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EFFECT OF HEAT TREATMENT ON COLOR, WEIGHT LOSS, SPECIFIC GRAVITY AND EQUILIBRIUM MOISTURE CONTENT OF TWO LOW MARKET VALUED TROPICAL WOODS

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ABSTRACT

Despite of the great diversity of Brazilian forests, only a few species are well known and commercialized, but many others could provide wood with potential applications in the wood industry. In this study, heat treatment was analyzed as a way to add value to low market valued tropical woods. The effect of heat treatment performed at 160 and 220°C was assessed on color, weight loss, specific gravity, and equilibrium moisture content (EMC) of cedroarana (*Cedrelinga catenaeformis*) and cedro-marinheiro (*Guarea trichilioides*) woods. Heat treatments caused weight loss, darkening, and decreased EMC, as the maximum temperature increased. However, no changes in specific gravity were observed as a function of heating. Cedro-marinheiro has presented the highest changes in color and weight as a function of heating, while cedroarana has shown the highest reduction in EMC. Heat treatments improved some wood properties, being a good approach to improve low market valued tropical woods. Results also suggest that resilience to reduction of hygroscopicity after heating might be related to wood specific gravity.

KEYWORDS: Heat treatment, thermal treatment, weight loss, specific gravity, equilibrium moisture content, color changes.

INTRODUCTION

Tropical timbers have high commercial value in market and wood industry due to their good appearance, excelent physical, mechanical, and machinability properties. Despite of the immense tree diversity of tropical forests in Brazil, only a few species are well known, explored and sold on local markets, but many others can provide wood with good properties and high potential for applications in the wood industry.

The most current methods for wood preservation are the chemical treatments. However, due to high toxicity of some preservative products and the raising environmental awareness, many of these heavy-metal based products have been banned in Europe and North-America. Therefore, the wood product manufacturers have been demanded to look for alternative and clean technologies to modify wood-based products and improve their properties.

In this context, thermal treatment appears as an environmental friendly and low cost way to modify and improve the properties and color of wood products. In this process, heat is applied to wood under atmospheric conditions and in presence or scarcity of oxygen (Brito et al. 2006) at temperatures within the range of 100 to 250°C (Guedira 1988; Vovelle and Mellottee 1982). Compared to unmodified wood, thermally treated wood exhibits higher dimensional stability, lower hygroscopicity, changes in colors and better resistance to fungi and weathering (Johansson, 2008; Mohebby and Sanaei 2005; Guedira, 1988; Vovelle and Mellottee 1982). The new properties of the treated material present several interesting aspects for the wood industry, being suitable to exterior and interior applications. Thus, the heat treatment can be considered as a sustainable solution to add value to wood.

During the heat treatment, wood is completely dried and its hygroscopic nature is changed by means of the destruction of OH-groups. As wood is dried in response to relatively low temperatures (below 120°C), water is firstly evaporated from the cells lumens, reaching the fiber saturation point (FSP), and subsequently removed from the cell walls. In response to a higher range of heating temperatures (above 120-150°C), the dynamics of thermal degradation of wood is a complex research topic, as wood contains a complex variety of cellular and polymeric structures and chemistry (Winandy and Rowell 1984). The actual effect of thermal degradation on wood properties is related to its chemical composition, as the wood chemical components (cellulose, hemicelluloses and lignin) show different behaviors when exposed to heat (Prins et al. 2006).

The changes in chemical composition of wood caused by heating result in lower hygroscopicity, with a major effect on both dimensional stability and resistance to fungi. In order to reach a selective degree of depolymerisation of hemicelluloses during the treatment, relatively mild heating conditions can be applied to limit unwanted side reactions, which could affect wood mechanical properties (Tjeerdsma et al. 1998). Species with higher hemicellulose content might present a substantial increase in hydrophobicity after treatment. In addition to changes in the hydrophobic character, the densification of lignin chain is also a factor to explain the increased resistance to fungi in thermally treated woods (Duchez and Guyonnet 1998).

The change in wood hygroscopicity has also been attributed to chemical changes in wood components or physical changes in relative crystallinity at the surface (Bourgois and Guyonnet, 1988; Obataya et al. 2000). According to Poncsak et al. (2005), when heat is applied to wood, the hemicelluloses, wood polymer relatively easy to hydrolyse, starts to be degraded and its mass significantly decreases with increasing the residence time and temperature of treatment. The relative crystallinity proportion of wood increases due to crystallization in quasicrystalline region in wood cellulose and in hemicelluloses. At the same time, during a heat treatment, esterification of hydroxyl groups and cross-linking reactions take place. As a consequence of the factors above, the OH groups available for moisture adsorption in wood are reduced, decreasing the higroscopicity and EMC of wood.

Brito et al. (2006) studied the specific gravity of *Eucalyptus grandis* wood submitted to thermal treatment at 120, 140, 160, 180 and 200ºC. The authors report that the specific gravity of thermally treated wood is not different from that obtained from natural wood. However, in a previous study by Kortelainen et al. (2005) on samples of Scots pine and Norway spruce thermally treated at 130 and 230°C, both weight loss and specific gravity reduction by heat were reported.

Concerning the color changes, some studies have already reported that thermal treatment induced wood darkening and reddening (Johansson and Morén 2006; Lopes 2010; Aksoy et al. 2011). Other authors, however, report reduction of red tonality as a function of heating (Pincelli 1999; de Moura and Brito 2011). The blue-yellow coordinate, in turn, tends to decrease with heating (Pincelli 1999; Lopes 2010; Aksoy et al. 2011; de Moura and Brito 2011). In general, heat treated wood is often appreciated for its light-brown to dark-brown appearance (Viitanen et al. 1994).

Cedroarana (*Cedrelinga catenaeformis*) and cedro-marinheiro (*Guarea trichilioides*) are often considered as low market valued tropical woods, due to their light and non-attractive colors and to the lack of technologic knowledge on their properties. In this work, the thermal treatment aims to improve some characteristics and properties of cedroarana and cedro-marinheiro woods. In this context, the effect of the heat treatment performed at 160 and 220°C was assessed on some physical aspects (color, weight loss, specific gravity, and equilibrium moisture content) of these two tropical woods.

MATERIAL AND METHODS

Testing materials

Cedroarana (*Cedrelinga catenaeformis*) and cedro-marinheiro (*Guarea trichilioides*) wood samples were used in this study. The choice of these species was based on a research about the main low-quality and low-valued wood species in sawmills located in the State of São Paulo, Brazil. Both wood species are qualified as low-quality wood due to their natural imperfections (e.g. knots, cross grain etc), drying defects and unattractive color. Altough these wood species are already milled in Brazil, it is believed that thermal treatment could add more market value to them.

Commercial 12 % EMC flat-sawn sapwood boards were obtained for each species from a sawmill in the municipality of Piracicaba, State of São Paulo. These boards were sawn into samples measuring 54 cm (L) by 6 cm (T) oriented-grain. Then, these boards were freshly planed to a thickness of 2.3 cm (R). In total, 7 boards (including 1 untreated control sample) were chosen from each species, avoiding natural defects like knots, shakes, cross grain, reaction wood and insects damage.

Wood samples had previously undergone chemical characterization analyses, by means of determination of Klason lignin (L %) and total extractives soluble in ethanol: toluene at a 2:1 ratio, and in hot water (E %). The holocellulose content (cellulose and hemicelluloses; H %) was calculated by subtracting the sum of lignin and extractives from the total dry weight being analyzed (H % = 1 – (E % + L %)). Wood samples used in this experiment are chemically characterized as shown in Tab. 1.

Tab. 1: Means of lignin, extractives and holocellulose contents of cedro-marinheiro (Guarea trichilioides) and cedroarana (Cedrelinga catenaeformis) woods.

Species	Contents	Lignin	Extractives	Holocellulose
Cedro-marinheiro	Percent content (%)	29.50 (0.19)	20.45 (0.37)	50.05 (0.34)
	Weighted content ($g.cm^{-3}$)	0.18 (0.19)	0.12 (0.37)	0.31 (0.34)
Cedroarana	Percent content (%)	29.35 (0.18)	10.51 (0.33)	60.14 (0.27)
	Weighted content ($g.cm^{-3}$)	0.12 (0.18)	0.04 (0.33)	0.25 (0.27)

Values in brackets are coefficients of variation of means (CV %)

Weighted content ($g.cm^{-3}$): percent contents are weighted as absolute values as a function of specific gravity (0.611 $g.cm^{-3}$, for cedro-marinheiro; 0.411 $g.cm^{-3}$, for cedroarana).

Thermal treatments

The thermal treatments were performed in the Integrated Laboratories of Chemistry, Cellulose and Energy (LQCE) of the Forest Sciences Department at Escola Superior de Agricultura "Luiz de Queiroz", University of São Paulo (ESALQ/USP). The boards were thermally treated in an electrical resistance oven equipped with a forced-air system having a nominal volume of 0.45 m^3. The maximum temperatures applied were 160 and 220°C with heating rates of 0.03 and 0.05°$C.min^{-1}$, respectively. These temperatures were chosen based on previous studies of Vovelle and Mellottee (1982) and Crow and Pickles (1971), and the heating rates employed were based on those applied by Deglise and Magne (1987) and Graham et al. (1984). Fig. 1 depicts the programs of the thermal treatments applied.

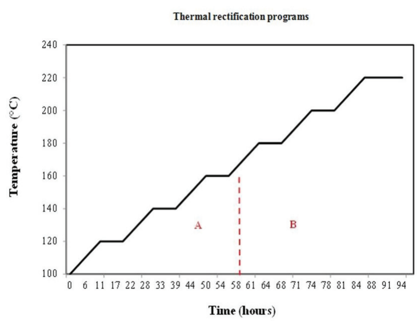

Fig. 1: Heat treatments (A = 160°C; A + B = 220°C).

Prior to applying the thermal treatments, all wood samples were dried in a forced-air oven at 103 ± 2°C to record the oven-dried mass. For both species and maximum temperatures tested, three repetitions were applied. After each thermal treatment, the samples were kept inside the oven for cooling until reaching 30°C. Then, all the thermally treated samples were placed again on a forced-air oven at 103 ± 2°C for 48 hours to obtain their oven-dried mass after the heat treatment. This measure was used to evaluate the weight loss (WL) caused by the thermal treatments applied, according to the following formula:

$$WL = (M_i - M_f)/M_i \times 100 \quad (1)$$

where: M_i and M_f - the oven-dried masses before and after the thermal treatment, respectively.

Color assessment

Color measurements of thermally treated and control wood samples were performed using a spectrophotometer Konica Minolta CM-2500D connected to a microcomputer using a D65 illuminant and 10° standard observer angle. Two color measurements were undertaken on both tangential faces of boards, totalizing four measurements per sample. The diameter of spots for color measurement used was 8 mm. Color measurements of wood samples were performed at conditions of 24°C and 60 % relative humidity.

The color parameters measured by the spectrophotometer were determined according to the CIE L*a*b* color system, where L* describes lightness and a* and b* describe the chromatic coordinates on the green-red and blue-yellow axes, respectively.

Wood specific gravity assessment

The changes in wood specific gravity caused by heat treatment were evaluated. For this purpose, specimens with 6 cm (L) x 3 cm (T) x 2 cm (R) were obtained from boards using a circular saw. The specific gravity of the specimens was determined by the hydrostatic balance method (Barrichello 1983) and the dry mass was measured by the oven dry basis (Rasmussen 1968; Galvão and Jankowsky 1985). Firstly, the treated and control specimens were saturated in an autoclave, then, their mass in air and immersed in water were determined using a hydrostatic balance. These specimens were then oven-dried in a forced-air oven at 103 ± 2°C until reaching a constant mass, obtaining their oven-dried mass. The specific gravity (SG) of the treated and control specimens were determined according to the equation below:

$$SG = M_{0D}/(M_{air} - M_{water}) \quad (2)$$

where: M_{0D} - the anhydrous mass (oven-dried) of the specimens, and
M_{air} and M_{water} - the saturated mass of the sample in air and immersed in water, respectively.

Equilibrium moisture content (EMC)

The changes in EMC caused by the heat treatments were evaluated on the same specimens that were subjected to evaluation of specific gravity. For this purpose, all specimens were placed in a conditioning chamber at 65 ± 5 % relative humidity and 20 ± 2°C. The specimens were kept under these conditions until they reached an equilibrium constant mass and their weights have been recorded. The equilibrium moisture content was calculated before and after the heat treatment using the equation below:

$$EMC = (M_C - M_{OD})/M_{OD} \times 100 \quad (3)$$

where: MC and MOD - the mass of the specimen in equilibrium at the conditioning chamber and the oven-dried mass, respectively.

Statistical analysis

Statistical data assessment was performed by means of Analysis of Variance (ANOVA), multiple comparisons among means (Tukey's tests), as well as general descriptive statistics, and correlations analysis (Pearson) to evaluate relationship among the variables studied. Prior to the statistical data analysis using ANOVA, tests for data normality and variance homogeneity were run in order to verify whether data respected the assumptions of the test.

RESULTS AND DISCUSSION

The effect of heat treatment at 160 and 220°C on color, weight loss, specific gravity and equilibrium moisture content (EMC) of cedroarana and cedro-marinheiro woods is shown in Tabs. 2 and 3. In general, the heat treatments applied caused changes in color in both species evaluated, the weight of the samples was reduced, the wood EMC decreased, and specific gravity remained unchanged.

Tab. 2: Multiple statistical comparisons among means for lightness index (L), red-green coordinate (a*), and blue-yellow coordinate (b*) of thermally treated samples of cedro-marinheiro (Guarea trichilioides) and cedroarana (Cedrelinga catenaeformis) woods.*

Species	Thermal treatment	L*	C1	C2	a*	C1	C2	b*	C1	C2
Cedro- marinheiro	Untreated	53.5 (1.9)	B	a	13.7 (1.5)	B	b	24.6 (2.4)	C	b
	160°C	51.2 (2.5)	B	a	13.5 (2.2)	B	b	21.5 (3.3)	B	b
	220°C	41.6 (3.2)	A	a	10.3 (0.7)	A	b	17.1 (1.8)	A	a
Cedroarana	Untreated	55.8 (7.7)	B	a	8.3 (6.8)	A	a	18.4 (5.4)	A	a
	160°C	53.3 (4.9)	B	a	8.2 (2.4)	A	a	19.0 (6.8)	A	a
	220°C	46.7 (2.8)	A	b	8.1 (4.9)	A	a	17.7 (4.5)	A	a

Means within a column followed by the same letter are not significantly different at the 5 % probability level (Tukey's multiple comparison tests).

Values in brackets are coefficients of variation of means (CV %)

C1: Capital letters are for thermal treatment comparisons, for each species separately.

C2: Lowercase letters are for species comparisons, for each thermal treatment separately.

Tab. 3: Multiple statistical comparisons among means for specific gravity (SG), equilibrium moisture content (EMC), and weight loss (WL) of thermally treated samples of cedro-marinheiro (Guarea trichilioides) and cedroarana (Cedrelinga catenaeformis) woods.

Species	Thermal treatment	SG	C1	C2	EMC (%)	C1	C2	WL (%)	C1	C2
Cedro- marinheiro	Untreated	0.611 (1.63)	A	b	9.3 (1.1)	C	a	-	-	-
	160°C	0.628 (3.18)	A	b	8.6 (2.3)	B	a	0.34 (5.88)	A	a
	220°C	0.650 (1.53)	A	b	6.1 (4.9)	A	a	6.59 (9.71)	B	b
Cedroarana	Untreated	0.411 (2.43)	A	a	11.6 (0.9)	B	b	-	-	-
	160°C	0.435 (6.89)	A	a	10.8 (5.6)	B	b	0.29 (3.44)	A	a
	220°C	0.451 (6.65)	A	a	8.0 (1.3)	A	b	1.58 (5.69)	B	a

Means within a column followed by the same letter are not significantly different at the 5 % probability level (Tukey's multiple comparison tests).

Values in brackets are coefficients of variation of means (CV %)

C1: Capital letters are for thermal treatment comparisons, for each species separately.

C2: Lowercase letters are for species comparisons, for each thermal treatment separately.

Color

For both maximum temperatures tested, cedroarana and cedro-marinheiro woods became darker (i.e. decrease in L* coordinate) and presented a reduction in a* and b* coordinates. These changes in color were higher with increasing maximum temperature. These results are in accordance with the findings of Aksoy et al. (2011), who assessed the color changes of oven heat-treated scot pine at 150, 175, and 200°C. The authors reported that oven heat-treated wood at 175 and 200°C acquired darker tonality, while b* coordinate decreased. The decrease in values of a* and b* coordinates means that heat treated wood got less saturated color compared to untreated wood. All these improvements in color could add value to both wood species. Indeed, color is a very important wood property for the final consumer choice. As the decorative look often prevails, color is a determining factor for the selection of a specific wood (Esteves at al. 2008). Hence, since species with a pale couloured wood are usually regarded as less attractive to the market, heat treatment could be useful at darkening and giving the wood a "tropical wood aspect".

As seen in Tab. 2, cedro-marinheiro wood has shown higher changes in color than those observed in cedroarana wood after heating, which suggests that the former species is more susceptible to color changes by heat than the latter one.

During heating, the wood color is modified due to the release of byproducts from the degradation of hemicelluloses (Sehlstedt-Persson 2003; Sundqvist 2004) and other wood components, such as extractives, which confer colors to wood (McDonald et al. 1997). According to Tjeerdsma et al. (1998), Mitsui et al. (2001) and Bekhta and Niemz (2003), the oxidation of products such as quinines also contributes to changes in the color of wood.

Weight loss (WL)

As expected, the weight loss of both wood species increased as a function of the maximum temperature in heat treatment, and the most pronounced weight loss occurred at 220°C. This result corroborates that obtained by Gunduz and Aydemir (2009), who investigated the weight loss and the specific gravity of heat-treated Camiyani Black Pine wood at temperatures of 160, 180 and 200°C for 2 and 6 hours. Their results showed that increasing heat treatment maximum temperature decreased specific gravity and increased weight loss.

The weight loss of heat treated samples at 160 and 220°C were respectively of 0.34 and 6.59 %, for cedro-marinheiro wood, and of 0.29 and 1.58 %, for cedroarana wood. As seen in Tab. 3, cedro-marinheiro wood presented a higher weight loss than that observed in cedroarana wood. This indicates that the former species is more susceptible to thermal degradation than the latter one. This behavior must be caused by differences in chemical composition between both species. The weight loss of the thermally treated wood samples is due to the degradation of wood polymers (cellulose, hemicelluloses and lignin), mainly the hemicelluloses in this range of temperature, which are the most thermally-sensitive wood components (Poncsak et al. 2005; Yildiz et al. 2006; Kocaefe et al. 2007; Mohebby and Sanaei 2005).

Specific gravity (SG)

Tab. 3 also shows the specific gravity of thermally treated and control samples of cedroarana and cedro-marinheiro woods. It can be seen that the range of maximum temperatures tested could not induce significant changes in specific gravity of both wood species studied. Similar results were also found by Brito et al. (2006).

Equilibrium moisture content (EMC)

The variation of EMC of cedroarana and cedro-marinheiro woods, as a function of heat treatments, is shown in Tab. 3. As the maximum temperature of treatment increased, the EMC was lineary reduced. The highest temperature in heat treatment (220°C) has caused a more severe degradation of wood hydrophilic carbohydrates, inducing higher weight loss and further decrease in EMC of wood species studied.

According to Tab. 3, cedroarana wood showed a higher reduction of EMC caused by the heat treatment than cedro-marinheiro did. The explanation for this behavior might be related to the level of crystallinity of wood cellulose after heating. In fact, as cedro-marinheiro has a greater specific gravity and thicker cell walls than cedroarana, this species might not reach a severe degree of relative crystalinization of wood cellulose after heat treatment at the range of temperatures studied (160 and 220°C) as cedroarana did.

Moreover, cedro-marinheiro wood contained an amount of holocellulose per unit of volume 24 % higher than that observed in cedroarana (Tab. 1), thus the amount of hemicelluloses still remaining in cedro-marinheiro cell walls after heating might keep part of its original hygroscopicity. Therefore, cedro-marinheiro wood did not present a higher reduction of EMC because it might still possess a greater amount of hemicelluloses not degraded by heat during the treatments at the range of temperatures applied, and a greater amount of not-esterificated hydroxyl groups are still available, in comparison with cedroarana wood. Thus, it is hypothesized, for the range of temperature studied, that although cedro-marinheiro presented a higher weight loss caused by heating, this reduction in mass was not enough to critically eliminate hemicelluloses and water-sorption hydroxyl groups, to further reduce the EMC.

CONCLUSIONS

In this study, heat treatment caused changes in wood color, weight and EMC, when compared with control wood. However, no changes in specific gravity were observed.

The increase in maximum temperature caused wood darkening and weight loss, as well as a reduction of the EMC. Cedro-marinheiro has presented the highest changes in color and weight as a function of heating, while cedroarana has shown the highest reduction in the EMC.

This study shows that heat treatment confers attractive color and lower hygroscopicity to wood, being a good way to add value to low market valued wood. Results also suggest that harder woods might posses a higher resilience to reduction of hygroscopicity, owing to a higher amount of water-sorption hydroxyl groups remaining in cell walls after heating.

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