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The effect of heat treatment on wood-water relationship and mechanical properties of commercial uruguayan plantation timber *Eucalyptus grandis*

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ABSTRACT

Heat treatment of wood is well known to enhance its resistance against biodegradation. This is an effect of the lower equilibrium moisture content, caused by the thermal degradation of structural wood components. At the same time, mechanical properties are known to decrease with temperature and treatment duration. In the study at hand, samples of Uruguayan *Eucalyptus grandis* (W. Hill ex Maiden) were thermally modified in two different temperature regimes, with distinct time for each heat treatment. We found the hygroscopicity to be reduced. Elasticity was not altered significantly by the heat treatment. Anyways, the maxima of compressive, bending and impact bending strengths decreased with increasing temperature. Brinell hardness showed be only slightly influenced by heat. Treated *E. grandis* may be a suitable substitute for typical tropical hardwood applications like deckings and outdoor utilization.

INTRODUCTION

Eucalyptus spp.is an angiosperm genus originally from Australia and being commercially planted in the whole subtropics, especially South America. Around 120 yrs. ago it was introduced in Uruguay. The Uruguayan forestry sector made enormous efforts in the plantation sector. Today more than 800,000 ha are available, of which only 10 % are situated in the Rivera, Tacuarembó and Paysandú departments. The here planted species *E. grandis*is primarily used for timber, solid wood and plywood products (DIEA, 2010).

Still the high value utilization of *E. grandis*as solid product is below its potential. Reasons are the low durability and insufficient dimensional stability. Native tropical species like Ipê (*Tabebuia* spp.) or Massaranduba (*Manilkara bidentata*) are more durable and show advantages in mechanical properties. Strong environmental concerns, rising timber prices and recently introduced market restrictions, like the European timber trade regulation (EUTR), demand for suitable alternatives.

Basics of wood-water relationship:

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The moisture content (MC) influences many material properties, especially the mechanical properties. The fibre saturation point (FSP) is the threshold MC at which only physically/chemically bond water is present. It is estimated to be around 30%, depending on density and sorption direction (Simpson and TenWolde, 1991). Another important wood characteristic is its equilibrium moisture content (EMC). As Simpson and TenWolde (1991) describe, the MC of wood below the FSP is a function of relative humidity and ambient temperature. Wood suffers seasonal and daily EMC changes through changes in the surrounding air. Depending on either water is adsorbed (wetting) or desorbed (drying) the absolute EMC differs. This effect is also known as water hysteresis (Simpsons and TenWolde, 1991).

Heat induced chemical changes:

Heat treatment is known to effect hygroscopicity by influencing EMC and water hysteresis effect. In practice, this is relevant for dimensional stability of timber products (Ibach, 2001). The heat treatment parameters, as temperature, heating rate (hr) duration, atmosphere gas and pressure as well as the wood's chemical composition influence the degree of modification degree.

Wood cell walls are based on cellulose, hemicelluloses, as well as lignin of which each shows different decomposition temperatures. Extractives possess much lower decomposition temperatures. Substances like lipids, resins, gum, starch and simple metabolic intermediates are non-structural components which rather influence surface properties (Miller, 1999). Those compounds may already be changed or decomposed at low (< 130 °C) temperature (White and Dietenberger, 2010). On the other hand, new compounds emerge due to degradation of structural macro-molecules. This does not affect mechanical properties, but effects the wood-water relationship characteristics (Esteves and Pereira, 2009).

The strength decrease due to heat treatment is a consequence of reactions of the three structural lignocellulose components.

As the first structural molecule, hemicellulose starts with de-acetylation reactions in low temperature range, i.e. below 130 °C. The resulting acetic acids then additionally catalyse depolymerisation of structural polysaccharides.

Cellulose, due to its high crystallinity, is less susceptible to heat and keeps being stable up to 260 °C. Before that, amorphous regions of the cellulose degrade. Hence, the overall portion of crystalline cellulose increases. Subsequently, less free hydroxyl groups are accessible for water molecules. At temperatures above 200 °C, even lignin may cleave off polyphenolic components. However, those rather cross-link with other structural molecules than to decompose, which leads to less accessible hydroxyl groups and an apparently higher lignin content (Esteves and Pereira, 2009).

Complete decomposition of hemicellulose, cellulose and lignin is only possible at 225 °C, 300 °C and more than 300 °C, respectively (Sundqvist, 2004).

Once wood is treated at temperatures above 300 °C, a severe wood degradation, i.e. carbonization is the result. The known commercial heat treatment processes happen to be in oxygen-reduced atmospheres and temperatures between 180 to 260 °C. Treatment durations range from a few minutes to several hours (Ibach, 2001).

MATERIAL AND METHODS

Two different heat treatments were applied on pre-dried *E. grandis*. The sample material was cut from a commercial plantation in Uruguay. The sample was divided in three groups: heat treatment 1 (H1); heat treatment 2 (H2); control samples (K). The initial MC was determined through the oven-drying method. The initial and final density was estimated by dividing the sample weight at 12 % by the sample volume at 12 %.

Heat treatment:

The control sample was pre-dried at a muffle furnace, for around 24 h at 80 °C and conditioned at standard climate (20 °C, 65 %) for three months. The sample groups H1 and H2 were treated in a modified muffle furnace in a saturated steam atmosphere. The H1 samples were heated until 150 °C (h_r: 0.36 °C/min), staying at this temperature for 4 h. The H2 samples were heated until 200 °C (h_r: 0.75 °C/min) and stayed at that temperature for 3 h. After the treatment, the samples stayed 24 h inside the muffle (cooling time) proportionating a stress relief period for the wood and avoid possible micro cracks.

Wood-water relationship (sorption):

All sample groups were analysed on a sorption balance system SPSx-1µ (Pro Umid, Ulm), following the principles of DIN EN ISO 12517 (2005). The isotherm graphs were done with six different climates (RH: 20, 35, 50, 65, 80, 92 [%]) at a constant temperature of 20 °C. By continual weighing of the specimens the point of constant weight was identified and defined as the regarding EMC equilibrium. Solid samples (1cm³ cubes) as well as flaked samples have been tested this way.

Wood-water relationship (wettability):

The contact angle was measured with a goniometer (Krüss DSA4). A sessile drop of 5 µldeionized water was placed on the surface of the wood specimen. The contact angle between the drop and the wood surface was video-captured and measured after t=5 s. The higher the contact angle is, the lower the wettability of the surface.

Mechanical properties:

As shown in table 1, four mechanical tests were conducted, according to their respective DIN standard. The indices (see table 1) relate to the testing method and will be used throughout the article at hand.

Static bending and compression tests were done on an UTM (Zwick/Roell Z050). For impact bending, the machine was operated with a non-instrumented pendulum (150 Nm hammer). For the Brinell hardness the Zwick/KG (Z 425) machine was utilized. Due to the dark colours of the treated specimens, an exact diameter measurement had to be facilitated with a carbon paper negative.

Table 1: Mechanical tests and their corresponding DIN.

MECH. TESTS	DIN
Static bending $_{\mathbf{SB}}$	51286 (1978)
Impact bending $_{\mathbf{IB}}$	52189 (1981)
Compressive strength $_{\mathbf{CS}}$	52185 (1976)
Brinell hardness $_{\mathbf{HB}}$	1524 (2011)

RESULTS AND DISCUSSION

The moisture content of the samples prior to treatment was found to be 15.7 % in average. The applied temperature regimes influenced mechanical and physical properties at different degrees. The bulk density dropped from initially 0.62 g/cm³ (K) to 0.55 g/cm³ (H1) and 0.56 g/cm³ (H2). The observed density loss (i.e. mass loss) of about 12 % was significant (H1), but did not increase with rising temperature (H2).

Wood-water relationship (sorption):

The treatments reduced the hygroscopicity. Anyway, H1 lowered the EMC at maximum RH only by 1.9 %, while H2 decreased the regarding EMC by 5.2 %. It can be explained due the thermal modification of wood components, namely hemicellulose, cellulose and lignin. The hydroxyl groups of the hemicelluloses bind water chemically. The applied heat reduced the available OH^- groups. The less OH^- groups are available, the lower the wood's hygroscopic capacity to adsorb humidity from the surrounding air. The H1 treatment resulted in less EMC change than H2. The relation between EMC and RH in adsorption and desorption is shown in isothermal curves in figure 1 (solid) and 2 (flaked).

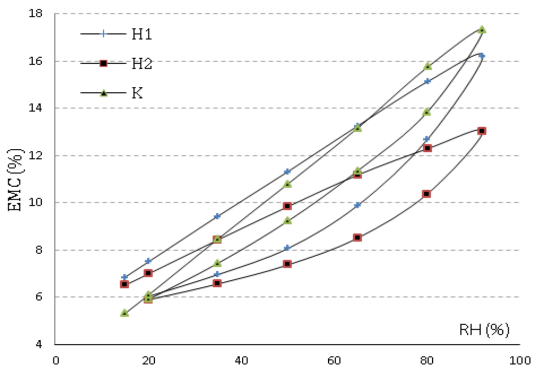

Fig. 1: Sorption isotherms for flaked samples from groups K, H1 and H2.

When integrating the area under the sorption isothermal curves and comparing them to each other, we found the hysteresis effect to be increased with the heat treated samples (H1<H2<<K). For solid samples, the reduction was less than for flaked samples. The higher specific surface area of the flaked samples facilitated the interaction between sample and surrounding humidity.

Wood-water relationship (wettability):

The contact angles of treated wood was found to be higher than the K samples. The samples shown in figure 3 show the increase of 19.9 % (H1) and 31.9 % (H2) compared to K.

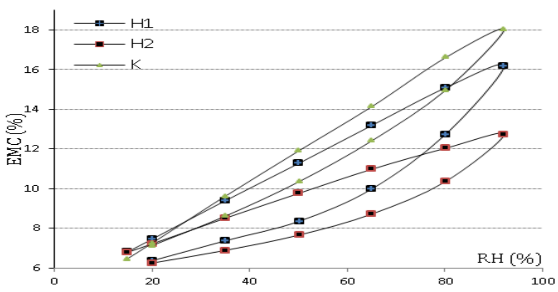

Fig. 2: Sorption isotherm for solid samples from groups K, H1 and H2.

Fig. 3: Typical sessile water drops showing the differently wettable surface of K, H1 and H2.

Hence, the higher treatment temperature resulted in significantly higher contact angle (see also table 3). For H2, some specimens presented superficial micro cracks, facilitating entrance of water droplets. Anyway, the slightly higher contact angle of H2 is not significant.

Table 2: Contact angle (°) between sample and drop as well as the regarding COV (%).

Sample group	Contact angle
K	$53{,}50^{A}$ (15,01)
H1	$64{,}17^{B}$ (22,48)
H2	$69{,}29^{B}$ (14,16)

Mechanical properties:

While mechanical strengths were negatively influenced by the heat treatment, elastic properties suffered only slight decrease. The results are summarized in table 4. Flexural modulus of rupture (MOR_{SB}), compressive strength (MOR_{CS},) as well as the impact bending strength (a_{IB}) reduced significantly by 22.0 %, 8.8 % and 61.11 % for H1 and 26.0 %, 14.35 % and 62,96 % for H2, respectively. Brinell hardness (BHN) seemed to be lower after both treatments, anyway the high coefficients of variance made those results inconclusive.

Taking K as reference, H1 showed slightly lower values for MOE_{SB} and MOE_{CS} (2.97 % and 7.09 %). When comparing to H2 apparently an increase in MOE_{SB} (3.87 %) and a decrease in MOE_{CS} (4.96 %) was observed. However, there was no significant difference between H1 and H2 for MOE_{SB}. The changes of MOE_{CS} were statistically different from K but not significantly different one to each other.

The MOR is the maximum tension that a material can resist (Hiziroglu, 1972). MOR_{SB} and MOR_{CS} show differences between H1 and H2. The samples tested on compression suffered a stronger strength decrease than the flexural samples.

Cellulose is the main compound of wood and its mass loss is related to the loss of strength properties. Mass loss of hemicelluloses is connected to increasing brittleness of wood. Although elastic moduli did not lower significantly, H1 and H2 made the material more brittle. During the mechanical tests abrupt ruptures were observed.

Wood is an orthotropic material and its strength properties depend on the load direction (longitudinal, radial or axial). Wood fibres are arranged longitudinally. It provides higher $MOE_{CS}$when the load (compression) is applied parallel to the fibre.

Table 3: Modulus of elasticity (MOE), modulus of rupture (MOR), impact bending strength (a_{IB}), Brinell hardness (BHN), their regarding coefficient of variance (COV [%]), as well as the Fisher–Snedecor distribution (F-Ratio).

Sample group	MOE_{SB} (N/mm²)	MOR_{SB} (N/mm²)	MOE_{CS} (N/mm²)	MOR_{CS} (N/mm²)	a_{IB} (kg*m/cm²)	BHN (N/mm²)
K	12241^{A} (12,48)	100^{A} (12,06)	15707^{A} (17,75)	$62{,}70^{A}$ (13,49)	$0{,}54^{A}$ (20,38)	$20{,}52^{A}$ (25,18)
H1	11877^{A} (13,22)	78^{B} (24,41)	14593^{B} (18,68)	$57{,}18^{B}$ (13,94)	$0{,}21^{B}$ (44,01)	$17{,}72^{A}$ (32,23)
H2	12715^{A} (18,12)	74^{B} (25,04)	14928^{B} (15,14)	$53{,}70^{C}$ (14,04)	$0{,}20^{B}$ (34,51)	$18{,}97^{A}$ (35,38)
F-Ratio	1,31	16,52	4,61	30,95	107,59	1,47

Conclusion:

The treatment of *E. grandis*showed promising results for low temperature (H1) and even high temperature treatment (H2). Elasticity losses were insignificant or very few, while the strength and failure mode were clearly affected by both treatments. The loss of hardness is negligible which makes the treatment interesting for decking products.

On the other hand, sorption behaviour improved significantly, hence dimensional stability as well. The worse wettability may lead to challenges in gluing of engineered timber products.

Further investigations shall focus temperatures in between 150 °C and 200 °C as well as shorter treatment durations. This way, the undesirable side-effects (micro-cracks) should be avoided in practice. Small internal cracks reduce drastically strength properties.

Additionally, practical experiments regarding dimensional stability and glue application should be conducted.

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