PHYSICAL AND CHEMICAL CHANGES IN JUVENILE AND MATURE WOOD OF *SHIZOLOBIUM PARAHYBA* CAUSED BY THERMAL MODIFICATION

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In the Brazilian Atlantic Forest, the pioneer species *Schizolobium parahyba*, known as guapuruvu, is a fastgrowing tree with potential for ecological restoration in forest management projects. However, an undesirable feature of this wood is the pronounced presence of juvenile wood leading to poor dimensional stability. This study evaluates the effect of various thermal modification intensities on the physicochemical properties of *S. parahyba* juvenile and mature wood. Boards sawn from 15-year-old trees were thermally modified at three different temperatures (180, 200 and 220 °C). Thermal modification decreased the density (up to 9%), moisture content (up to 9.1%) and volumetric swelling (up to 54.1%). For chemical properties, when the wood was submitted to thermal modification at 220 °C, significant increase was detected in the respective extractive (up to 286.5%) and lignin content (up to 41.7%), and reduction in the holoceluloses (up to 25.0%). It was demonstrated that basic density was not the most suitable parameter for evaluating the quality of thermally modified wood. The influence of thermal modification was equal in juvenile and mature wood.

Keywords: Guapuruvu, density of wood, dimensional stability, holocelluloses content, thermally modified wood

INTRODUCTION

Schizolobium parahyba (guapuruvu), a pioneer native species of Brazil, has high potential for ecological restoration projects in the Atlantic Forest (Engel & Parrota 2001). It represents fast-growing trees, featuring light colored wood (Richter et al. 1974, Trianoski 2010, Athanásio-Heliodoro 2015). Its commercial plantation is inexpressive, although very similar to *S. amazonicum* (paricá), which represents a large part of reforestation area (90,000 ha) in the northern region of Brazil (IBÁ 2016).

Variation in the properties of wood occurs due to several factors, such as tree species, silviculture and especially the wood anatomy. Juvenile wood is also called pith wood or core wood which is formed near the tree center, displaying a cylindrical shape with a diameter almost uniform from the base to the top of the stem (Zobel & Van Buijtenen 1989, Bao et al. 2001, Calonego et al. 2005). It differs from mature wood (outer wood) in chemical properties, fiber length, density and dimensional stability (Zobel & Van Buijtenen 1989, Bao et al. 2001, Calonego et al. 2005, 2014, Lobão et al. 2012, Severo et al. 2012, 2016,). The fiber length in juvenile wood obtained from 15-year-old *S. parahyba* is 1.196 mm, whereas in mature wood is 1.492 mm. Anatomical characterisation shows that the juvenile wood is confined to a distance of 109.1 mm from the pith (Athanásio-Heliodoro 2015).

Richter et al. (1974) reported that the basic density of *S. parahyba* varies from 0.24 to 0.27 g cm⁻³. The basic density, maximum volumetric, and tangential and radial shrinkages of this wood are 0.39 g cm⁻³, 10.03%, 8.31% and 1.72%, respectively (Bortoletto Jr & Belini 2002). According to Trianosky (2010), the basic density of this wood is 0.39 g cm⁻³, while the respective extractives, insoluble lignin and holocelluloses contents are 8.65%, 21.01% and 69.39%, respectively.

The density at 12% moisture content in juvenile wood of *S. amazonicum* ranges between 0.312 and 0.544 g cm⁻³, whereas in mature wood, between 0.535 and 0.623 g cm⁻³ (Lobão et al. 2012). This physical property, maximum volumetric, and tangential, radial and longitudinal shrinkages in the juvenile wood of *S*. *parahyba* are 0.289 g cm⁻³, 10.66%, 6.86%, 2.50% and 0.36%, whereas in mature wood, they are 0.375 g cm⁻³, 12.35%, 7.43%, 2.88% and 0.36%, respectively (Athanásio-Heliodoro 2015).

Heat treatment of wood at high temperatures (~200 °C) or thermal modifications cause degradation of hemicelluloses and amorphous region of cellulose, contributing towards crystallinity of the polymer (Bhuiyan et al. 2000, Mburu et al. 2007, Esteves & Pereira 2009, Bächle et al. 2010, Severo et al. 2012, 2016, Umar et al. 2016,). In addition, a cross-linkage between the lignin and the polymer occurs, which causes increase in biological resistance, decrease in hygroscopicity and improvement in dimensional stability (Mburu et al. 2007, Esteves & Pereira 2009, Bächle et al. 2010, Calonego et al. 2012, 2014, Severo et al. 2012, 2016, Umar et al. 2016). Moreover, the juvenile wood of both Pinus elliottii var. elliottii as Eucalyptus grandis is characterised by high lignin content when compared with mature wood, and consequently it presents an adverse effect on the chemical modification (Severo et al. 2012, Calonego et al. 2014). Hevea brasiliensis presents opposite behavior due to sugar content in the cell lumen of mature wood (Severo et al. 2016).

The aim of this study was to evaluate the effects of various intensities of thermal modification on the physicochemical properties of *S. parahyba* juvenile and mature wood.

MATERIALS AND METHODS

Collection of materials

Wood samples were obtained from four 15-yearsold *S. parahyba* (Fabaceae - Caesalpinioideae) trees, from the Lageado Farm of the State University of São Paulo, located in Botucatu (22° 50' S, 48° 24'W), São Paulo, Brazil. The trees were felled and sectioned into 2.5 m logs. The logs with diameters between 33 and 38 cm were cut into flat-sawn boards. Later, the boards were dried down to 10.0% moisture content in a dry kiln.

Thermal modifications of boards

A dried board from each tree was planed to 32-mm thickness and sawed into smaller pieces measuring 0.60 m in length. Regions with cracks and knots were discarded. A piece measuring $32 \times 180 \times 600$ mm in size (tangential x radial x longitudinal) from each board was kept in

its original condition (unmodified wood), and the other three pieces were thermally modified (thermally modified wood).

Parts of pieces were placed in an electric oven with a programmable controller and thermally modified in the Laboratory of Wood Drying and Preservation from UNESP, Botucatu, São Paulo, Brazil. The thermal modification started at an initial temperature of 100 °C over a period of 14 h and then increased at 1.34 °C min⁻¹ up to 180 °C, and maintained over a period of 2.5 h under air atmosphere condition (Severo & Calonego 2011). Same procedure was performed for thermal modification at final temperature of 200 °C and 220 °C. After thermal modification, the oven was turned off, and the wood pieces were conditioned in natural climate until they reached 30 °C.

Standard specimens were sawn from all the pieces (unmodified and thermally modified) according to the method in ABNT NBR-7190 (1997) for physical characterisation of juvenile and mature wood. According to Athanásio-Heliodoro (2015), the juvenile wood zone was defined as being located up to 109 mm from the pith, as identified by the fiber length of *S. parahyba*. Specimens of wood measuring $20 \times 30 \times 50$ mm in size (tangential x radial x longitudinal) were removed approximately 20 mm from the pith and 20 mm from the bark. Each specimen was cut perfectly to produce tangential, radial and longitudinal directions and was then measured.

Although the ABNT NBR-7190 (1997) standard states that the necessary number of specimens to characterise physical properties of wood is six, twelve specimens were obtained from five boards to characterise each thermal modification (unmodified wood and thermally modified wood at 180 °C, 200 °C and 220 °C), aggregating 48 specimens.

Physical properties of the wood

The unmodified and thermally modified specimens were placed in an oven at 103 ± 2 °C and maintained until they reached 0% moisture content. Subsequently, the specimens were placed in a climatic chamber adjusted to 21 °C and 65% relative humidity (RH) until they reached equilibrium moisture content. The samples were then weighed, and their dimensions were measured by using a 0.01-g accuracy balance and a 0.01-mm accuracy micrometer. Following these measurements, the specimens were submerged in water until the cell walls were completely saturated. Then the dimensions of specimens were again measured and weighed. The evaluation of basic density, density in absolutely dry condition, density at equilibrium moisture content, equilibrium moisture content and maximum swellings were performed according to ABNT NBR-7190 (1997) standard.

Chemical properties of the wood

Samples of unmodified and thermally modified mature and juvenile wood were transformed into chips and then milled. The material used for chemical analysis was classified between 40 and 60 mesh. The extractives content was determined by extraction sequences with ethanol/toluene 1/2 (v/v), and ethanol and hot water (TAPPI T 12 wd-82 1999). The acid-insoluble Klason lignin (TAPPI T 222 om-98 1999), and holocelluloses (ASTM D-1104 1978) contents were determined in extractive-free wood.

To evaluate differences in physical and chemical properties, a Kolmogorov-Smirnov's normality test at 5% significance was performed. All variables had normal distribution. A parametric two-way test (ANOVA) at 5% significance was then performed taking into account the type of wood (two levels) and the thermal treatment (four levels), as well as Tukey's test at 5% significance for comparison of the means.

RESULTS AND DISCUSSION

Physical properties of thermally modified wood The basic density of unmodified *S. parahyba* was 0.235 g cm⁻³ for juvenile and 0.246 g cm⁻³ for mature wood. The density of these kinds of wood at equilibrium moisture content was 0.274 and 0.286 g cm⁻³, respectively (Table 1). The results of this study are similar to those presented by Bortoletto Jr. and Belini (2002), Richter et al. (1974) and Trianoski (2010). The densities of unmodified mature wood were not different than those of juvenile wood. This behavior is similar with the one showed by Athanásio-Heliodoro (2015), who related the non-influence of the type of wood on density of the species studied.

Basic density of *S. parahyba* wood did not change significantly by thermal modification, although it promoted a decrease in the oven-dry weight of specimens. However, it was found that the saturated volume of specimens presented a steep decrease with thermal modification. This fact is explained by the better influence of thermal modification on dimensional stability of wood than on weight loss. Calonego et al. (2012) reported that basic density is not the most suitable parameter for evaluating the quality of thermally modified wood.

Thermal modification decreased density significantly up to 8.8% and 9.1% and moisture content up to 9%, in juvenile and mature wood. Thermal modification at 220 °C showed the greatest significant reductions in physical property. The juvenile and mature wood presented density at 9% of 0.250 g cm⁻³ and 0.260 g cm⁻³, respectively. The reduction in density of thermally modified wood can be explained by the changes in chemical composition due to degradation of extractives and cell wall compounds, mainly sugars from hemicelluloses (Esteves & Pereira 2009, Severo et al. 2016). The results were consistent with those obtained by Severo et al. (2012, 2016) and Calonego et al. (2014), who thermally modified the juvenile and mature wood from other species.

Table 1 shows that the respective equilibrium moisture contents (EMC) in juvenile and mature wood of unmodified *S. parahyba* were only 9.2% and 8.9% after acclimatization at 21°C and 65% RH. According to USDA (1999) these lower values in EMC can be attributed to the phenomenon known as hysteresis.

The EMC of mature wood was significantly smaller than that of juvenile wood. This is a result of the lower cell wall thickness of fibers from juvenile wood, which facilitates the diffusion of moisture in wood (Zobel and Van Buijtenen, 1989Calonego et al. 2005, Severo et al. 2012).

The current study also showed that thermal modification from 180 °C to 220 °C promoted significant EMC reductions up to 51.1% and 42.7% in juvenile and mature wood from *S. parahyba*. Similar improvements were cited for thermally modified wood of other species (Mburu et al. 2007, Bächle et al. 2010, Severo et al. 2012, Calonego et al. 2012, 2014,). The greatest weight loss in mature wood due to thermal modification explains the smaller reduction in EMC. Chemical changes lead to higher lignin reactivity with formation of crosslinks, leading to less absorption of water, which explains the decrease of EMC (Mburu et

1.5 NS

13.3 *

			Basic density (g cm ⁻³)								
Thermal	N	Juvenile wood				Mature we	bod				
modification	1	CV	Average	Red. or (Inc.) (%)	CV	Average	Red. or (Inc.) (%)	Red. or (Inc.) (%			
Unmodified	12	9.6	0.235 ab		12.0	0.246 ab		(4.7) ^{NS}			
180 °C	12	4.5	0.246 a	(4.7)	7.1	0.259 a	(5.3)	(5.3) ^{NS}			
200 °C	12	10.9	0.239 b	(1.7)	6.5	0.234 b	4.9	2.1 ^{NS}			
220 °C	12	11.6	0.232 b	1.3	4.3	0.240 b	2.4	(3.4) ^{NS}			
			Density	v at 9% moistu	re conte	ent - 21 ºC e (65% RH (g cn	n ⁻³)			
Thermal	N -		Juvenile w	ood		Mature we	bod	D 1			
modification		CV	Average	Red. or (Inc.) (%)	CV	Average	Red. or (Inc.) (%)	Red. or (Inc.) (%			
Unmodified	12	9.6	0.274 ab		12.0	0.286 ab		(4.4) ^{NS}			
180 °C	12	4.6	0.281 a	(2.6)	5.8	0.301 a	(5.2)	(7.1) ^{NS}			
200 °C	12	10.8	$0.267 \mathrm{\ bc}$	2.6	6.6	0.262 bc	8.4	$1.9 ^{\text{NS}}$			
220 °C	12	11.7	0.250 c	8.8	4.5	0.260 c	9.1	(4.0) ^{NS}			
			Equi	librium woistu	re Conte	ent at 21 ºC o	e 65% RH (%)			
Thermal	N		Juvenile w	ood		Mature we	bod				
modification	N	CV	Average	Red. (%)	CV	Average	Red. (%)	Red. or (Inc.) (%			
Unmodified	12	1.9	9.2 a		1.3	8.9 a		3.3 *			
180 °C	12	3.1	$7.7 \mathrm{~b}$	16.3	1.5	$7.7 \mathrm{ b}$	13.5	0.0 NS			

Table 1.	Densities and equilibrium moisture content of thermally modified Schizolobium parahyba juvenile
	and mature wood

where N - repeat number of samples, CV - coefficient of variation, Red. – reduction, Inc. – increase. RH – relative humidity; average followed by the same letters in same column denote significant difference by Tukey test at probability 95% between thermal modifications; * within rows denote significant difference by F test at probability 95% between type of wood; same letters in same column and ^{NS} within rows denote non-significant difference

5.6

7.7

6.7 c

5.1 d

28.3

51.1

al. 2007, Esteves and Pereira 2009, Bächle et al. 2010).

4.7

3.8

6.6 c

4.5 d

12

12

The volumetric, tangential, radial and axial swelling in juvenile wood from unmodified *S. parahyba* was 9.67, 6.63, 2.45 and 0.40%, respectively. In mature wood, the respective swellings were 10.26, 7.26, 2.49 and 0.30%, respectively (Table 2). The axial swelling of mature wood from unmodified material is different from that of juvenile wood, whereas other swellings are not different in both types of wood. It was found that the axial swelling of juvenile wood was greater (25.0%) than mature wood. The results were consistent with previous studies on the swellings of unmodified *S. amazonicum* and

dimensional stability of unmodified *S. parahyba* (Bortoletto Jr. and Belini 2002, Melo et al. 2013, Athanásio-Heliodoro 2015). Several authors explained that longitudinal swelling can be greater in juvenile wood because the wood has greater microfibril angles when compared with mature wood (Zobel & Van Buijtenen 1989, Bao et al. 2001).

24.7

42.7

The effect of thermal modification on dimensional stability of *S. parahyba* wood is shown in Table 2. The results show that juvenile wood thermally modified from 180 °C to 220 °C had significant reductions of up to 54.1, 55.2, 46.9, and 67.5% in volumetric, tangential, radial and axial linear swellings, when compared with

200 °C

220 °C

		Maximum volumetric swelling (%)							
Thermal	Ν		Juvenile W	lood		Mature W	lood	- Red. or (Inc.) (%)	
modification	1	CV	Average	Red. or (Inc.) (%)	CV	Average	Red. or (Inc.) (%)		
Unmodified	12	15.0	9.67 a		14.0	10.26 a		(6.1) ^{NS}	
180 °C	12	3.5	9.07 b	6.2	4.6	9.11 b	11.2	(0.4) ^{NS}	
200 °C	12	4.7	7.34 с	24.1	5.6	7.08 с	31.0	3.5 ^{NS}	
220 °C	12	3.8	4.44 d	54.1	13.5	4.99 d	51.4	(12.4) ^{NS}	

Table 2. Swellings of thermally modified Schizolobium parahyba juvenile and mature wood

		Maximum tangential swelling (%)							
Thermal	Ν	Juvenile wood				Mature w	D I		
modification	1	CV	Average	Red. or (Inc.) (%)	CV	Average	Red. or (Inc.) (%)	- Red. or (Inc.) (%)	
Unmodified	12	18.3	6.63 a		19.4	7.26 a		(9.5) ^{NS}	
180 °C	12	8.4	$6.05 \mathrm{b}$	8.8	7.0	6.31 b	13.1	(4.3) ^{NS}	
200 °C	12	5.7	5.26 с	20.7	9.7	5.05 c	30.4	4.0 ^{NS}	
220 °C	12	8.0	2.97 d	55.2	16.4	3.30 d	54.5	(11.1) ^{NS}	

		Maximum radial swelling (%)							
Thermal	Ν	Juvenile wood				Mature wo	od		
modification	1	CV	Average	Red. (%)	CV	Average	Red. (%)	— Red. or (Inc.) (%)	
Unmodified	12	55.5	2.45 a		12.0	2.49 a		(1.6) ^{NS}	
180 °C	12	19.9	2.54 a	(3.7)	7.9	2.34 a	6.0	7.9 ^{NS}	
200 °C	12	7.2	$1.77 \mathrm{\ b}$	27.8	10.9	1.82 b	26.9	(2.8) ^{NS}	
220 °C	12	12.4	1.30 c	46.9	9.1	1.50 c	39.8	(15.4) ^{NS}	

			Maximum longitudinal swelling (%)								
Thermal	Ν		Juvenile wo	od		Mature wo					
modification	1	CV	Average	Red. (%)	CV	Average	Red. (%)	— Red. or (Inc.) (%)			
Unmodified	12	20.2	0.40 a		13.8	0.30 a		25.0 *			
180 °C	12	33.0	0.31 b	22.5	32.7	0.30 a	0.0	3.2 ^{NS}			
200 °C	12	27.6	0.20 c	50.0	27.7	0.11 b	63.3	45.0 *			
220 °C	12	32.7	0.13 d	67.5	33.2	0.13 b	56.7	0.0 ^{NS}			

where N - repeat number of samples, CV - coefficient of variation, Red. – reduction, Inc. – increase; average followed by the same letters in same column denote significant difference by Tukey test at probability 95% between thermal modifications; * within rows denote significant difference by F test at probability 95% between type of wood; same letters in same column and ^{NS} within rows denote non-significant difference

unmodified wood. Thermally modified mature wood under the same temperature presented reductions of up to 51.4, 54.5, 39.8 and 56.7% in the respective swellings.

Improvement of dimensional stability in thermally modified wood is a result of the increase

in crystallinity and the width of crystallites due to degradation of hemicelluloses and free hydroxyl groups in the amorphous region of cellulose, and cross-linking of polymers of wood (Bhuiyan et al. 2000, Esteves and Pereira 2009, Bächle et al. 2010, Severo et al. 2012, 2016).

The influence of thermal modification on juvenile wood from S. parahyba was equal to mature wood. However, thermally modified Pinus and Eucalyptus wood presented an adverse effect on the modification of juvenile wood because of their high lignin content, compared with mature wood (Severo et al. 2012, Calonego et al. 2014). The results presented in this study also differed from Severo et al. (2016) who concluded that the influence of thermal modification at 220 °C on the properties of mature wood from H. brasiliensis was lower than in juvenile wood. The sugars and starches present in the cellular lumen periphery of the stem can delay thermal degradation of wood cell wall, and therefore maintain the matured rubberwood closer to its original condition.

In verifying the effects of thermal modification on the physical properties of *S. parahyba* wood, it was found that temperatures ranging up to 220 °C improved dimensional stability without loss of material through internal cracks. These results were similar to previous reports on thermally modified wood of other species (Bhuiyan et al. 2000, Mburu et al. 2007, Calonego et al. 2012, Severo et al. 2012).

Chemical properties of thermally modified wood

The extractive, lignin and holocelluloses contents of juvenile wood from unmodified *S. parahyba* were 1.78, 25.32 and 80.81%, respectively, while

Table 3. Extractives, lignin and holocelluloses content of thermally modified *Schizolobium parahyba* juvenile and mature wood

		Extractives content (%)							
Thermal	N		Juvenile w	boc		Mature wo	ood		
modification	1	CV	Average	Red. or (Inc.) (%)	CV	Average	Red. or (Inc.) (%)	Red. or (Inc.) (%)	
Unmodified	4	24.8	1.78 a		37.4	3.25 a		(82.6) ^{NS}	
180 °C	4	29.2	2.79 ab	(56.7)	41.8	3.66 ab	(12.6)	(31.2) ^{NS}	
200 °C	4	12.3	3.61 b	(102.8)	24.1	4.24 b	(30.5)	(17.5) ^{NS}	
220 °C	4	5.9	6.88 c	(286.5)	20.0	5.06 с	(55.7)	26.5 ^{NS}	

	_	Insoluble klason lignin content (%)							
Thermal	N -		Juvenile wo	bod		Mature wo	ood	Dad	
modification	1	CV	Average	Red. or (Inc.) (%)	CV	Average	Red. or (Inc.) (%)	Red. or (Inc.) (%)	
Unmodified	4	3.0	25.32 a		5.3	22.58 a		10.8 *	
180 °C	4	3.5	24.47 a	3.4	10.9	21.97 a	2.7	10.2 *	
200 °C	4	2.9	24.45 a	3.4	3.9	23.93 a	(6.0)	2.1 *	
220 °C	4	3.6	31.21 b	(23.3)	6.9	32.00 b	(41.7)	(2.5) *	

		Holocelluloses content (%)							
Thermal	N -		Juvenile wood			Mature woo	od		
modification	1	CV	Average	Red. (%)	CV	Average	Red. (%)	- Red. or (Inc.) (%)	
Unmodified	4	2.2	80.81 a		3.2	80.59 a		0.3 *	
180 °C	4	1.4	78.95 a	2.3	2.1	80.95 a	(0.5)	(2.5) *	
200 °C	4	5.7	$72.00 \mathrm{\ b}$	10.9	4.1	$74.62 \mathrm{\ b}$	7.4	(3.6) *	
220 °C	4	4.5	60.63 c	25.0	4.9	66.63 c	17.3	(9.9) *	

where N - repeat number of samples, CV - coefficient of variation, Red. – reduction, Inc. - increase; average followed by the same letters in same column denote significant difference by Tukey test at probability 95% between thermal modifications; * within rows denote significant difference by F test at probability 95% between type of wood; same letters in same column and ^{NS} within rows denote non-significant difference

in mature wood were 3.25, 22.58 and 80.59%, respectively (Table 3). These results were similar to previous studies on hardwoods, in general, as well as *S. parahyba* (Fengel and Wegener 1989, Trianoski 2010).

The effect of thermal modification on the chemical properties of *S. parahyba* wood is shown in Tables 3. Thermal modification caused significant increase in extractives content for juvenile and mature wood (up to 286.5% and 55.7% respectively), and a significant decrease in holocelluloses content (25.5% for juvenile wood and 17.3% for mature wood). The greatest effect for both juvenile and mature wood occurred at 220 °C. It was assumed that the increase in extractives content during thermal modification was due to the degradation of holocelluloses.

Several other studies have shown that thermal modification of wood leads to hemicellulose degradation, resulting in an increase of the extractives and lignin contents (Esteves & Pereira 2009, Bächle et al. 2010, Severo et al. 2012, 2016, Umar et al. 2016). A significant increase in lignin content for both juvenile (23.3%) and mature wood (41.7%) occurred with thermal modification at 220 °C. These results were similar to previous studies that reported a general trend, where the relative mass proportion of lignin increased with both elevated temperature and extended modification time, with a simultaneous decrease in sugar content (Mburu et al. 2007, Severo et al. 2012, 2016, Umar et al. 2016).

CONCLUSIONS

This study showed that thermal modification of *S. parahyba* wood decreased density at 9% moisture content (of up to 9.1%) and volumetric swelling (up to 54.1%). For chemical properties, significant increase was detected in the respective extractives (up to 286.5%) and lignin contents (up to 41.7%), and reduction in holoceluloses (up to 25.0%), when the wood was submitted to thermal modification at 220 °C. The results demonstrated that basic density was not the most suitable parameter for evaluating the quality of thermally modified wood. The influence of thermal modification at 180–220 °C in juvenile wood was equal to mature wood.

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