Ciência da Madeira (*Brazilian Journal of Wood Science*)

6(3): 223-231, 2015
DOI: 10.12953/2177-6830/rcm.v6n3p223-231

Recebido: 04-12-2015 Aceito: 13-12-2015

Utilization of a thermomechanical process to enhance properties of hardwood used for flooring

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ABSTRACT The aim of this study was to evaluate the physical and mechanical properties of four thermomechanically treated tropical woods for the production of hardwood flooring. The following species were selected: cedrinho (*Erisma uncinatum* Warm.), angelim (*Hymenolobium* sp.), tachi preto (*Tachigali myrmecophila* Ducke) and mandioqueira (*Qualea paraensis* Ducke). Samples measuring 100 mm x 300 mm were thermomechanically treated in a hydraulic press at 145°C. Physical, mechanical and colorimetric properties were assessed. Thermomechanical treatment was efficient to decrease the moisture content and surface wettability of woods. Apparent density of angelim and mandioqueira woods increased significantly. The same woods presented the highest compression ratio. Wood color of all species became darker, mainly due to the reduction of lightness. Average values of mechanical properties increased in comparison to average values of reference.
Keywords: densification, flooring, tropical hardwood.

Introduction

The Amazon forest is the largest rainforest of the Earth, which covers 40% of the South American continent and has an enormous biodiversity. Nowadays, the production of flooring from tropical hardwood is concentrated in 12 to 14 wood species. Usually, these species have a great natural durability, high density and high mechanical properties. Therefore, there is a great exploration pressure on these species and for this reason it is extremely important to study lesser known wood species to be used to manufacture flooring. One possibility if bring low-density wood species, which do not have suitable properties for being used as flooring raw material. Nevertheless, these materials should be modified in order to meet the product's requirement such as dimensional stability, biological durability and mechanical properties.

In this context, the thermomechanical modification might be an excellent alternative to modify these wood species. Thermomechanical modification is a densification technique that combines heat treatment with mechanical compression. In this treatment, the densification occurs by cell wall buckling, which reduces void spaces volume (KUTNAR et al., 2009). However, in order to ensure suitable properties of the treated wood, the treatment should occurs at an ideal temperature, in which the amorphous polymers of wood (lignin and hemicelluloses) pass from the glassy state to the rubbery state (WOLCOTT et al., 1990; AKERHOLM; SALMÉN, 2004). It is defined as the glass transition temperature (Tg) and it varies with species and moisture content used. At this point, the wood can be compressed without occurrence of collapse in the cell wall structure. Previous studies using thermomechanical modification of tropical wood are recent and scarce as those made by Del Menezzi et al. (2014) and Arruda

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and Del Menezzi (2013). Taking in account these challenges, the project "Sustainable Model for the Brazilian Wood Flooring Production Chain" funded by International Tropical Timber Organization (ITTO) and Brazilian Government started in 2011 and covers the entire supply chain related to wood flooring.

The overall objective is to contribute to the sustainable use and to increase the efficiency in the use of forest resources, from forest to final product. Specifically, the present paper aimed at assessing physical and mechanical properties of woods used for flooring which were subjected to a thermomechanical modification process.

Material and Methods

Raw material

Wood from four species planted in a well-managed and legally established exploratory forest areas belonging to the Brazilian Government were selected: cedrinho (*Erisma uncinatum* Warm.) angelim (*Hymenolobium* sp.) tachi preto (*Tachigali myrmecophila* Ducke) and mandioqueira (*Qualea paraensis* Ducke). The boards were cut in 100 mm (width) x 300 mm (length) with thickness ranging from 20 mm to 25 mm. A square sample measuring 30 mm x 30 mm was to cut from each board to measure previously apparent density, equilibrium moisture content (EMC), wettability and color variables.

Thermomechanical Treatment

Preliminary tests were conducted and the temperature of 145°C was chosen to apply the treatment. Before the treatment, each board was carefully measured and weighted to determine initial apparent density. The nominal pressure was equivalent to 25% of perpendicular compression strength ($f_{c,90°}$) obtained from Serviço Florestal Brasileiro (2013), which meant 1.4 MPa for cedrinho, 2.8 MPa for angelim, 2.3 MPa for tachi preto and 3.6 MPa for mandioqueira. The internal temperature of samples was measured by a thermocouple inserted through a hole in wood. The samples were compressed until the internal temperature of wood reach around 100°C, which is approximately the glass transition temperature (Tg) characterized by the softening behavior of amorphous polymers. When the temperature of the polymer approaches Tg, the stiffness of the material decreases rapidly and the compression is facilitated. Each species has a unique behavior when subjected to this treatment. Therefore, the time required to reach 100°C was different for each species.

Another important fact observed during treatment was the automatic adjustment of the press. During pressing, as already mentioned, the heat promotes the softening of wood polymers and in consequence the compression of wood. Therefore, while wood is being compressed, the hydraulic press was readjusting pressure to keep it constant. It means that each press adjustment imparted a reduction of thickness in wood and this observation helped to control the pressing time.

When the material reached around 100°C and the press adjusted the pressure about three or four times, 50% of pressure was released. Five minutes later, 100% of pressure was released. The samples remained in hydraulic press for further five minutes without pressure and with constant temperature, characterizing a thermal treatment (Figure 1). This stage was important to help release compression stresses, which are inherent to any compressed material. After treatment, board thickness and mass were immediately determined.

The mass loss (ML, %) and the reduction in thickness (RT, %) were measured according to Equations 1 and 2. These measurements were done immediately after the thermomechanical treatment and every week for there weeks, where the wood samples were kept in a conditioning room (20°C ± 3 °C and 65% ± 1% RH).

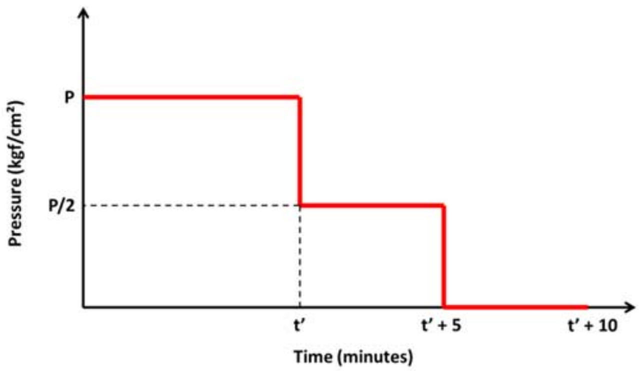

Figure 1. Diagram of the thermomechanical treatment used to modify the wood.

$$ML = \left(\frac{M_i - M_f}{M_i}\right) \times 100 \quad (1)$$

$$RT = \left(\frac{T_i - T_f}{T_f}\right) \times 100 \quad (2)$$

Where: M_i and T_i = mass and thickness initial; M_f and T_f = mass and thickness final.

Physical Properties

The EMC and the density were measured before and after the thermomechanical treatment. For density, samples were measured with a digital caliper at four points and the mass was obtained with a digital balance. For the EMC measurement the samples was dried in oven with air circulation at constant temperature of 103±2 °C until constant mass. After treatment, density was monitored at conditioning room and the EMC was measured two months after the treatment. Thickness and volumetric swelling, and water absorption were also measured. The swelling samples were measured with digital caliper at four points in thickness and three points in width and length. The water absorption samples were evaluated with digital scale. Samples were kept immersed in distilled water and the measurements were done at two, 24, 96 and 144 hours, after immersion.

Wettability and color measurement

The wettability was evaluated using a goniometer (model Krüss DSA30). The drop volume used was around 10 μl of distilled water and the contact angle was measured for two minutes by sessile droplet method, totaling 120 measurements. Data obtained at 60 seconds was used to run the statistical analyzes. The color measurement was performed using the CIEL*a*b* system (L*, a*, b*, C and h*). Color parameters were measured in a X-Rit spectrophotometer (model Color Eye XTH-X) using an illuminant D65 with angle of 10°. Color difference (ΔE) was determined by Equation 3. Fourteen measurements per species were performed. Natural wood color was determined using a method established by Camargos; Gonçalez (2001).

$$\Delta E = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2} \quad (3)$$

Where: ΔL*, Δa*, Δb* = variation between the treated and untreated samples.

The average of the samples was analyzed by Paired t-test.to identify differences in wettability and color before and after the treatments. Significant differences between the species were analyzed by ANOVA and Tukey, post-hoc test.

Mechanical properties

The following mechanical properties were assessed according to the standard ASTM D-143 (2009): static bending (E_M and fm), Janka hardness (f_H) and parallel compression strength ($f_{c,0°}$).These tests were conducted at an universal testing machine (EMIC, model DL30000) with a maximum load capacity of 300 kN. For the static bending, the samples were cut at dimensions of 35 mm x 300 mm and for hardness the dimensions were 33 mm x 50 mm. For $f_{c,0°}$ the length ranged according the thickness. Using the same width of static bending samples (35 mm) and the same slenderness used at the standard, the length was 90 mm for cedrinho, 70 mm for angelim and mandioqueira and 80 mm for tachi preto.

Results and Discussion

Treatment Characteristics and Wood Densification

Figure 2 shows that in all species heating behavior was similar. In the first stage, the heating of boards is fast and its reached at 100°C after around 5-10 minutes (Table 1). After this fast heating step, the internal temperature increased gradually up to reach a maximum of 136°C in mandioqueira wood samples.

Nevertheless, a pattern between the species was observed. The first adjustment of the press occurred between 80°C and 95°C, which indicates the glass transition temperature of wood (Table 1). From this stage, the board is compressed and its thickness tends to decrease. Regarding the time of treatment, the longest treatment was 28 minutes and 35 seconds, which was required for the treatment of tachi preto wood. On the other hand, the time required by angelim and mandioqueira woods was similar: 21 minutes and 55 seconds and 22 minutes and 55 seconds, respectively. Therefore, this treat ment minutes and 55 seconds, respectively. Therefore, this treatment can be considered fast in comparison to those currently used to treat wood, which require hours of even days -

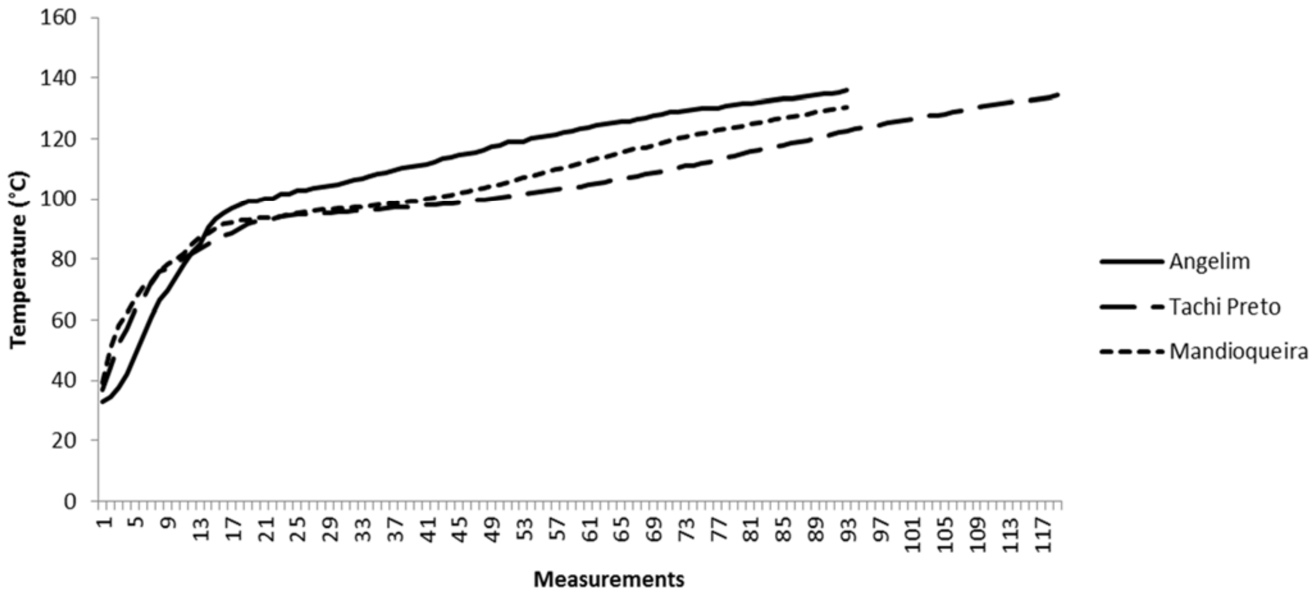

Figure 2. Internal temperature curves obtained during the thermomechanical treatment of the woods.

Table 1. Time and internal temperatures reached during thermomechanical treatment.

	Angelim		**Tachi preto**		**Mandioqueira**	
Steps	**Time (min)**	**Temp. (°C)**	**Time (min)**	**Temp. (°C)**	**Time (min)**	**Temp. (°C)**
Start	0:00	32.8	0:00	37.0	0:00	39.4
1st press adjustment	03:07	90.0	06:00	94.9	02:15	79.9
2nd press adjustment	05:20	101.6	--	--	04:47	93.8
3rd press adjustment	--	--	--	--	09:29	99.9
Release 50% of pressure	11:55	117.0	18:35	110.0	12:55	103.0
Release 100% of pressure	16:55	128.0	23:35	124.8	17:55	122.0
Final	21:55	135.0	28:35	133.0	22:55	136.0

such as ThermoWood.

Before the thermomechanical treatment, wood density was 0.70 g.cm^{-3} for cedrinho, 0.71 g.cm^{-3} for angelim, 0.73 g.cm^{-3} for tachi preto and 0.81 g.cm^{-3} for mandioqueira. Table 2 illustrates that only wood density of angelim and mandioqueira woods increased significantly after the thermomechanical treatment. Density increased from 0.71 g.cm^{-3} to 0.79 g.cm^{-3} in angelim wood and from 0.81 g.cm^{-3} to 0.85 g.cm^{-3} in mandioqueira wood. The effect of slight densification can be verified in the low values of the reduction in thickness (RT). Usually, the RT values are close to the percentage of the parallel compression strength, which was applied with 25%. The highest value of RT was found for angelim. Average value of RT for angelim wood was 17.42% immediately after the treatment and 16% after the period in conditioning room (Table 3). Mandioqueira wood presented the second highest value of $RT_{(f)}$, 12.41%.

The values measured after acclimatization ($RT_{(f)}$) were lower the $RT_{(at)}$ especially due to the gradual increase in thickness, which represents the natural trend of compressed material to return own initial thickness and due to the absorption of humidity. The difference between $RT_{(at)}$ and $RT_{(f)}$ was significant only in tachi preto and mandioqueira wood samples, which means that for these species the thickness returned at greater level (Table 3). The densification of wood may be explained by the viscoelastic behavior of amorphous polymers of wood, which change drastically to the viscous state when they reach the Tg (glass transition temperature). According to other studies (ARRUDA; DEL MENEZZI, 2013, BEKTHA et al. 2012), it is well-known that the RT is significant affected by the increment of temperature and time of treatment. Thus, the temperature used in this treatment, 145°C, may not be sufficient to results in a higher densification in cedrinho and tachi preto wood.

Table 2. Statistical analysis for apparent density (D_{ap}), comparing before treatment values with after treatment and before treatment values with final values.

Property	Cedrinho	Angelim	Tachi preto	Mandioqueira
	g.cm^{-3}			
$D_{ap\,(bt)}$	0.70a^{NS}	0.71a**	0.73a^{NS}	0.81a**
$D_{ap(at)}$	0.68b^{NS}	0.78ab**	0.72ab^{NS}	0.84a**
$D_{ap(bt)}$	0.70^{NS}	0.71**	0.73^{NS}	0.81**
$D_{ap(f)}$	0.70b^{NS}	0.79ab**	0.72ab^{NS}	0.85a**

(bt) = before treatment; (at) = after treatment; (f) = final. **,^{NS} significant and non-significant respectively in T-Test at 0.05 level. Same letters indicate that there is no difference between the species analyzed within the property by Tukey Test at 5% of probability of error.

Table 3. Statistical analysis for reduction of thickness (RT), mass loss (ML) and equilibrium moisture content (EMC).

Property	Cedrinho	Angelim	Tachi preto	Mandioqueira
	%			
$RT_{(at)}$	8.23c^{NS}	17.42a^{NS}	9.41c**	13.58b**
$RT_{(f)}$	8.12c^{NS}	16.00a^{NS}	8.27c**	12.41b**
$ML_{(at)}$	11.05a**	8.88b**	11.18a**	10.94a**
$ML_{(f)}$	8.98a**	6.40b**	6.67b**	6.17b**
$EMC_{(bt)}$	12.48a**	9.49b**	10.84b**	10.46b**
$EMC_{(f)}$	6.40c**	5.36d**	9.36a**	8.40b**

(bt) = before treatment; (at) = after treatment; (f) = final. **,^{NS} significant and non-significant respectively in T-Test at 0.05 level. Same letters indicate that there is no difference between the species analyzed within the property by Tukey Test at 5% of probability of error.

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The mass loss (ML) measured immediately after the treatment ranged from 8.88% to 11.18% (Table 3). After acclimatization, ML decreases and ranged from 6.17% to 8.98%. ML decreases because the wood reaches the equilibrium with the environment and adsorbs water. The $EMC_{(bt)}$ ranged from 9.49% to 12.48% and the $EMC_{(f)}$ ranged from 5.36% to 9.36% (Table 3). The statistical analysis showed that the difference between $EMC_{(bt)}$ and $EMC_{(f)}$ was significant, indicating that treatment was responsible to decrease the EMC. Many authors (BOONSTRA; TJERDSMA, 2006; ROWELL, 1981; KOCAEFE et al., 2008) mentioned that hemicelluloses are the most heat sensitive wood polymer and their degradation directly affects wood water adsorption ability.

Hygroscopicity and Dimensional Stability

It is well-known that all densified materials after compression tend to release the stresses that are accumulated within the material (Del Menezzi et al. 2009). These stresses are most evident when the material is immersed in water. Thus, the thickness swelling (TS) is the sum of the natural swelling of the wood and the swelling caused by release of the compressive stresses.

At first 2 hours of immersion, the values of TS, VS and WA are lower and increased over the time (Table 4). Among the species, cedrinho presented the lowest values of TS up to 144 hours of immersion. Note that the TS value is directly linked to RT, since one of the components of TS is caused by the compression in the material thickness direction. Thus, Table 3 shows that the lowest value of RT, 8.12% for cedrinho, is connected to the lower TS, 2.99%.

Mandioqueira wood presented the highest value (13.53%) of VS for 144h of immersion. On the other hand, cedrinho wood presented the lowest average value (5.45%). The WA 2h ranged from 3.02% for angelim to 5.04% for tachi preto. After 144 hours, WA increased, ranging from 32.99 at cedrinho to 45.49% at tachi preto, but the differences between the means were not statistically significant by Tukey test (Table 4).

Table 4. Results for dimensional stability parameters, thickness swelling (TS), volume swelling (VS) and water absorption (WA).

Property	C*	A	T	M
	%			
TS 2h	0.56b	1.76a	2.12a	1.49a
VS 2h	0.96b	2.29a	2.88a	2.16a
WA 2h	3.23b	3.02b	5.04a	3.77ab
TS 24h	1.71b	6.90a	5.63a	5.50a
VS 24h	3.25b	8.54a	7.26a	8.26a
WA 24h	13.13b	14.35b	19.55a	14.41b
TS 96h	2.70b	10.02a	7.22a	8.52a
VS 96h	4.96b	12.19a	8.90ab	12.73a
WA 96h	27.59b	29.33b	39.86a	30.22b
TS 144h	2.99c	10.91a	7.45b	9.02ab
VS 144h	5.45b	13.28a	9.23ab	13.53a
WA 144h	32.99a	34.54a	45.49a	33.91a

Same letters indicate that there is no difference between the species analyzed within the property by Tukey Test at 5% probability of error. * C = cedrinho; A = angelin; T = tachi preto; M = mandioqueira.

Color Variation and Wettability

Before the thermomechanical treatment, wood color was reddish-brown, yellow-brownish, olive yellow and olive brown, respectively for cedrinho, angelim, tachi preto and mandioqueira. According to Camargos and Gonçalez (2001), clearer wood presents the parameter L* higher than 56.

After the thermomechanical treatment, L* and b* decreased significantly, resulting in a reddish and brownish appearance of wood. The decrease of L* in wood heat treatments was reported in other studies (ESTEVES et al., 2008; CHARRIER et al., 2002). These studies attributed the wood darkening to the oxidation of some substances on wood surface. Average values of b* decreased after the thermomechanical treatment, except in agelim wood. This behavior corroborates with the wood darkening.

The increasing of a* paramenter directly affects wood darkening. Significant increase of a* was observe only for angelim and mandioqueira woods. In heat-treated woods is also expected the decrease of chroma (C) that is, the color of the wood becomes less saturated. This occurred only in cedrinho and tachi preto wood samples. The values of hue (h*) decreased, which confirms the wood darkening. Increase of h* results in wood with yellow tones, while decrease of h* results in wood with red tones. In this study, all species presented a decrease of h*, which confirms the tendency of reddish tones in heat-treated woods. Cedrinho wood had the highest variation of color (ΔE), 19.21, followed by tachi preto wood, 10.25.

In general, the contact angle at 60 seconds (Table 6) increased after the thermomechanical treatment. This hydrophobic behavior of wood occurs when this is subjected to thermal treatment above the glass transition temperature where a conformational rearrangement of wood polymers occurs, as a result of the lignin plastification. Reducing the wettability is important when the product requires that the surface has low interaction with water, as is the case of natural hardwood floors. But in some cases, where wood surface requires be sealed, painted or varnished this reduction of wettability can be a problem.

Mechanical Properties

The values of reference for mechanical properties were obtained from Serviço Florestal Brasileiro (2013). These values are only a general reference to the species obtained in previous studies conducted in the Brazilian Forest Products LaboratoryThe modulus of elasticity (E_M) ranged from 10483 MPa for cedrinho to 19988 MPa for mandioqueira (Table 7). Only in treated samples of cedrinho the value of E_M were lower than

Table 5. Results for color measurement analyzing the condition of the parameter before and after treatment and the total variation of color.

Parameters	Cedrinho	Angelim	Tachi preto	Mandioqueira
L* before	51.17**	60.60**	58.18**	58.48**
L*after	32.35**	53.42**	51.15**	51.03**
a* before	14.07^{NS}	14.45**	9.21^{NS}	13.71**
a* after	13.88^{NS}	15.52**	10.16^{NS}	16.47**
b* before	25.71**	31.44^{NS}	33.13**	29.88**
b* after	22.36**	31.35^{NS}	30.78**	28.20**
C before	29.31**	34.62^{NS}	34.41**	32.93^{NS}
C after	26.32**	34.99^{NS}	32.44**	32.79^{NS}
h* before	61.32**	65.29**	74.61**	65.48**
h* after	58.21**	63.61**	71.85**	59.87**
ΔE	19.21a	8.37b	10.25b	9.29b

^{**,NS}significant and non-significant respectively in T-Test at 0.05 level. Same letters indicate that there is no difference between the species for ΔE by Tukey Test at 5% probability of error.

Table 6. Results for wettability analyzing the condition of the contact angle before and after treatment.

Contact Angle	Cedrinho	Angelim	Tachi preto	Mandioqueira
CA before	80.52a^{NS}	44.82b^{NS}	55.37b^{NS}	67.84a^{NS}
CA after	95.64a^{NS}	76.00a^{NS}	93.41a^{NS}	89.25a^{NS}

^{**,NS} significant and non-significant respectively in T-Test at 0.05 level. Same letters indicate that there is no difference between the species for AC by Tukey Test at 0.05 level.

reference. The others values increased after treatment. For angelim wood the values increased in 65.14%, in tachi preto increased 36.83% and in mandioqueira the values were 59.12% higher. The modulus of rupture (f_m) had similar behavior to E_M. Only cedrinho had value of f_m lower than reference. Values of f_m ranged from 62.24 MPa for cedrinho to 160.78 MPa for angelim (Table 7). FM increased 43.59% in angelim, 30.68% in tachi preto and 48.02% in mandioqueira.

Table 7. Results for mechanical properties of samples after the thermomechanical treatment.

Property	*C	A	T	M
E_M (MPa)	10,483	19,609	15,039	19,998
Reference*	10,795	11,874	10,991	12,561
f_M (MPa)	62.24	160.78	137.22	159.34
Reference*	87.54	111.97	105.00	107.65
Hardness (N)	2,613	6,943	6,611	5,796
Reference*	3,864	6,325	5,511	4,766
$f_{c,0°}$(MPa)	44.91	59.32	67.52	72.37
Reference*	49.75	56.33	56.72	58.88

Note: * Serviço Florestal Brasileiro (2013). * C = cedrinho; A = angelin; T = tachi preto; M = mandioqueira.

This result is very important for the strength of treated wood. It is well-known that, usually, in heat treatments, the wood loses strength due to depolymerization reactions, especially hemicelluloses which are less stable than lignin and are more sensitive to temperature. Changes in the composition or loss of hemicelluloses may contribute significantly to changes in the strength properties of the treated wood to high temperatures. Thus, as the temperature used was not very high (145°C) there was no loss of stiffness and mechanical strength of the wood. On the contrary, these properties were slightly improved even though there has not been a great densification of wood.

Janka hardness (H) measures the resistance to penetration of objects in the wood surface and is an important property for hardwood floors. Values ranged from 2659.12 N for cedrinho to 6942.68 N for angelim (Table 7). Treatment resulted in increased in H of 9.76% for angelim, 19.95% for tachi preto and 21.60% for mandioqueira. Values in cedrinho decreased, as E_M and f_m. Table 7 shows the $f_{c,0°}$ values. Except for cedrinho wood, the values of treated samples were higher than reference. The improvement was about 5.31% for angelim, 19.02% for tachi preto and 22.90% for mandioqueira.

Conclusion

The thermomechanical treatment increased significantly the density of angelim and mandioqueira wood. Likewise, these species presented the highest values of thickness reduction. The others species were not significantly densified, probably because a relatively low temperature (145°C) was used. For all species, Thermomechanical treatment ensured good dimensional stability, as perceived by the thickness swelling lower than the compression rate. This meant that the compressed shape was retained in part after immersion in water. Cedrinho had the most significant reduction of EMC, 48.7%, followed by angelim with 43.7%. The color of all species became darker, with predominantly reddish and brownish tones. This is ideal for clearer woods when wishes to imitate tones of darker woods. The wettability of woods was reduced and it was represented by the increase in CA after treatment. Except for cedrinho, all species had increase in mechanical properties after thermomechanical treatment. Furthermore, the industry can control the colors of the floor, making a more homogeneous product, with better mechanical properties.

Acknowledgements

To the International Tropical Timber Organization that funded the project “Sustainable Model for the Brazilian Wood Flooring Production Chain – PD 433/06”.

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