  are significantly higher, reaching up to 9.3 %. In order to limit the mass loss to a safe  $ML = 5\%$ , the recommended heat treatment would be  $200^{\circ}\text{C}/2$  h. Under these conditions, the anti-swelling efficiency would be  $ASE_{v, lime} = 53.2\%$ , significantly higher than the one obtained by Olarescu and Campean (2012) under similar experimental conditions and at the same mass loss value of 5 %, with spruce wood originating from the same forest parcel ( $ASE_{v, spruce} = 42.3\%$ ).

Apart from its aesthetical importance, color is a good indicator of the heat treatment efficiency: the darker wood color becomes, the more efficient the heat treatment can be considered.

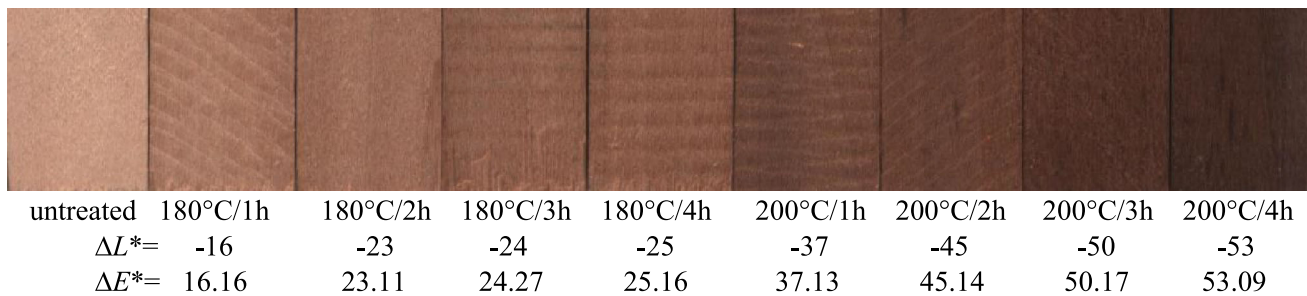
Figure 1 presents the color modification of lime wood under the influence of different heat treatment conditions.

Out of the three color coordinates ( $L^*a^*b^*$ ), the lightness had the greatest decrease ( $\Delta L^*$  up to  $-53$  at  $200^{\circ}\text{C}/4$  h), while the change of  $a^*$  and  $b^*$  coordinates was insignificant ( $<5$ ). Thus, the greatest part of the total color change  $\Delta E^*$  was caused by the lightness change (Fig. 1).

Considering that both the mass loss and the total color change are indicators of the heat treatment efficiency, it seemed interesting to establish a mathematical correlation between them, which would enable determining one indicator (i.e. mass loss) by measuring only the other (i.e. color change). A logarithmic regression equation was found:

**Table 1** Mass loss, anti-swelling efficiency and hygroscopicity reduction of heat-treated lime wood compared to untreated wood

Heat treatment conditions	Mass loss ML, %	Anti-swelling efficiency			Hygroscopicity reduction $HR_{20^{\circ}\text{C}, 65\%}$ , %
		$ASE_r$ , %	$ASE_{tg}$ , %	$ASE_v$ , %	
180 °C/1 h	1.3 ± 0.2	10.8	16.3	13.5	7.9
180 °C/2 h	1.7 ± 0.1	13.1	24.3	19.3	12.4
180 °C/3 h	2.1 ± 0.2	17.2	33.5	26.7	13.1
180 °C/4 h	2.4 ± 0.3	19.0	34.4	26.9	13.8
200 °C/1 h	4.3 ± 1.1	43.1	56.2	52.1	22.5
200 °C/2 h	5.0 ± 0.4	45.9	57.2	53.2	26.5
200 °C/3 h	6.7 ± 0.4	47.1	59.5	59.1	28.8
200 °C/4 h	9.3 ± 0.7	53.9	66.4	66.4	33.0



**Fig. 1** Change of lime wood color as effect of different heat treatment conditions ( $\Delta L^*$  lightness change,  $\Delta E^*$  total color change)

$$\Delta E^* = 20.071 \cdot \ln(ML) + 10.023 \quad (R^2 = 0.98) \quad (1)$$

Based on the obtained experimental values, mathematical correlations were established between the anti-swelling efficiency ( $ASE_v$ ) and the mass loss (Eq. 2), and between the hygroscopicity reduction ( $HR_{20^\circ\text{C}, 65\%}$ ) and the mass loss (Eq. 3), respectively. Both turned out to also be logarithmic functions, same as in the case of the color change-mass loss dependency.

$$ASE_v = 27.381 \cdot \ln(ML) - 0.6962 \quad (R^2 = 0.91) \quad (2)$$

$$HR_{20^\circ\text{C}, 65\%} = 13.159 \cdot \ln(ML) + 3.8616 \quad (R^2 = 0.90) \quad (3)$$

These regression equations enhance estimating the two efficiency parameters for any different heat treatment conditions, provided the mass loss is known.

#### 4 Conclusion

The present research led to the following findings:

1. The heat treatment of lime wood at 180 °C causes insignificant mass loss (<2.5 %), even after 4 h of treatment. Consequently, the heat treatment efficiency can be considered only moderate: the volumetric ASE ranges in this case only up to 27 % and the hygroscopicity reduction up to 13.8 %.
2. Lime wood is quite sensitive to temperature. This fact is supported by the significant increase of the mass loss and heat treatment efficiency at 200 °C. Even a short (1 h) treatment at this temperature leads to better results than long-term (4 h) treatment at 180 °C. All analyzed properties (color change, anti-swelling efficiency and hygroscopicity reduction) doubled their values at 200 °C/1 h compared to 180 °C/4 h.
3. The results concerning the dimensional stabilization of lime wood at 200 °C/4 h are quite spectacular, considering the obtained ASE values after water immersion:  $ASE_r, 200^\circ\text{C}/4\text{ h} = 54\%$ ,  $ASE_{tg}, 200^\circ\text{C}/4\text{ h} = 66\%$ ,  $ASE_v, 200^\circ\text{C}/4\text{ h} = 66\%$  and the hygroscopicity reduction

$HR_{20^\circ\text{C}/65\%} = 33\%$ . However, these values are correlated with a high mass loss ( $ML_{200^\circ\text{C}/4\text{ h}} = 9.3\%$ ).

4. In order to limit the mass loss to a safe 5 %, the recommended heat treatment would be 200 °C/2 h. The less dark color with this treatment conditions (Fig. 1) is more pleasant and the results concerning the dimensional stabilization and hygroscopicity reduction are also satisfying:  $ASE_v, 200^\circ\text{C}/2\text{ h} = 53\%$  and  $HR_{20^\circ\text{C}/65\%} = 26\%$ .
5. The correlations between the different properties determined in this research and the mass loss associated with the eight different heat treatment conditions can be employed as a user-friendly tool for rapid determination of the heat treatment effects as a function of the one with highest visual impact and which is also rapidly measured at low costs: the color change. Thus, the color scale in Fig. 1 allows the user to choose the heat treatment conditions as a function of the desired color. Then, one may see the expected mass loss for these conditions from Table 1. The estimated mass loss value further leads, by means of the regression equations in Eqs. 2 and 3, to the estimated anti-swelling efficiency and hygroscopicity reduction, respectively.

The results obtained will be realized in the manufacturing of solid wood panels made of heat-treated lime wood lamellas for outdoor uses. Further research concerning the dimensional stability and shape stability of these panels under outdoor conditions will be carried out.

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