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# EFFECT OF THE THERMAL TREATMENT ON THE CHEMICAL COMPONENTS, SORPTION, AND SHRINKAGE PROPERTIES OF *Tectona grandis* JUVENILE WOOD

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## ABSTRACT

The effect of thermal treatment on the chemical components, equilibrium moisture content (EMC), and shrinkage of teak juvenile wood was studied. Heartwood and sapwood samples were thermally-treated at 180 °C and 200 °C. Extractive, Klason lignin, holocellulose, and  $\alpha$ -cellulose contents, as well as pH on untreated and thermally-treated woods, were determined. The EMC was reached at five relative humidity (RH) levels using saturated salt solutions: 86 % (KCl), 76 % (NaCl), 58 % (NaBr), 33 % (MgCl<sub>2</sub>), and 0 % (P<sub>2</sub>O<sub>5</sub>). Linear and volumetric shrinkages were calculated for all EMCs. The ratio of sorption (S), coefficient of shrinkage (*h*), and fiber saturation point (FSP) were also determined. Thermally-treated wood exhibited lower holocellulose and  $\alpha$ -cellulose contents than untreated wood and increased acidity due to degradation of the hemicelluloses. The thermal treatment reduced the EMC of heartwood and sapwood. However, sapwood was more sensitive to RH variations than heartwood regardless of the treatment. Thermally-treated woods had higher hygroscopic and dimensional stabilities, and lower FSP than untreated wood. The thermal treatment did not affect radial shrinkage of the heartwood between 33 % and 86 % RH. Heartwood was more sensitive to the effect of the thermal treatment on shrinkage and degradation of cell wall polymers compared to sapwood.

**Keywords:** Chemical modification, dimensional stability, equilibrium moisture content, shrinkage, teak, *Tectona grandis*.

## INTRODUCTION

Thermal modification is a technique that has been extensively studied in the last years because it enhances several wood properties such as lower hygroscopicity and higher dimensional stability (Giebelier 1983) and decay resistance (Weiland and Guyonnet 2003). The treatment provokes physical and chemical changes to wood, such as extractive volatilization, formation of new compounds (e.g. anhydrosugars and phenolic compounds), degradation of the hemicelluloses (Esteves *et al.* 2011), changes in the lignin structure (Kim *et al.* 2014),

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increase of crystalline cellulose (Li *et al.* 2015), and mass losses (García *et al.* 2012), among other modifications. These chemical changes reduce the OH groups available in cell walls, promoting lower hygroscopicity and higher dimensional stability to thermally-treated woods.

The teak (*Tectona grandis*) plantations in Brazil have shorter cutting cycles (15 to 25 years) compared to those in its native region and other regions around the world (Tsukamoto Filho *et al.* 2003, FAO 2000) due to favorable edaphoclimatic conditions and management techniques. However, teak wood from fast-grown plantations exhibits high proportions of juvenile wood and sapwood, which have different anatomical, physical, chemical, and aesthetic properties than those of mature wood and heartwood (Bhat *et al.* 2001, Lopes 2012, Darmawan *et al.* 2015). Studies have shown that the proportion of juvenile wood measured at breast height (1,3 m) is 80-100 % for 20-year-old trees (Bhat *et al.* 2001) and 100 % for 10-year-old trees (Darmawan *et al.* 2015). Juvenile wood presents, for example, lower density, lower shrinkage, shorter fibers, higher microfibril angle, and lower bending strength than mature wood (Darmawan *et al.* 2015). Teak heartwood presents good dimensional stability and high biological durability due to some substances such as caoutchouc (Yamamoto *et al.* 1998), tectoquinone ( $\beta$ -methyl anthraquinone), 2-(hydroxymethyl) anthraquinone, and lapachol (Niamké *et al.* 2011). On the other hand, teak sapwood presents a lighter color, lower dimensional stability and low resistance to the attack of xylophagous organisms than heartwood (Lopes 2012, Motta *et al.* 2013). Therefore, treatments to improve the properties of sapwood are required to increase the yield of this raw material. Previous studies have shown that thermal treatments provide color uniformity to pieces of teak wood containing heartwood and sapwood (Lopes *et al.* 2014). In addition, thermally-treated teak wood can show higher color stability after exposure to ultraviolet radiation depending on the treatment conditions (García *et al.* 2014).

Studies about the dimensional stability of wood are essential for its use because it depends on the relationships between specific gravity and the relative humidity (RH) of the air (Tsoumis 1991). Particularly for teak species, studies have reported the volumetric swelling (Kartikawati *et al.* 2020) and anti-swelling efficiency of heat-treated and untreated 15-year-old teak wood from Indonesia (Priadi *et al.* 2019), the maximum shrinkage of teak wood from the West Africa region (tree age was not specified) (Govorčín *et al.* 2010) among others. However, there are no studies on the dimensional stability of thermally-treated teak juvenile wood under different RH conditions, in particular teak wood from fast-growing plantations in Brazil, which highlights the originality of our study. In general, teak wood is popular in outdoor applications (Weaver 1993), which require materials with attractive color, high dimensional stability under different climatic conditions, and high resistance to pathogens and weathering (Kokutse *et al.* 2006).

With the ultimate goal of contributing to a better understanding of the properties of teak wood after heat treatment and to enhance the use of teak wood from young plantations in Brazil, this study aimed, in the first part, to assess the effect thermal treatments on the chemical composition of juvenile teak wood (heartwood and sapwood), while in the second part, we studied the EMC and the variation of shrinkage under different RH of thermally-treated wood.

## MATERIAL AND METHODS

### Material and thermal treatment

Twelve-year-old teak (*Tectona grandis* Linn. f.) trees were harvested from a plantation located in Caceres city, Mato Grosso State, Brazil (Latitude: 16° 04' 14" S, Longitude: 57° 40' 44" W). Based on previous studies (Bhat *et al.* 2001, Flórez *et al.* 2014, Darmawan *et al.* 2015), we have assumed that the samples contained only juvenile wood as they were obtained from fast-growing young trees. In addition, samples were taken from the second log cut from the diameter at breast height, where the proportion of juvenile wood is greater. Samples of 150 mm (L) x 75 mm (T) x 20 mm (R) were prepared from heartwood or sapwood.

Wood samples were heat-treated in a muffle furnace of 600 mm x 600 mm x 700 mm equipped with a temperature and time controller. The treatment was performed at four steps: (1) heating up to 100 °C for 2 h, (2) heat increasing from 100 °C to final temperature (180 °C or 200 °C) for 30 min, (3) thermal exposure at the final temperature for 2,5 h; and (4) cooling for approximately 24 h. Before and after thermal treatment, samples were conditioned at 20 °C and 65 % RH until mass equilibrium was reached.

## Chemical analysis

Chemical analyses were done according to ASTM-D1105-96 (2001) and ASTM-D1106-96 (2001) to determine extractive, Klason lignin, holocellulose, and  $\alpha$ -cellulose contents. The extractive contents were obtained after extractions in a Soxhlet apparatus for 24 h using three organic solvents of increased polarity: cyclohexane, ethyl acetate, and methanol.

The Klason lignin was obtained after treatment of approximately 300 mg extractive-free dry ground wood with 72 % sulfuric acid ( $\text{H}_2\text{SO}_4$ ) in a water bath at 30 °C for 1 h, followed by dilution to 84 mL distilled water and refluxing for 4 h. The residue was washed with 500 mL hot distilled water and oven-dried at 103 °C  $\pm$  2 °C for 24 h. The Klason lignin content was estimated by dividing the oven-dry weight of the residue (insoluble lignin) by the weight of the extractive-free wood.

The holocellulose fraction was determined by the chlorination method using 2,5 g of extractive-free dry wood added of 80 mL hot distilled water, 0,5 mL acetic acid, and 1 g sodium chlorite ( $\text{NaClO}_2$ ). The mixture was heated in a water bath at 70 °C, and another dose of 0,5 mL acetic acid and 1 g  $\text{NaClO}_2$  were added every 1 h until the fibers were completely separated. The mixture was left for 24 h without reagent addition and was then filtered under vacuum and washed with distilled water until the yellow color and chlorine odor completely disappeared. The residue was oven-dried at 103 °C  $\pm$  2 °C for 24 h and weighed. The holocellulose content was then estimated by dividing the oven-dry weight of the residue (holocellulose) by the weight of the extractive-free wood.

The  $\alpha$ -cellulose was obtained after treatment of 2 g dry holocellulose with 10 mL of 17,5 % sodium hydroxide ( $\text{NaOH}$ ) in a water bath at 20 °C. Another 5 mL of 17,5 %  $\text{NaOH}$  was added to the mixture at 5 min intervals for a total treatment of 45 min. A 33 mL distilled water at 20 °C was added and left for 1 h. The mixture was filtered under vacuum and subsequently washed with 100 mL of 8,3 %  $\text{NaOH}$  and with distilled water. The cellulose residue was oven-dried at 103 °C  $\pm$  2 °C for 24 h. The cellulose content was estimated by dividing the oven-dry weight of the cellulose residue by the weight of the dry holocellulose residue.

The pH of wood was determined as described by Lelis (1995). A 5 g of ground wood oven-dried at 103 °C  $\pm$  2 °C for 24 h was mixed with distilled water at room temperature and left for 24 h. The mixture was filtered and the pH was measured with an electronic pHmeter.

Three replicates per treatment were used for each chemical component and pH analysis.

## Sorption tests

Samples of 20 mm (L) x 15 mm (T) x 13 mm (R) were prepared with the untreated and heat-treated wood pieces of each wood type: heartwood and sapwood. Firstly, the samples were full saturated following a mild procedure in three steps: (1) adsorption in desiccators containing potassium chloride - KCl (86 % RH) at 21 °C with mass measurements until equilibrium was reached; (2) adsorption in desiccators over deionized water until mass equilibrium was reached; and (3) immersion in deionized water for seven days until full saturation was reached. Afterward, samples were separated into five matched groups. One group was immediately put in a desiccator over phosphorus pentoxide -  $\text{P}_2\text{O}_5$  (0 % RH). The other four groups were placed for desorption in desiccators at four levels of RH: 86 %, 76 %, 58 %, and 33 %, by using saturated salt solutions of potassium chloride (KCl), sodium chloride (NaCl), sodium bromide (NaBr), and magnesium chloride ( $\text{MgCl}_2$ ), respectively. A total of 42 samples, 7 replicates for each treatment (wood type (sapwood and heartwood) and temperature (control or untreated, and treated at 180 °C, and 200 °C)) were used for each sorption condition. Samples were placed into two desiccators per RH condition.

Desorption tests were conducted simultaneously on all samples using sorption vats as described by Hernández and Pontin (2006). The temperature of the vats was established at 21 °C. These vats provided temperature control of  $\pm$  0,01 °C for long periods and thus allowing control of RH in the various desiccators serving as small sorption chambers. The samples were placed in plastic supports with a perforated base to promote the exchange of moisture. The weight of samples was measured periodically without removing them from the desiccators until a constant mass was reached.

All mass of samples was taken to the nearest 0,001 g and their dimensions were measured in the three principal directions with a micrometer to the nearest 0,001 mm after full saturation. Dimensions and mass of the four groups of conditioned samples were also measured once their corresponding sorption equilibrium

condition was reached and then they were immediately oven-dried at 103 °C for 24 h. Samples dimensions at the oven-dried state were measured after a 20 min period of cooling over P<sub>2</sub>O<sub>5</sub>.

### Equilibrium moisture content and ratio of sorption

The EMC of untreated and thermally-treated woods was calculated with the mass of the samples at equilibrium and the oven-dried mass, expressed as a percentage of oven-dry mass.

The sorption ratio (S) is a parameter of hygroscopicity proposed by Noack *et al.* (1973) to characterize the sensitivity of the EMC variation ( $\Delta EMC$ ) related to the variation in RH ( $\Delta RH$ ) (Equation 1). This parameter assumes that there is a linear relationship between EMC and RH. Therefore, the S-ratio was calculated for desorption between 33 % and 76 % RH.

$$S = \frac{\Delta EMC}{\Delta RH} \quad (1)$$

### Shrinkage and coefficient of shrinkage

The linear shrinkages in the tangential ( $\beta_T$ ), radial ( $\beta_R$ ), and longitudinal ( $\beta_L$ ) directions were calculated in percentage. Volumetric shrinkage was estimated as the sum of these three directional measurements ( $\beta_V$  involves the product  $\beta_T \times \beta_R$  in order to achieve sufficient accuracy within the range of  $\beta_T$  and  $\beta_R$  values normally found for wood, as described by Skaar (2012)) (Equation 2). Shrinkages of untreated and thermally-treated woods were obtained for all EMCs.

$$\beta_V (\%) = \beta_T + \beta_R + \beta_L - (\beta_T \times \beta_R) \quad (2)$$

The coefficient of shrinkage ( $h$ ) is a general index of dimensional stability proposed by Noack *et al.* (1973) to determine the swelling/shrinkage of the wood for every 1 % RH. The  $h$ -coefficient was calculated with the volumetric shrinkage ( $\Delta\beta_V$ ) for desorption between 33 % and 76 % RH (Equation 3).

$$h = \frac{\Delta\beta_V}{\Delta RH} \quad (3)$$

### Statistical analysis

Statistical analyses were done with a STATISTIC 10.0 software. Analysis of variance (ANOVA) and Tukey's tests (at 0,01 of the probability level) were used to verify the effects of the wood type (heartwood and sapwood) and temperature (control or untreated and treated at 180 °C, and 200 °C) on the extractives, Klason lignin, hemicellulose, and  $\alpha$ -cellulose contents, pH, EMC, and shrinkage components ( $\beta_T$ ,  $\beta_R$ ,  $\beta_L$ , and  $\beta_V$ ). The normality and homogeneity of variance were verified by the Shapiro-Wilk and Levene's tests.

## RESULTS AND DISCUSSION

### Chemical changes

A significant interaction was found between wood type (heartwood, sapwood) and temperature for all chemical components (extractive, Klason lignin, holocellulose, and  $\alpha$ -cellulose) and pH (Table 1 and Table 2). The untreated heartwood had a higher extractive content than the untreated sapwood. The extractive content gradually increased as the temperature of treatment increased for both wood types (Table 3). The total extrac-

tive content of treated heartwood and sapwood respectively increased by 53 % and 64 % at 180 °C and by 122 % and 100 % at 200 °C compared to untreated woods. Similar behavior for the extractive content was observed by other authors after thermal treatments (Gašparík *et al.* 2019, Bellon 2013, Lengowski 2011). Gašparík *et al.* (2019) found an increase of 56 % on teak extractive after the ThermoWood® treatment at 210 °C. Also, the extractive content increased by 135 % (Bellon 2013) and 158 % (Lengowski 2011) in the 160 °C-treated teak wood from VAP HolzSysteme® industrial process compared to untreated wood. However, these studies did not separate heartwood from sapwood material. The increase of the extractive content is not related to the natural extractives of wood, which are in large part volatilized during the thermal treatment, but to by-products formed mainly from the degradation of the hemicelluloses. According to International ThermoWood Association (2003), temperatures ranging from 120 °C to 180 °C increase the extractive content of wood while the temperature up to 230 °C causes a decrease in extractives.

**Table 1:** F-values obtained from the ANOVA of the effects of the wood type (heartwood, sapwood) and temperature treatment on the extractive content of teak wood.

Source of variation	Extractive content			Total extractive content
	Cyclohexane	Ethyl acetate	Methanol	
Wood type	37,4**	1367,2**	722,5**	34,9**
Temperature	865,1**	443,4**	375,0**	1218,6**
Wood type*Temperature	111,8**	15,4**	23,3**	24,7**

\*\*Significant at the 1 % probability level.

**Table 2:** F-values obtained from the ANOVA of the effects of the wood type (heartwood, sapwood) and temperature treatment on the chemical components and pH of teak wood.

Source of variation	Klason lignin	Holocellulose	$\alpha$ -cellulose	pH
Wood type	6,1*	784,0**	1312,7**	2212,1**
Temperature	6,6*	707,2**	33,3**	512,1**
Wood type*temperature	2,0 <sup>ns</sup>	109,1**	2,6 <sup>ns</sup>	21,6**

\*Significant at the 5 % probability level \*\* Significant at the 1 % probability level; <sup>ns</sup> not significant.

**Table 3:** Means of the extractive contents of untreated and thermally-treated teak heartwood and sapwood.

Wood type	Temperature (°C)	Extractive content (%)			Total extractive content (%)
		Cyclohexane	Ethyl acetate	Methanol	
Heartwood	Control	1,60 (0,12) d	2,63 (0,05) e	2,63 (0,05) c	6,86 (0,21) c
	180	2,15 (0,08) b	4,58 (0,05) b	3,73 (0,08) b	10,46 (0,25) b
	200	4,52 (0,05) a	5,13 (0,08) a	5,56 (0,10) a	15,21 (0,40) a
Sapwood	Control	0,51 (0,06) e	2,70 (0,05) e	0,61 (0,08) f	3,82 (0,16) d
	180	1,93 (0,03) c	3,23 (0,08) d	1,12 (0,08) e	6,28 (0,01) c
	200	2,41 (0,05) b	3,71 (0,08) c	1,50 (0,05) d	7,62 (0,03) c

The number in parentheses are standard errors. Means within a column followed by the same letters are not significantly different at the 1 % probability level.

**Table 4:** Means of the chemical component contents and pH of untreated and thermally-treated teak heartwood and sapwood.

Wood type	Temperature (°C)	Klason Lignin (%)	Holocellulose (%)	$\alpha$ -cellulose (%)	pH
Heartwood	Control	29,3 (1,5) b	63,6 (0,4) b	45,4 (1,1) c	5,55 (0,03) d
	180	31,3 (1,5) ab	57,0 (0,2) d	42,6 (1,4) c	4,86 (0,02) e
	200	35,7 (3,1) a	47,3 (0,8) e	39,6 (1,4) d	4,72 (0,09) f
Sapwood	Control	29,7 (3,2) ab	66,5 (0,6) a	60,8 (1,6) a	6,53 (0,01) a
	180	27,7 (1,5) b	63,6 (0,7) b	58,6 (0,8) ab	5,72 (0,03) c
	200	31,3 (1,5) ab	59,4 (0,1) c	57,5 (0,5) b	5,92 (0,02) b

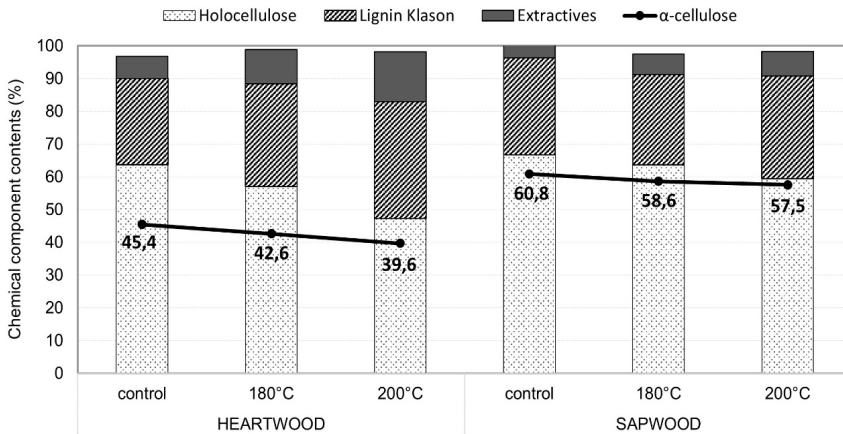
The number in parentheses are standard errors. Means within a column followed by the same letters are not significantly different at the 1 % probability level.

The treatment at 200 °C increased the Klason lignin content in heartwood but not in sapwood (Table 4). The increase in lignin content was also found in thermally-treated teak (Bellon 2013, Lengowski 2011), Caribbean pine (Poubel *et al.* 2013), and eucalypt (de Moura *et al.* 2012) woods. Several works have reported chemical changes in lignin structure due to thermal treatments. Chemical analysis of thermally-treated teak wood assessed by Fourier-transform infrared (FTIR) spectroscopy showed an increase in band intensity at 1328  $\text{cm}^{-1}$  assigned to C-H vibration in cellulose and C<sub>1</sub>-O vibration in syringyl derivative-condensed structures in lignin, which suggest the formation of condensed structures (Li *et al.* 2015). Kim *et al.* (2014) also reported depolymerization (cleavage of  $\beta$ -O-4 linkage) and condensation between lignin fragments during thermal treatment. Although the thermal treatment affects lignin due to depolymerization, hemicelluloses reach a more advanced stage of degradation starting at 180 °C compared to lignin, which results in an apparent lignin increase in relation to the other components of the cell wall (Sundqvist 2004).

The untreated sapwood exhibited the highest holocellulose and  $\alpha$ -cellulose contents (Table 4). According to Stamm (1964), hemicellulose and cellulose (amorphous portion and crystallite surfaces) are the main responsible for the hygroscopicity of wood while lignin plays a secondary role.

Holocellulose content decreased gradually with the temperature increase for both wood types (Table 4). The holocellulose content of treated heartwood and sapwood decreased by 10 % and 4 % at 180 °C, and by 26 % and 11 % at 200 °C, respectively. The holocellulose decrease in treated woods was probably due to the degradation of hemicelluloses. The results regarding the heartwood and sapwood behavior confirm those of previous works. Lopes *et al.* (2018) used infrared spectroscopy to assess the chemical modification of the surface of thermally-modified teak wood. A more pronounced decrease in OH<sup>-</sup> groups in heartwood than in sapwood after heat treatment was observed. The authors provide a good discussion about the different chemical changes in teak heartwood and sapwood after heat treatment and the greater sensitivity of the heartwood to thermal degradation compared to sapwood, but it is not yet clear why this occurs. No significant differences were found between  $\alpha$ -cellulose contents of untreated and 180 °C-treated woods. On the other hand, the  $\alpha$ -cellulose content decreased in the 200 °C-treated heartwood and sapwood. Among the chemical constituents of the cell wall, hemicelluloses are primarily consumed because they are easily degraded at low temperatures (140 °C) while cellulose is more resistant to thermal degradation (Sundqvist 2004). Other authors reported a holocellulose decrease of 24-25 % in teak (Bellon 2013, Lengowski 2011), 18,6 % in *Eucalyptus grandis*, and 4,6 % in *Pinus taeda* woods after thermal treatment at 160 °C by the VAP HolzSysteme® process (Lengowski 2011).

In summary, the chemical changes caused by the thermal treatment were more important in heartwood than in sapwood (Figure 1). The apparent total extractive content (or by-products formed mainly from the degradation of hemicelluloses) increased more in the thermally-treated heartwood than in the thermally-treated sapwood due to greater susceptibility to thermal degradation of the former. The relative lignin content increased by 20 % in 200 °C-treated heartwood while no significant change was found in the sapwood. The holocellulose content decreased more in heartwood than in sapwood at both thermal treatment degrees. The  $\alpha$ -cellulose content decreased by 12,8 % in heartwood and 5,4 % in sapwood after the treatment at 200 °C. Studies realized by Lopes *et al.* (2018) using FTIR spectroscopy also reported a more important thermal degradation in heartwood than in sapwood.



**Figure 1:** Chemical component contents of untreated and thermally-treated teak wood.

The untreated heartwood was more acid (pH = 5,55) than the untreated sapwood (pH = 6,53) (Table 4). The high acidity of heartwood compared to sapwood has often been reported in the literature for several hardwood species (Kakavas *et al.* 2018, Passialis *et al.* 2008). Untreated woods usually have a pH near to 5,0-5,5 (Boonstra *et al.* 2007), however, pH values depend on the extraction conditions (Geffert *et al.* 2019). Lower pH values are found with hot water extraction compared to cold water, due to the greater release of acetic acid (Geffert *et al.* 2019). Therefore, the extraction method used in our study can explain the high pH values found in untreated teak woods, principally sapwood. The thermal treatment significantly decreased the pH values of both types of wood, principally of the heartwood. The most expressive chemical degradation in the heartwood after thermal treatment can be also confirmed by its higher acidity. The increased acidity in thermally-treated woods can be explained by the hemicellulose degradation and formation of acetic acid. This acid also acts as catalysts in the depolymerization process of the cellulose microfibrils breaking them into smaller chains (Boonstra *et al.* 2007), in addition to the condensation and degradation reactions in lignin structure (Li *et al.* 2015) that result in the formation of phenolic groups (Kim *et al.* 2014).

### Equilibrium moisture content

The ANOVAs showed respectively a significant effect of the heat treatment and wood type on the EMC for all moisture sorption conditions, except in the anhydrous state (Table 5). As expected, the analysis of untreated samples showed that the EMC was lower in heartwood than sapwood (Table 6). This can be attributed to the higher proportion of extractives present in heartwood (Table 3). Previous work about the chemical composition of teak juvenile wood assessed by FTIR spectroscopy also showed a higher extractive content (quinones, oils, and waxes) in heartwood than sapwood (Lopes *et al.* 2018). In fact, several works have shown that teak wood and other tropical woods have a hydrophobic behavior due to the presence of extractives in their tissues (Hernández 2007a, Jankowska *et al.* 2017). Furthermore, EMC decreased due to the heat treatments (control to 180 °C to 200 °C) for all moisture sorption conditions and wood types (Table 6). For instance, the EMC of heartwood and sapwood treated at 180 °C significantly decreased 3,9 % and 1,1 % MC compared to the corresponding untreated samples in desorption at 86 % RH, respectively. EMC decreased even more for samples treated at 200 °C, namely 4,9 % for heartwood and 4,7 % MC for heartwood and sapwood at the same RH. For all cases, the decrease in EMC due to the heat treatment was higher for heartwood than for sapwood.

**Table 5:** F-values obtained from the ANOVAs of the effects of the wood type (heartwood, sapwood) and temperature treatment on the EMC and each shrinkage component.

0% RH					
Source of variation	EMC	$\beta_T$	$\beta_R$	$\beta_L$	$\beta_V$
Wood type	4,5 <sup>ns</sup>	29,3 <sup>**</sup>	35,1 <sup>**</sup>	0,01 <sup>ns</sup>	40,7 <sup>**</sup>
Temperature	1,3 <sup>ns</sup>	16,4 <sup>**</sup>	26,6 <sup>**</sup>	4,8 <sup>ns</sup>	29,5 <sup>**</sup>
Wood type*temperature	1,9 <sup>ns</sup>	4,4 <sup>*</sup>	3,1 <sup>ns</sup>	0,01 <sup>ns</sup>	5,2 <sup>*</sup>
33% RH					
Source of variation	EMC	$\beta_T$	$\beta_R$	$\beta_L$	$\beta_V$
Wood type	14,3 <sup>**</sup>	21,9 <sup>**</sup>	12,0 <sup>**</sup>	3,3 <sup>ns</sup>	19,3 <sup>**</sup>
Temperature	67,6 <sup>**</sup>	11,7 <sup>**</sup>	2,4 <sup>ns</sup>	0,8 <sup>ns</sup>	5,8 <sup>**</sup>
Wood type*temperature	4,7 <sup>*</sup>	3,4 <sup>*</sup>	1,6 <sup>ns</sup>	0,4 <sup>ns</sup>	4,8 <sup>*</sup>
58% RH					
Source of variation	EMC	$\beta_T$	$\beta_R$	$\beta_L$	$\beta_V$
Wood type	40,1 <sup>**</sup>	80,5 <sup>**</sup>	25,05 <sup>**</sup>	0,6 <sup>ns</sup>	34,3 <sup>**</sup>
Temperature	84,9 <sup>**</sup>	12,8 <sup>**</sup>	2,3 <sup>ns</sup>	1,75 <sup>ns</sup>	9,8 <sup>**</sup>
Wood type*temperature	3,4 <sup>*</sup>	2,2 <sup>ns</sup>	2,6 <sup>ns</sup>	0,33 <sup>ns</sup>	3,7 <sup>*</sup>
76% RH					
Source of variation	EMC	$\beta_T$	$\beta_R$	$\beta_L$	$\beta_V$
Wood type	49,1 <sup>**</sup>	112,1 <sup>**</sup>	15,2 <sup>**</sup>	0,01 <sup>ns</sup>	83,7 <sup>**</sup>
Temperature	107,5 <sup>**</sup>	20,8 <sup>**</sup>	2,8 <sup>ns</sup>	1,7 <sup>ns</sup>	14,3 <sup>**</sup>
Wood type*temperature	2,6 <sup>ns</sup>	3,7 <sup>*</sup>	2,2 <sup>ns</sup>	2,2 <sup>ns</sup>	5,9 <sup>**</sup>
86% RH					
Source of variation	EMC	$\beta_T$	$\beta_R$	$\beta_L$	$\beta_V$
Wood type	126,9 <sup>**</sup>	34,6 <sup>**</sup>	2,8 <sup>ns</sup>	2,2 <sup>ns</sup>	13,2 <sup>**</sup>
Temperature	101,1 <sup>**</sup>	6,1 <sup>**</sup>	1,9 <sup>ns</sup>	2,7 <sup>ns</sup>	2,2 <sup>ns</sup>
Wood type*temperature	12,6 <sup>**</sup>	2,3 <sup>ns</sup>	0,1 <sup>ns</sup>	0,1 <sup>ns</sup>	1,6 <sup>ns</sup>

\* Significant at the 5% probability level \*\* Significant at the 1 % probability level; <sup>ns</sup> not significant.

**Table 6:** Means of the equilibrium moisture content (EMC) of untreated and thermally-treated teak heartwood and sapwood for different sorption conditions at 21 °C.

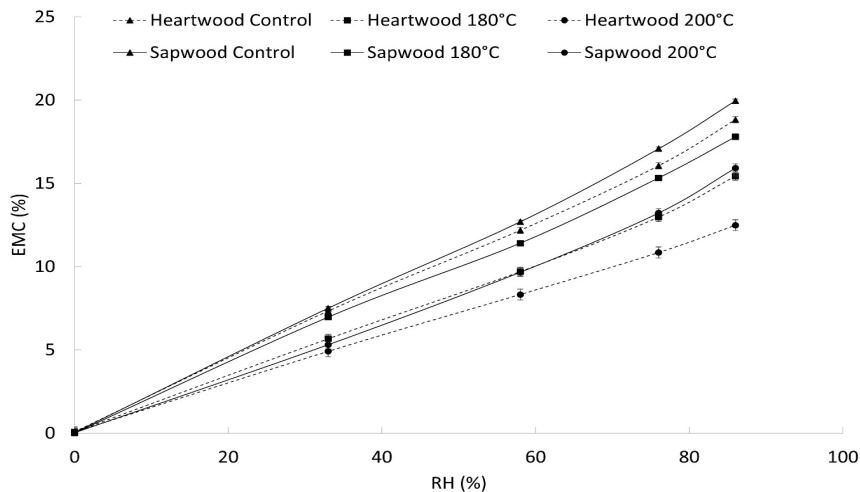
Temperature (°C)	EMC in desorption (%)									
	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood
Control	0,09 Aa (0,08)	0,04 Aa (0,04)	7,3 Aa (0,1)	7,5 Aa (0,1)	12,2 Aa (0,2)	12,7 Aa (0,1)	16,1 Ba (0,2)	17,1 Aa (0,1)	19,9 Ba (0,3)	21,9 Aa (0,02)
180	0,07 Aa (0,06)	0,01 Aa (0,04)	5,7 Bb (0,2)	7,0 Aa (0,6)	9,7 Bb (0,3)	11,4 Ab (0,1)	13,0 Bb (0,3)	15,3 Ab (0,1)	16,0 Bb (0,4)	20,8 Ab (0,3)
200	0,05 Aa (0,03)	0,01 Aa (0,04)	4,9 Ac (0,2)	5,3 Ab (0,4)	8,3 Bc (0,3)	10,0 Ac (0,4)	10,5 Bc (0,5)	12,8 Ac (0,4)	15,0 Bb (0,7)	17,2 Ac (0,3)

The standard error is given in parentheses. Means followed by the same letters are not significantly different at the 5 % probability level. Uppercase letters: mean comparisons between wood types, for each temperature and RH separately. Lowercase letters: mean comparisons among thermal treatments within a column.

The difference in EMC between untreated samples and both treated woods (180 °C or 200 °C), increased as RH increased from 33 % to 86 %, for both heartwood and sapwood (Figure 2). The most important decrease in EMC occurred at 200 °C. The 200 °C-treated wood had lower holocellulose and  $\alpha$ -cellulose contents (Table 4) and higher total extractive content (by-products formed during thermal degradation) (Table 3) than other treatments. These chemical modifications may explain the lowest EMC of 200 °C-treated heartwood and sapwood

compared to the other treatments. Other studies have reported reductions in EMC of teak wood thermally-treated at 160 °C (Lengowski 2011, Bellon 2013).

Generally, the sapwood had higher EMC than the heartwood after the heat treatments (Table 6, Figure 2). This is due to the higher holocellulose and  $\alpha$ -cellulose contents in sapwood (Table 4) which are the most hygroscopic polymers of cell walls. The higher extractive content of treated heartwood could also affect the EMC because they decrease the wood hygroscopicity.



**Figure 2:** Equilibrium moisture content (EMC) in desorption of untreated and thermally-treated teak wood as a function of relative humidity (RH) at 21 °C.

### Shrinkage

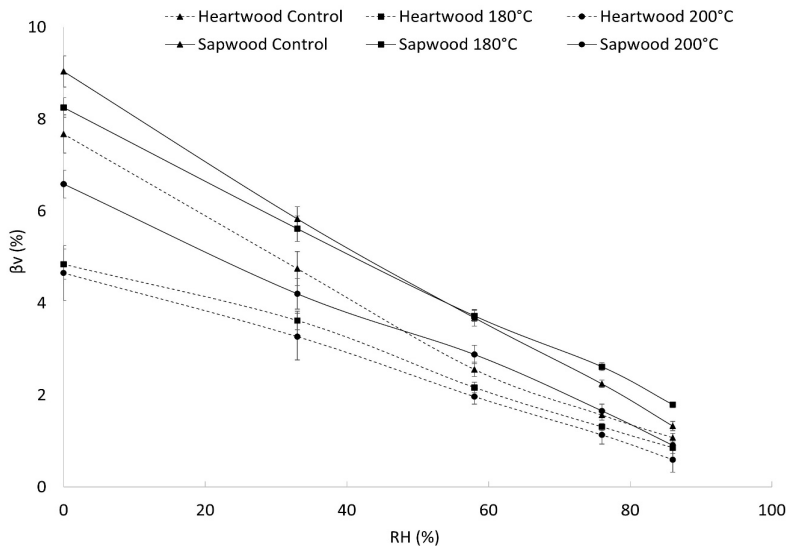
The results of untreated samples confirmed that teak wood presents low values of partial and total shrinkages compared to other tropical hardwoods (FPL 2010). As expected, untreated heartwood had lower total and partial linear and volumetric shrinkages than untreated sapwood at all moisture sorption conditions. The total  $\beta_T$ ,  $\beta_R$ ,  $\beta_L$ , and  $\beta_V$  of heartwood were of 4,6 %, 2,6 %, 0,5 %, and 7,7 % while those of untreated sapwood were of 5,2 %, 3,3 %, 0,5 %, and 8,8 %, respectively (Table 7). However, given the very low values, no significant difference was found between heartwood and sapwood for  $\beta_L$ . These results are similar to those reported by Miranda *et al.* (2011) to 50-70 years-old teak wood, which were 5,2 %, 3,5 %, and 7,6 % (mean from pith to bark) for  $\beta_T$ ,  $\beta_R$ , and  $\beta_V$ , respectively. The lower shrinkage in heartwood is attributed to the high extractive content observed in this wood type (Hernández 2007b, Jankowska *et al.* 2017).

**Table 7:** Means of the shrinkage of untreated and thermally-treated teak heartwood and sapwood for different sorption conditions at 21 °C.

Tangential shrinkage – $\beta_T$ (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	4,6 Ab (0,3)	5,2 Aa (0,4)	3,1 Ba (0,3)	3,8 Aa (0,2)	1,7 Ba (0,1)	2,5 Aa (0,1)	0,9 Ba (0,08)	1,5 Ab (0,07)	0,4 Ba (0,1)	0,9 Aa (0,1)
180	2,9 Ba (0,2)	5,2 Aa (0,1)	2,2 Bb (0,2)	3,8 Aa (0,1)	1,3 Bb (0,1)	2,5 Aa (0,06)	0,8 Ba (0,08)	1,8 Aa (0,05)	0,3 Ba (0,04)	0,8Aa (0,06)
200	2,7 Ba (0,3)	3,7 Ab (0,3)	2,1 Ab (0,3)	2,6 Ab (0,3)	1,2 Bb (0,07)	1,9 Ab (0,2)	0,5 Bb (0,1)	1,1 Ac (0,08)	0,3 Ba (0,04)	0,5 Ab (0,04)
Radial shrinkage – $\beta_R$ (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	2,6 Ba (0,2)	3,3 Aa (0,1)	1,5 Aa (0,1)	1,9 Aa (0,2)	0,8 Ba (0,07)	1,0 Aa (0,1)	0,5 Aa (0,06)	0,6 Aa (0,04)	0,4 Aa (0,03)	0,9 Aa (0,1)
180	1,5 Bb (0,1)	2,7 Ab (0,1)	1,2 Ba (0,1)	2,0 Aa (0,2)	0,7 Ba (0,04)	1,2 Aa (0,04)	0,5 Ba (0,02)	0,8 Aa (0,06)	0,3 Aa (0,05)	0,4 Aa (0,1)
200	1,7 Ab (0,2)	2,1 Ac (0,2)	1,2 Aa (0,2)	1,5 Aa (0,1)	0,7 Aa (0,1)	0,9 Aa (0,02)	0,4 Aa (0,06)	0,5 Aa (0,07)	0,1 Aa (0,03)	0,3 Aa (0,1)
Longitudinal shrinkage – $\beta_L$ (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	0,51 Aa (0,01)	0,50 Aa (0,04)	0,33 Aa (0,06)	0,23 Aa (0,02)	0,14 Aa (0,07)	0,15 Aa (0,07)	0,11 Aa (0,03)	0,12 Aa (0,02)	0,15 Aa (0,05)	0,10 Aa (0,04)
180	0,42 Aa (0,02)	0,43 Aa (0,06)	0,27 Aa (0,03)	0,24 Aa (0,04)	0,16 Aa (0,05)	0,08 Aa (0,04)	0,04 Aa (0,02)	0,09 Aa (0,04)	0,20 Aa (0,04)	0,16 Aa (0,03)
200	0,34 Aa (0,04)	0,34 Aa (0,04)	0,25 Aa (0,04)	0,19 Aa (0,04)	0,07 Aa (0,02)	0,04 Aa (0,05)	0,13 Aa (0,04)	0,07 Aa (0,02)	0,27 Aa (0,04)	0,19 Aa (0,06)
Volumetric shrinkage – $\beta_V$ (%)										
Temperature (°C)	0 % RH		33 % RH		58 % RH		76 % RH		86 % RH	
	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood	Heart-wood	Sap-wood
Control	7,7 Ba (0,4)	8,8 Aa (0,4)	4,7 Ba (0,3)	5,8 Aa (0,3)	2,7 Ba (0,2)	3,7 Aa (0,2)	1,5 Ba (0,1)	2,2 Ab (0,08)	0,9 Aa (0,1)	1,4 Aa (0,2)
180	4,9 Bb (0,3)	8,3 Aa (0,2)	3,6 Bb (0,3)	6,1 Aa (0,4)	2,2 Bab (0,1)	3,7 Aa (0,1)	1,3 Bab (0,08)	2,6 Aa (0,08)	0,6 Ba (0,2)	1,4 Aa (0,1)
200	4,7 Bb (0,5)	6,1 Ab (0,4)	3,9 Aab (0,5)	4,2 Ab (0,3)	2,0 Ab (0,2)	2,5 Ab (0,3)	1,1 Bb (0,2)	1,7 Ac (0,1)	0,7 Aa (0,07)	0,9 Aa (0,1)

The standard error is given in parentheses. Means followed by the same letters are not significantly different at the 5 % probability level. Uppercase letters: mean comparisons between wood types within a row, for each RH separately. Lowercase letters: mean comparisons among temperatures within a column, for each shrinkage type separately.

The ANOVAs showed a significant effect of the wood type and the thermal treatment on the linear (except for  $\beta_L$ ) and volumetric shrinkages for almost all moisture sorption conditions. The wood type showed a more important effect on partial and total shrinkages compared to the temperature treatment (Table 5). The thermal treatment decreased partial shrinkage in both heartwood and sapwood, however, this effect would be more important in sapwood (Table 7, Figure 3). Total linear and volumetric shrinkages also decreased with the thermal treatments for both wood types but this effect would be more important in heartwood (Table 7). At 200 °C, the  $\beta_T$  of the heartwood and sapwood decreased respectively by 41 % and 29 %,  $\beta_R$  decreased by 35 % and 36 % and  $\beta_V$  decreased by 39 % and 31 %, compared to untreated woods.



**Figure 3:** Volumetric shrinkage ( $\beta_v$ ) of teak wood as a function of relative humidity (RH) at 21 °C.

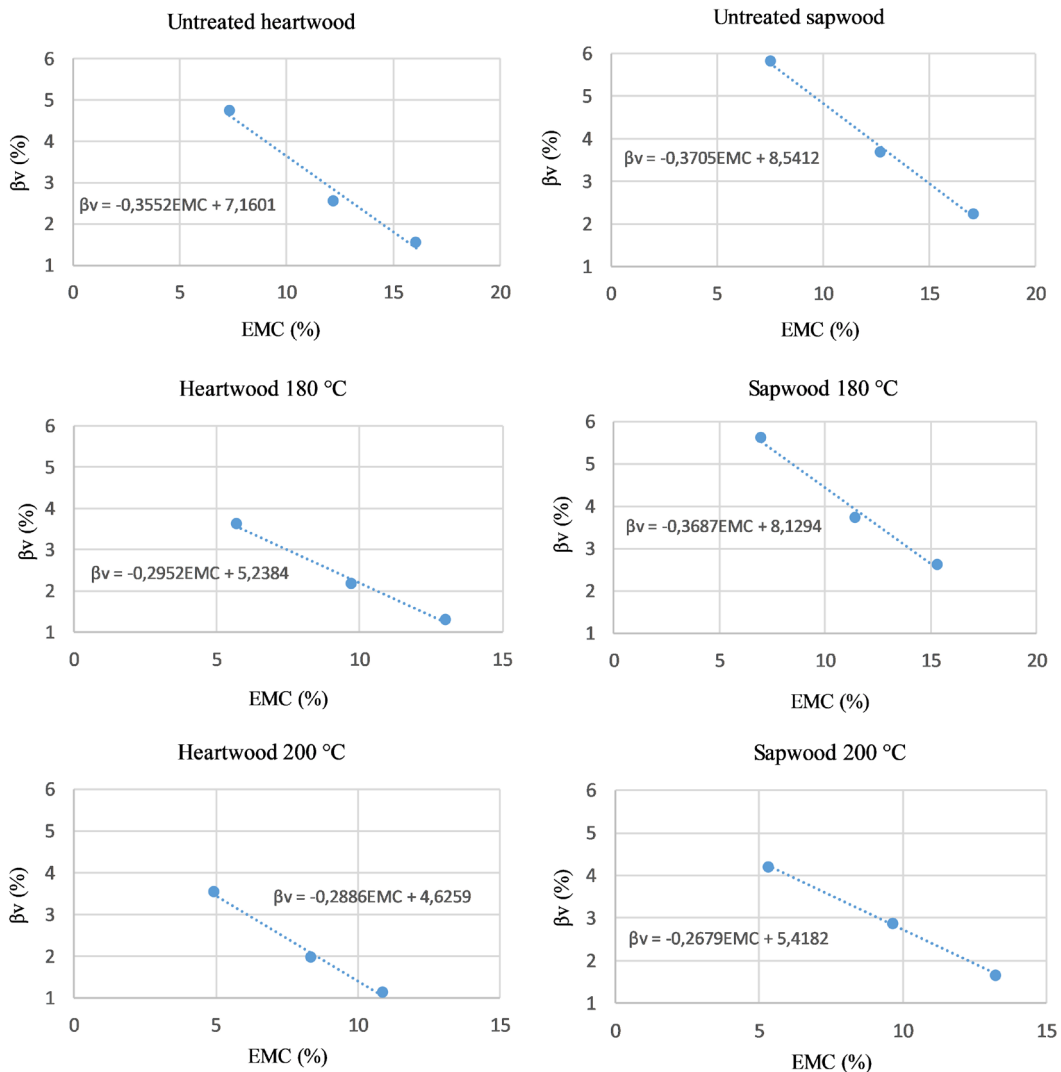
The effects of the thermal treatment were more important in  $\beta_T$  than in  $\beta_R$ . However,  $\beta_L$  was not affected neither by the wood type nor the thermal treatment (Table 5). The greater dimensional variations of the wood occur in the tangential direction from 2,4 % to 11 % followed by radial direction from 3,5 % to 5 % while those in the longitudinal direction are almost negligible from 0,1 % to 0,6 % (Kollmann and Côté Jr. 1968). Several factors can explain the anisotropy of wood such as anatomical and chemical structure, mainly due to restriction of rays in the radial direction and the helical arrangement of the cellulose microfibrils in the cell wall (Kollmann and Côté Jr. 1968). Nevertheless, the relationships between anatomical structure, chemical composition, extractive content, and density cause variations in shrinkage and swelling among wood species.

The hygroscopic and dimensional stability of teak wood were improved after thermal treatment. The increase in the temperature treatment resulted in a decrease of the EMC. The S-ratio was calculated from the EMC-RH curve in the highest sorption linear range from 76 % to 33 % RH (Figure 2). The S-ratio was higher in sapwood than in heartwood in all thermal treatments (Table 8). It clearly shows that sapwood is more sensitive to RH changes than heartwood. These results can be explained by the higher holocellulose and  $\alpha$ -cellulose contents in sapwood compared to heartwood (Table 4) and also by the lower natural extractive content in the untreated sapwood (Table 2). The caoutchouc found in the cell walls and lumens is responsible for the greater hydrophobicity of teak heartwood (Yamamoto *et al.* 1998) which has higher concentrations than that of the sapwood (Lopes *et al.* 2018). The highest decrease in the S-ratio was found with the 200 °C treatment in both wood types (heartwood and sapwood), which could be explained by the greater chemical changes.

**Table 8:** Ratio of sorption (S), coefficient of shrinkage (h), and estimated fiber saturation point (FSP) of untreated and thermally-treated teak woods.

Temperature (°C)	S-ratio		h-coefficient		FSP	
	Desorption 76 % - 33 % (% EMC / % RH)		Desorption 76 % - 33 % (% $\beta_v$ / % RH)			
	Heartwood	Sapwood	Heartwood	Sapwood	Heartwood	Sapwood
Control	0,203	0,223	0,074	0,083	20,2	23,1
180	0,170	0,194	0,054	0,070	17,7	22,1
200	0,138	0,184	0,056	0,059	16,0	20,2

Heartwood was more dimensional stable compared to sapwood in untreated and thermally-treated conditions. Thermally-treated woods showed lower h-coefficient values compared to untreated woods (Table 8), which means that the temperature treatments improve the dimensional stability for both wood types. For sapwood, the treatment at 200 °C was more efficient compared to 180 °C since it provided greater dimensional stability (h-lower coefficient). The curve of the  $\beta_v$  as a function of EMC from 33 % to 76 % RH was used to estimate the FSP by the intersection to zero shrinkage method for all samples (Figure 4). Sapwood exhibited a higher FSP than heartwood for all treatments. Heartwood FSP was similar to that found by Kokutse *et al.* (2010). The higher h-coefficient and higher FSP of the sapwood (Table 8) again show that it is less stable than heartwood and more prone to defects caused by the shrinkage of wood during drying. The greater dimensional stability of thermally-treated woods is due to lower hygroscopicity caused by chemical changes in the cell wall polymers which decrease the water absorption sites (mainly OH groups), and consequently the FSP, and shrinkage (Priadi and Hiziroglu 2013). Overall, the improvement of the dimensional stability of teak wood with temperature treatments, as a result of the diminution of its hygroscopicity (EMC, S, and FSP) and changes in its dimensions (h-values) will optimize the utilization of the wood in several end-uses, such as outdoor furniture, decks, and indoor flooring.



**Figure 4:** Volumetric shrinkage ( $\beta_v$ ) of teak wood as a function of equilibrium moisture content (EMC) between 33 % and 76 % RH.

## CONCLUSIONS

The effects of thermal modification of teak heartwood and sapwood on the chemical components, EMC, and shrinkage at various RHs were investigated. The following conclusions can be drawn:

The untreated heartwood had higher extractive and Klason lignin contents than those of untreated sapwood, which exhibited higher holocellulose and  $\alpha$ -cellulose contents.

The thermal modification decreased the holocellulose and  $\alpha$ -cellulose contents in heartwood and sapwood and increased the acidity of wood. The degradation of hemicelluloses started at 180 °C while that of  $\alpha$ -cellulose started at 200 °C.

Chemical changes were more important in the heartwood than in the sapwood.

The thermal modification reduced the EMC of heartwood and sapwood at sorption conditions from 33 % to 86 % RH. The sapwood was more sensitive to RH variations than heartwood, regardless of the temperature.

The thermal modification provided higher dimensional and hygroscopic stabilities to wood. Generally, the thermal treatment decreased the shrinkage of heartwood and sapwood, however, shrinkage in heartwood was more sensible to the temperature treatment compared to sapwood. Thermally-treated woods also exhibited lower S and FSP compared to untreated ones.

Overall, the results of this research lead to a better understanding of dimensional stability of teak wood from young fast-growing plantations after thermal treatment, which may help define, from a practical point of view, the optimal use of juvenile teak wood in different relative humidity conditions.

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