

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/282841784>

Surface chemical and color characterization of juvenile *tectona grandis* wood subjected to steam-drying treatments

Article in *Surface Review and Letters* · September 2015

DOI: 10.1142/S0218625X15500912

CITATION

1

READS

85

5 authors, including:



Alexander Berrocal

Costa Rican Institute of Technology (ITCR)

29 PUBLICATIONS 132 CITATIONS

SEE PROFILE



Roger Moya Roque

Costa Rican Institute of Technology (ITCR)

176 PUBLICATIONS 853 CITATIONS

SEE PROFILE



M, Rodríguez-Solís

Costa Rican Institute of Technology (ITCR)

9 PUBLICATIONS 2 CITATIONS

SEE PROFILE



Ricardo Starbird

Costa Rican Institute of Technology (ITCR)

18 PUBLICATIONS 34 CITATIONS

SEE PROFILE

Some of the authors of this publication are also working on these related projects:



Tropical tree plantation ecology and silviculture [View project](#)



Modeling of *Tectona grandis* heartwood formation [View project](#)

Surface Review and Letters, Vol. 23, No. 1 (2016) 1550091 (14 pages)
© World Scientific Publishing Company
DOI: 10.1142/S0218625X15500912



SURFACE CHEMICAL AND COLOR CHARACTERIZATION OF JUVENILE *TECTONA GRANDIS* WOOD SUBJECTED TO STEAM-DRYING TREATMENTS

ALEXANDER BERROCAL*, RÓGER MOYA[†]
and MARÍA RODRIGUEZ-SOLIS[‡]
*Escuela de Ingeniería Forestal
Instituto Tecnológico de Costa Rica
Apartado 159-7050, Cartago, Costa Rica
*aberrocal@itcr.ac.cr
†rmoya@itcr.ac.cr
‡maria.rodriguez@itcr.ac.cr*

RICARDO STARBIRD
*Escuela de Química, Instituto Tecnológico de Costa Rica
Apartado 159-7050, Cartago, Costa Rica
rstarbird@itcr.ac.cr*

FREDDY MUÑOZ
*Escuela de Ingeniería Forestal
Instituto Tecnológico de Costa Rica
Apartado 159-7050, Cartago, Costa Rica
fmunoz@itcr.ac.cr*

Received 4 June 2015
Revised 22 September 2015
Accepted 23 September 2015
Published

The color of *Tectona grandis* wood is an attribute that favors its commercialization, however, wood color from fast-growth plantation trees is clear and lacks uniformity. The aim of this work is to characterize steamed teak wood by means of the Fourier transform infrared spectroscopy (FTIR) and $L^*a^*b^*$ color systems. Two moisture conditions (green and 50%) and two grain patterns (flat and quarter) of boards were analyzed through the application of different steaming times (0, 3, 6, 9, 12, 15 and 18 h). The FTIR results showed that the bands at 1158, 1231, 1373 and 1419 cm^{-1} did not show any change with steaming, whereas the bands at 1053, 1108, 1453, 1506, 1536, 1558, 1595, 1652, 1683, 1700 and 1733 cm^{-1} presented a decrease in the intensity with the steaming time. The band at 1318 cm^{-1} was the only one that increased. Lightness (L^*) was the most affected parameter, followed by yellowness (b^*), while redness (a^*) showed the smallest change. Surface color change (ΔE^*) presented the lowest value between 3 h and 6 h of steam-drying in the boards with flat grain, whereas for boards with quarter grain, the smallest ΔE^* value was obtained after 18 h of steaming.

Keywords: Wood treatment; steam-drying treatment; surface analysis; tropical wood; color uniformity.

[†]Corresponding author.

A. Berrocal *et al.*

1. Introduction

Tectona grandis L.f. has been largely planted in many tropical regions, including Latin America, Asia, Africa and Oceania, covering approximately 6 million ha.¹ Thanks to its physical, mechanical and aesthetical properties, the wood of this tropical species has become one of the most important in international markets.²

Additionally, teak wood color is considered a major attribute regarding commercialization.³ Thulasidas *et al.*⁴ indicated that teak wood is a premier hardwood valued for the attractiveness of its golden yellow or brown color.

Teak color has been widely studied in the past few years.² The color of the wood from trees grown in plantations is lighter than the color of the wood from natural forests.^{5,6} For this reason, the price of the wood from trees from short-rotation plantations is lower in the timber market.⁴

In addition to its lighter color, the great variability of the heartwood color of teak wood is another inconvenience.^{3,7} For example, Moya and Berrocal⁷ found approximately 15% variation in the wood color parameters (lightness (L^*), redness (a^*) and yellowness (b^*)). Thulasidas *et al.*⁴ found a similar variation in wood from homegarden trees. Finally, Moya and Marín⁵ found 31–53% variation in the color parameters (L^* , a^* , b^*) in cloned trees.

There are various techniques to homogenize the color of the wood or to try to achieve more uniform darker colors.⁸ Steam-drying treatment has been known for a long time as one of the most effective methods to improve the dimensional stability, decay resistance and durability of wood, while simultaneously darkening the wood color.⁹ In terms of the mechanism for the dark color development, the properties and quantities of major chemicals and extractive compositions in wood are modified during the steam-heat treatment.⁹

Wood color can also be homogenized through drying. However, studies on how to obtain darker teak wood are still unfinished.² For example, in a first attempt, Berrocal and Agüero¹⁰ applied a system of preservation and coloring in order to homogenize the color of the sapwood and the heartwood. Salas and Moya,¹¹ meanwhile, found that lightness diminished while redness and yellowness increased after the process of wood drying with three different methods

(air, kiln and solar drying), thus resulting in darker wood.

Other forms of changing the color of teak wood have been implemented which focus on growing trees. They are focused on when the trees are growing. Recently, Moya and Marín⁵ proposed the genetic selection of trees with similar color conditions to that of trees growing in plantation conditions, in order to achieve darker and less variable improved teak wood.

The color change produced by steaming or drying is caused by chemical changes in the wood surface. The Fourier transform infrared spectroscopy (FTIR) has made it possible to perceive those changes. Specifically, changes occurring in the 800–1800 cm^{-1} band are being studied.¹² Huang *et al.*¹³ and Lionetto *et al.*¹⁴ have shown that employing the ratio I_{1316}/I_{1336} provides information concerning the process of degradation of the amorphous and crystalline cellulose zones during the steaming. Moreover, changes due to steaming at peaks at 1738, 1596 and 810 cm^{-1} show alterations in the wood hemicellulose and lignin.^{12,15}

Although efforts have been made to standardize the color of teak wood from plantation trees, little research has been conducted as to the changes produced by steaming in combination with drying on the wood of trees from fast-growth plantations. Therefore, the aim of the present study is to establish the changes of color (measured by $L^*a^*b^*$ color systems) as well as the chemical changes (by FTIR measurements) occurring in the wood surface, using various steaming times (0, 3, 6, 9, 12, 15 and 18 h) in flat and quarter pattern boards in two conditions of moisture content (MC): green and 50%. This work will allow to establish the best conditions regarding steaming time and MC of *Tectona grandis* with different grain patterns.

2. Materials and Methods

2.1. Provenance of the wood, sampling and moisture condition

For the present study, 11-year-old trees from a second thinning intervention in a plantation owned by Aserradero S&Q, located in Rincón de Osa in the province of Puntarenas, Costa Rica (8°40'38" N; 83°29'43" W), were used. *Tectona grandis* 11 years old with 3 × 3 m spacing (1100 trees·ha⁻¹). Stand

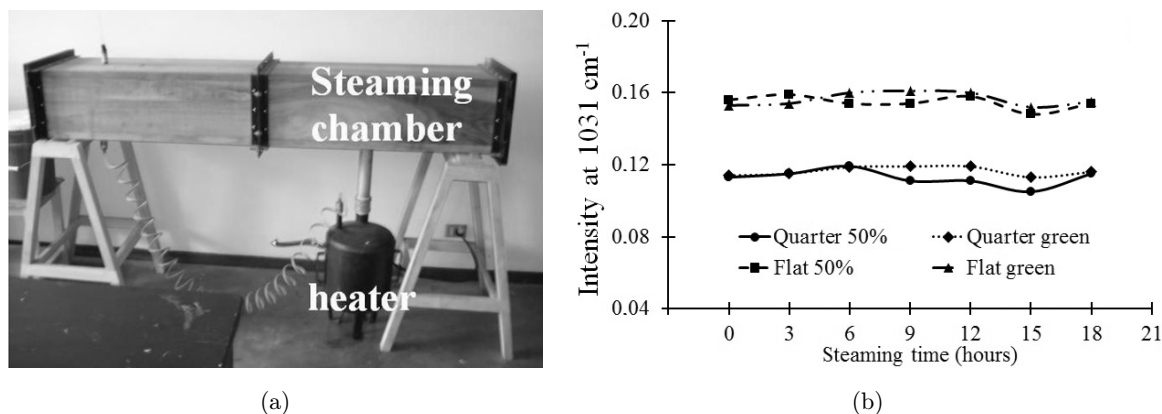
Surface Chemical and Color Characterization of Steam-Dried *Tectona grandis* Wood

Fig. 1. (a) Pilot steaming chamber utilized and (b) intensity at 1031 cm^{-1} band at different steaming times in *Tectona grandis* wood with flat pattern and quarter pattern with different steaming times.

density was $475\text{ trees} \cdot \text{ha}^{-1}$, with an average diameter at breast height (DBH) of 23 cm and 14 m height. The heartwood percentage at DBH varied from 65% to 80%. Approximately, four 2.5 m long logs were extracted from the selected trees. Nine trees were selected from plantation because this number of trees are typically used for determining wood properties.¹³

The logs were sawn using a grain pattern to produce 25-mm-thick boards, allowing for flat-grain- and quarter-grain-patterned boards. About 120 boards were selected, out of which 60 were flat-grain- and the remaining 60 were quarter-grain-patterned. The boards from each grain pattern were then separated into two groups of 30 boards each. One group patterned green condition — was stored to retain the moisture. A second group was air-dried to reach 50% MC. Once both conditions of MC were reached, seven test samples approximately 2.5 cm in width and 35 cm long were extracted from each one of the boards.

2.2. Steam-drying treatment

For each steam-drying test with water, 30 test samples of each grain pattern and moisture condition were used (7 steaming times \times 2 grain patterns \times 2 moisture conditions \times 30 samples = 840 samples). A steam pilot wooden chamber (200 cm \times 30 cm \times 30 cm) was used (Fig. 1) and the steam was provided by a 19-l water-heating tank. An electric resistance was employed to boil the water (Fig. 1).

Steam-drying treatments are described in Table 1; about seven different treatments were applied. For each grain pattern and for each moisture condition, four different baths were conducted. The treatments consist of one control sample and six treatments including steaming of the wood in six different drying times, with a difference of 3 h between them (Table 1). First, water steam was added into the chamber for a conditioning period of 3 h during which the chamber reaches approximately 70°C . The wood to be steam-dried is separated into each grain pattern and moisture condition in seven packages of 30

Table 1. Description of the steam-drying treatments applied to *Tectona grandis* wood in plantations.

No.	Steaming time (h)	Descriptions
1.	0	Control treatment, 0 h steaming and samples dried to 12% MC.
2.	3	Wood was steamed for 3 h and dried to 12% MC.
3.	6	Wood was steamed for 6 h and dried to 12% MC.
4.	9	Wood was steamed for 9 h and dried to 12% MC.
5.	12	Wood was steamed for 12 h and dried to 12% MC.
6.	15	Wood was steamed for 15 h and dried to 12% MC.
7.	18	Wood was steamed for 18 h and dried to 12% MC.

Notes: About 3 h of conditioning was applied in all treatments before initiating the steaming.

A. Berrocal et al.

Table 2. Summary of FTIR bands observed between 800 cm^{-1} and 1800 cm^{-1} in steam-treated *Tectona grandis* wood surface.

S. No.	Position (cm^{-1})	Peak assignments	Structural polymers
Peaks present in all <i>Tectona grandis</i> samples			
1.	810	C=O	Glucomannan
2.	1031	C–O stretch	Cellulose and hemicellulose
3.	1053	C–O stretch	Cellulose and hemicellulose
4.	1108	Aromatic skeletal and C–O stretch	Polysaccharides and lignin
5.	1158	C–O–C vibration	Cellulose and hemicellulose
6.	1231	C–O of syringyl ring	Lignin
7.	1318	C–O vibration	Lignin
8.	1373	C–H deformation	Cellulose and hemicellulose
9.	1419	C–H in-plane deformation with aromatic ring stretching	Lignin
10.	1453	CH deformation, asymmetry in CH_3 and CH_2	Cellulose
16.	1506	Aromatic skeletal vibration (C=C)	Lignin
19.	1595	Aromatic skeletal vibration (C=C)	Lignin
24.	1652	Conjugated C=O in quinones coupled with C=O stretching of various groups	Hemicellulose
25.	1717	C=O stretching	In the carboxylic acid in lignin
26.	1733	Conjugated C=O	Xylan in hemicelluloses
Peaks present in all quarter <i>Tectona grandis</i> wood samples			
	1616	Aromatic skeletal vibration (C=C)	Tannin
	1635	Aromatic skeletal vibration (C=C)	Tannin
	1675	C=O stretching in conjugated aromatic	Lignin

Source: Li et al.¹⁸ and Bonifazi et al.²³

boards each. The control package is left without steam and the other six packages are introduced into the chamber once it has been conditioned and steaming continues. Every 3 h, one package is taken out of the chamber and the wood is dried to reach 12% MC. The steaming time was selected according to previous research in tropical species.¹⁶

2.3. Measurement and analysis of the FTIR spectra of the surface of the wood

Once the steam-drying treatment with different times (0, 3, 6, 9, 12, 15 and 18 h) is finished for each grain pattern, three different boards were taken randomly from each time and grain pattern and two small samples were extracted from the surface of the board. Their dimensions (width \times length \times thickness) were $1\text{ cm} \times 1\text{ cm} \times 2 \times 1\text{ mm}$ (this part is in the surface of the board). The FTIR spectra of the three samples were measured on the surface of the wood by means of a Nicolet 380 FTIR spectrometer (Thermo Scientific) using a single reflectance ATR cell (equipped with a

diamond crystal). All data were recorded at room temperature, in the spectral range of $4000\text{--}700\text{ cm}^{-1}$, by accumulating 64 scans with a resolution of 1 cm^{-1} .

The FTIR spectra obtained were then processed by the softwares Spotlight 1.5.1, Hyperview 3.2 and Spectrum 6.2.0 developed by Perkin Elmer, Inc. Baseline correction was applied at $1800\text{--}800\text{ cm}^{-1}$ and the main components in this vibration band were identified. This band was selected as several studies have identified it as the range where the variation in the changes of the surface of the wood mostly occurs,^{17,18} which are described in Table 2. The height of each peak for each steaming time was recorded and standardized taking the I_{1031} band as a reference, given its stability in all the conditions studied. The ratio between the various peaks in the range of $800\text{--}1800\text{ cm}^{-1}$ (Eq. (1)) was then calculated, as well as the band at 1031 cm^{-1} (1) all times and different grain patterns. This band was selected because large differences were not observed among treatments. The intensity was 0.155 cm^{-1} in wood with flat pattern and 0.118 cm^{-1} in wood with radial pattern with different steaming times (Fig. 1(b)).

Ratio of intensity (I_n/T_{1031})

$$= \frac{\text{Intensity of peaks between } 1800 \text{ to } 800 \text{ cm}^{-1}}{\text{Intensity of } 1031 \text{ cm}^{-1}}. \quad (1)$$

2.4. Surface color measurement and determination of color change

For all conditions of MC and grain pattern, color was evaluated before the steam-drying treatment. Wood surface color was also determined after each steam-drying treatment once the wood reached approximately 12% MC. The MiniScan XE Plus¹⁹ spectrophotometer was utilized to obtain the values of the standardized chromatological system CIEL*a*b*. The range for this measure is from 400 nm to 700 nm, with 11 mm opening at the point of measurement. The observation of the reflection included the specular component (SCI mode), at an angle of 10°, which is normal for the surface of the specimen (D65/10); a visual range of 2° (Standard observer, CIE 1931) and an illumination standard of D65 (corresponding to daylight at 6500 K).

In the analysis of color change, the change in the color parameters (L^* , a^* , b^*) was calculated first with the aid of Eq. (2).

$$\Delta P = P_b - P_a, \quad (2)$$

where ΔP = represents the absolute value of wood color parameters (L^* , a^* or b^*) change between after and before steaming process. P_b is the wood color parameters (L^* , a^* or b^*) after the steam-drying treatment and P_a is the wood color parameters (L^* , a^* or b^*) before the steam-drying treatment.

Following, color change was determined, utilizing the parameter ΔE^* calculated according to the ASTM D 2244 standard²⁰ whose formula is detailed in Eq. (3). The color difference (ΔE^*) was determined for (i) color change that occurred in the wood surface, taking the color before steam-drying treatment as a model, (ii) color change after the steam-drying treatment and (iii) the surface color of wood coming from the natural forest and mature trees,⁵ aimed at establishing the treatment with the lowest color difference with respect to commonly commercialized wood of natural forests (over 100-year-old).

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

where ΔE^* = wood color difference, $\Delta L = L^*$ before steaming $-L^*$ after steaming; $\Delta a = a^*$ before steaming $-a^*$ after steaming and $\Delta b = b^*$ before steaming $-b^*$ after steaming. For color change in (ii), the values of L^* , a^* and b^* after steaming were substituted by $L^* = 44.94$, $a^* = 12.44$ and $b^* = 24.26$, which correspond to the color parameters measured in the wood coming from the natural forest.

2.5. Statistical analysis

In each grain pattern (flat or quarter pattern), a variance analysis (ANOVA) was applied. The aim was to know whether differences in the parameter before and after steam-drying treatment exist. The model included the following sources of variation: steaming time (t) at seven levels (0, 3, 6, 9, 12, 15 and 18 h), MC at two levels (green and 50%) and interaction between t and MC. The SAS GLM procedure (SAS Institute, Inc.) was used to conduct the analysis of variance.

In addition, a *forward stepwise* analysis was applied to determine the effects of the three color parameters Δa^* , Δb^* and ΔL^* on the teak wood color before and after steam-drying treatment, with wood from natural forests as a model. For the FTIR spectra, a scatter plot and then regression analysis were conducted taking into account the steaming time (x-axis) and the intensity ratio values (y-axis). The change in the intensity ratio peak assignment was thus observed for the different steaming times.

3. Results and Discussion

3.1. Surface chemistry of steamed *Tectona grandis* wood measured by means of the FTIR spectra

Although the entire range of the spectra (4000–400 cm^{-1}) was not presented, signals were present in all the steaming periods and grain patterns in the region 3500–2500 cm^{-1} , corresponding to stretching of O-H group (close to 3400 cm^{-1}), C-H and CH₂ asymmetric and symmetric stretching (2940 cm^{-1} and 2906 cm^{-1} , respectively) of the combination of cellulose, hemicelluloses and lignin.^{21,22}

A. Berrocal *et al.*

Peak assignments varied slightly in each type of wood (flat or quarter grain) and MC (green and 50%) in the band studied ($1000\text{--}1800\text{ cm}^{-1}$). Table 2 summarizes in its first part the common peaks for different types of wood treatment and presents the peak assignments, as well as to which polymer they are assigned, based on reports from Moore and Owen,²¹ Li *et al.*¹⁸ and Bonifazi *et al.*²³

The infrared spectra of the different treatments studied had revealed that the positions of most bands and their intensities in the fingerprint region are similar while some are slightly different (Fig. 2). It was found that the chemical components with the signal at 810, 1035, 1053, 1108, 1158, 1231, 1318, 1373 (C–H in-plane deformation for polysaccharides), 1419 (C–H in-plane deformation), 1453 (C–H deformation and aromatic skeletal vibrations), 1506 (aromatic skeletal vibrations), 1558, 1595 (aromatic skeletal vibrations), 1652 (conjugated carbonyl), 1683, 1700, 1717 and 1733 cm^{-1} (stretching of the carbonyl group C=O) are present in all surfaces in the different treatments (Figs. 2(a) and 2(b)), while chemical components with the signal at 1540, 1554, 1575, 1616, 1635, 1675 and 1695 cm^{-1} were observed in the FTIR spectra of the radial samples when the wood was not steam-treated (Figs. 2(c) and 2(d)).

As for the variation of the intensity ratio for each one of the peaks within the band of $800\text{--}1800\text{ cm}^{-1}$, it was observed that the signal at 1158, 1231, 1373 and 1419 cm^{-1} did not show any trend in the index, due to the treatment of the wood with different steaming times and grain patterns of the boards. Meanwhile, the signals at 1053, 1108, 1453, 1506, 1536, 1558, 1595, 1652, 1683, 1700 and 1733 cm^{-1} showed diminution of the index of vibration regarding the steaming time (Figs. 3(a), 3(b) and 3(d)–3(l)). The only vibration where the index augmented was 1318 cm^{-1} (Fig. 3(c)). In the same way, the variation coefficient varied from 10% to 22% at different intensities (Table 3) and any tendency was not found.

As for the signals at 1540, 1554, 1575, 1616, 1635, 1675 and 1695 cm^{-1} , present in the FTIR spectra of the quarter pattern samples of wood not treated with steam, they diminished or disappeared once the wood was steam-treated (Figs. 3(c) and 3(d)).

As for the bands associated to cellulose (1053 , 1373 , 1158 and 1453 cm^{-1}), a decrease in the signals in the band between 1053 cm^{-1} and 1453 cm^{-1} (Figs. 3(a) and 3(d)) was observed, while the signals

at 1318 cm^{-1} increased (Fig. 3(c)). Meanwhile, the signals 1373 and 1158 cm^{-1} did not show any modifications. The diminutions observed in the cellulose indicate either the occurrence of changes in its structure or the formation of other compounds, such as the formation of aliphatic alcohols during steaming,¹⁸ which becomes evident in the change of the intensity at 1055 cm^{-1} .

A major aspect to emphasize of the steaming process in *Tectona grandis* wood, is the variation in the intensity of the signals at 1053 cm^{-1} (Fig. 3(a)) and the increment in the intensity at 1318 cm^{-1} (Fig. 3(c)). These changes are designated to increase the glucose ring stretching vibration, and may be due to the cleavage and dehydration of amorphous carbohydrates and/or crystallization of the paracrystalline region of cellulose.¹⁴ The increment in the intensity ratio in the signal at 1318 cm^{-1} (Fig. 3(c)) indicates a decrease in the percentage of cellulose crystallinity.¹⁸ This reveals that the amorphous regions of the teak wood increase with the steaming time.¹⁴

The changes in the intensity of the signals associated with hemicelluloses and lignin — shown in peaks at 1733 cm^{-1} (O-acetyl-4-O-methylglucurono-xylan) (Fig. 3(l)), and 1595 cm^{-1} (belonging to C=O stretching vibrations in the carboxyl group of glucuronic acid unit in xylan) (Fig. 3(h)) — to the aromatic skeletal vibrations plus C–O stretch of lignin²⁴ and to vibrations caused by the equatorially aligned hydrogen at the C₂ atom in the mannose residue of glucomannan,¹⁵ clearly indicating the changes in the structure of the hemicelluloses and the structure of the lignin of teak wood as a result of steaming.

There is an important reference at 810 cm^{-1} intensity. Any signal was found in this intensity for quarter, flat or moisture conditions (Fig. 4). According to Guo *et al.*,¹² in softwood species, the 810 cm^{-1} , correspondent to C=O in the O=C–OH group of the glucuronic acid unit of the glucomannan band, decreased by 47% in steam wood. They affirmed that this chemical change might be related to the effect of the compression which presumably created more porous structures in earlywood due to the heavy distortion of the cell wall but caused a closure of lumens in latewood. Such pores, small cracks and more open lumens would facilitate penetration of steam, leading to higher degradation of hemicellulose structures in the earlywood.¹² However, this structural change was not found in *Tectona grandis*, showing with this

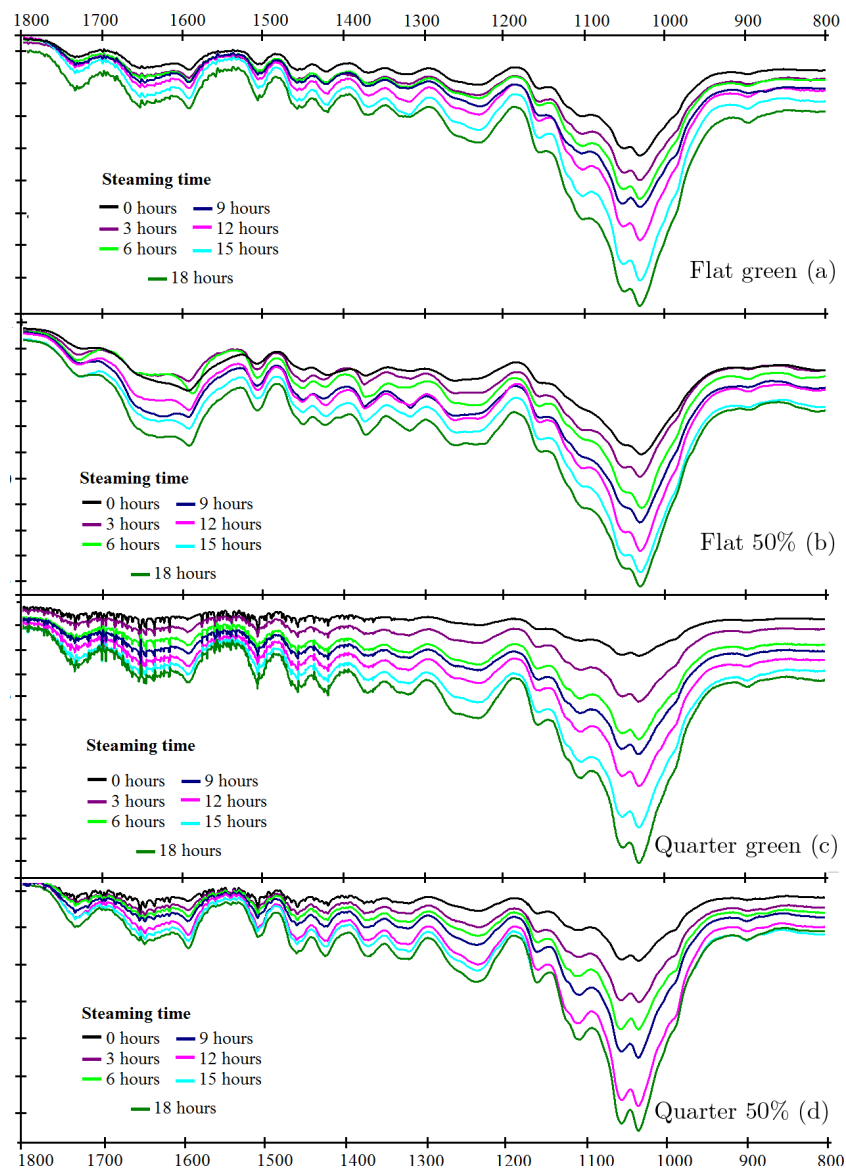
Surface Chemical and Color Characterization of Steam-Dried *Tectona grandis* Wood

Fig. 2. FTIR spectra of steamed *Tectona grandis* wood with flat pattern (a) and (b) and quarter pattern (c) and (d) with different steaming periods.

result the differences between softwood and hardwood species.

Nevertheless, those changes behaved differently with the steaming time and the grain pattern. For example, with the change of intensity at 1595 cm^{-1} (Fig. 3(h)), associated to lignin, the main change occurs at 3 h of steaming, with no more changes thereafter. On the other hand, regarding the intensity associated to xylan, an increment in the ratio I_{1733}/I_{1031} is observed with the steaming time (Fig. 3(l)), indicating further change in the structure of this hemicellulose due to the steaming process.

The intensity of vibration of the xylan band at 1453 cm^{-1} (Fig. 3(d)) ascribed to CH_2 symmetric bending on the xylose ring¹⁵ only showed small changes for the different steaming times. Because the 1456 cm^{-1} peak, associated to the xylan backbone, was nearly unaffected, it is probable that no major degradation of the xylan backbone had occurred, and that the primary effect on the xylan was a side group splitting.¹²

The larger decrease in the relative signal between 1596 cm^{-1} to 1733 cm^{-1} (Figs. 3(h)–3(l)), especially during the first 3 h of steaming of the flat pattern

A. Berrocal et al.

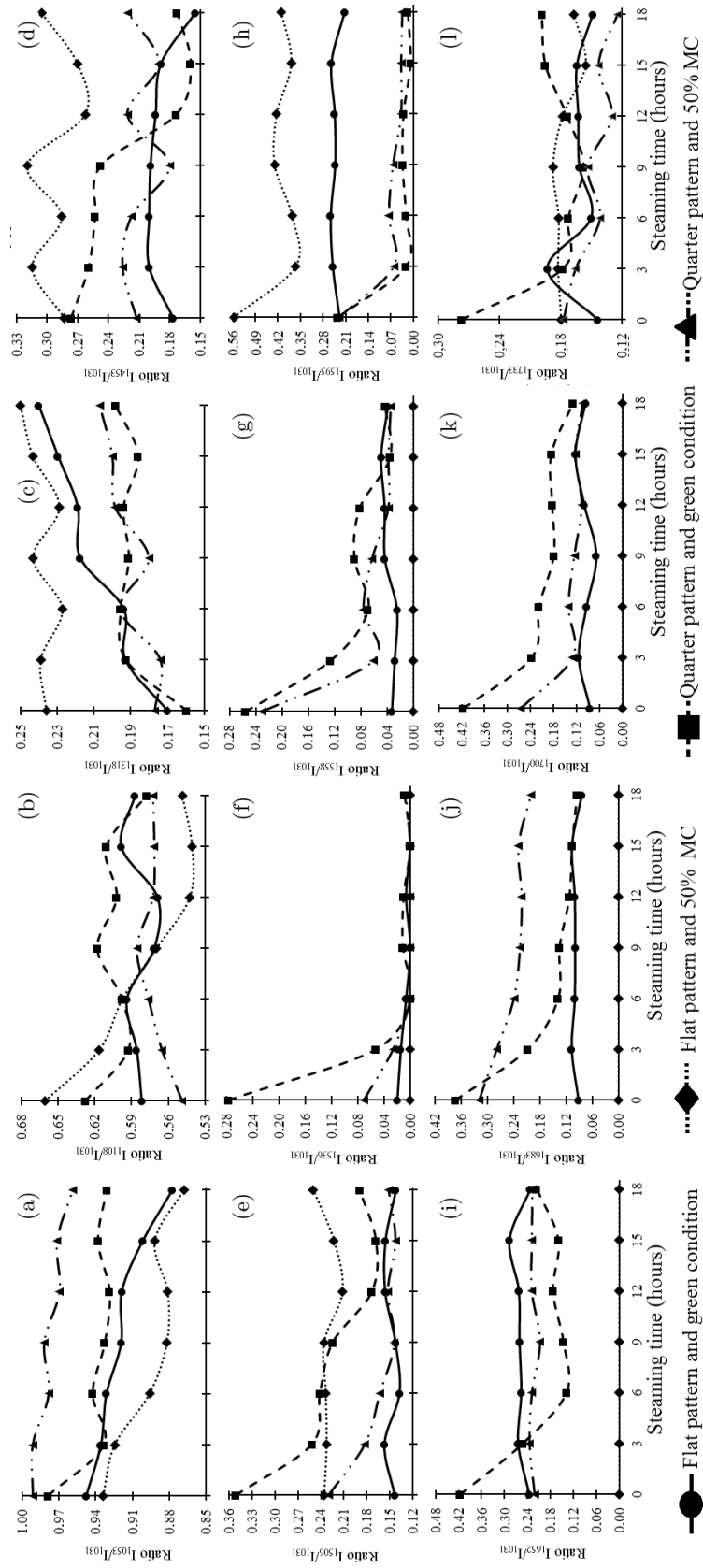


Fig. 3. Changes in the ratio of intensity (I_n/T_{1031}) band at different steaming times in *Tectona grandis* wood with flat pattern and quarter pattern with different steaming times.

Surface Chemical and Color Characterization of Steam-Dried *Tectona grandis* WoodTable 3. Coefficient of variation (%) of change in the ratio of intensity ($I_n/T1031$) band at different steaming times in *Tectona grandis* wood with flat pattern and quarter pattern with different steaming times.

IT	TS(h)	Radial grain		Tangential grain		IT	TS(h)	Radial grain		Tangential grain	
		50%	Green	50%	Green			Green	50%	Green	50%
1053	0	16	23	21	17	1108	0	19	23	13	20
	3	15	14	14	19		3	15	16	15	20
	6	22	20	15	24		6	15	32	16	23
	9	19	29	15	22		9	12	30	13	22
	12	18	24	13	20		12	18	26	20	24
	15	9	18	14	31		15	12	19	19	35
1318	18	17	21	16	22	18	12	23	23	21	
	0	17	10	11	21	1453	0	22	15	21	18
	3	23	13	15	15		3	21	17	21	20
	6	20	24	17	22		6	20	16	20	18
	9	20	21	17	18		9	16	17	18	19
	12	20	15	18	24		12	22	18	20	21
15	18	15	13	21	15		16	17	18	17	
1506	18	18	14	14	21	18	18	21	22	20	
	0	16	12	24	21	1536	0	15	15	17	18
	3	12	19	19	20		3	21	15	16	16
	6	19	17	17	20		6	22	16	18	17
	9	16	22	26	23		9	19	14	20	20
	12	25	19	19	24		12	16	17	15	13
15	17	17	22	21	15		22	20	17	16	
1558	18	14	25	24	21	18	19	19	12	14	
	0	15	12	15	12	1595	0	15	12	15	17
	3	13	11	15	10		3	10	15	17	19
	6	12	11	14	10		6	13	17	18	18
	9	11	11	12	13		9	17	18	16	16
	12	14	13	15	11		12	14	11	16	13
15	11	13	13	13	15		19	14	15	18	
1652	18	11	12	13	9	18	21	12	16	17	
	0	17	13	17	13	1683	0	13	23	17	20
	3	16	12	19	17		3	16	18	18	20
	6	17	17	16	17		6	17	18	18	20
	9	17	16	17	19		9	15	20	20	21
	12	14	11	15	19		12	19	22	22	20
15	15	13	15	19	15		15	21	22	19	
1700	18	18	12	15	16	18	15	21	19	19	
	0	19	22	22	12	1703	0	19	14	16	12
	3	15	19	23	14		3	15	17	13	14
	6	14	19	20	12		6	14	13	17	12
	9	16	15	18	12		9	16	15	18	12
	12	13	21	23	11		12	13	13	13	14
15	19	16	23	12	15		14	16	13	13	
18	14	22	15	14	18	14	13	15	13		

Notes: IT: intensity; TS: time of steaming.

samples, and the steady diminution in quarter pattern samples (Fig. 3(h)), in combination with the lower change in intensity at 1506 cm^{-1} (Fig. 3(e)), indicate that a loss of the C=O group linked to the aromatic skeleton of lignin has probably occurred.

This could indicate that cross-links have been formed between aromatic units in the lignin. Obviously, different behaviors are observed for hemicelluloses and lignin in relation to the degradation under steam conditions. This points to the degradation of

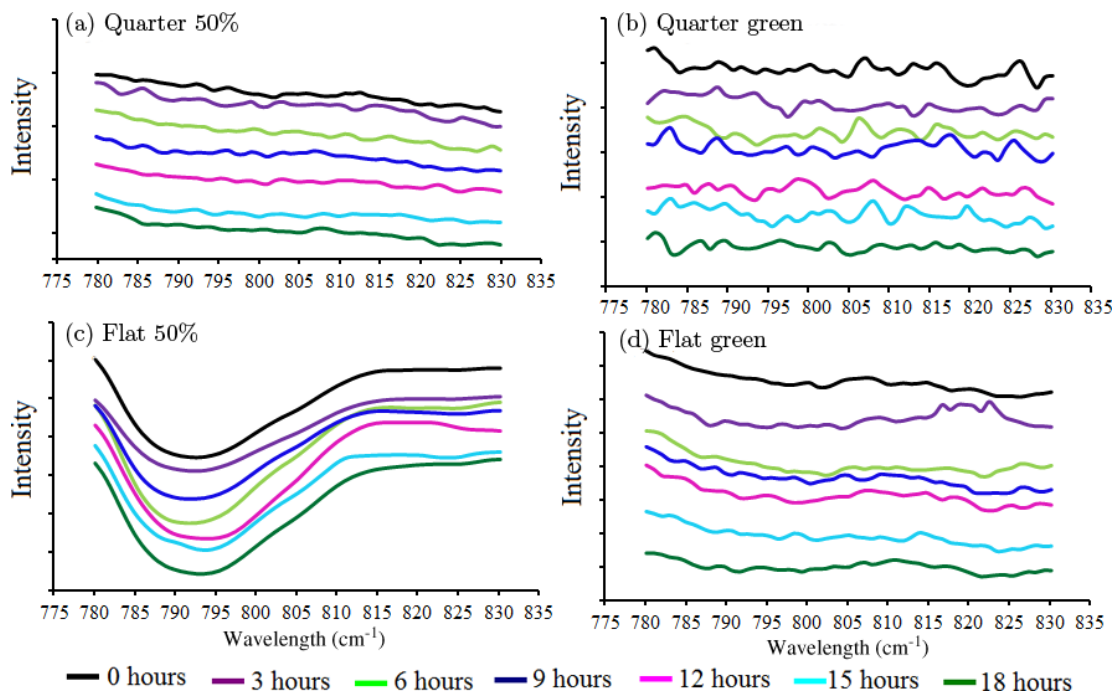
A. Berrocal *et al.*

Fig. 4. FTIR spectra from 775 cm^{-1} to 835 cm^{-1} of steamed *Tectona grandis* wood with quarter pattern (a) and (b) and flat pattern (c) and (d) with different steaming periods.

hemicelluloses and lignin following different pathways. Lignin cross-linking is probably a radical reaction²⁵ which might be favored by the increased density of the wood material while the hemicellulose degradation is probably more favored by the more open access to dissolution of carbohydrates.²⁶

3.2. Color change in *Tectona grandis* surface wood induced by steaming and drying

Wood color before the steam-drying treatment showed differences by grain pattern and MC. Flat pattern boards in the two moisture conditions studied (green and 50%) presented lower values of lightness (L^*) and higher values of redness (a^*) relative to quarter pattern boards. As for yellowness (b^*), the color showed no difference between flat and quarter patterns before steaming (Table 4). Differences in color by grain pattern have been pointed out for several species.^{27,28}

The differences in the color parameters L^* and a^* and the lack of difference among parameter b^* , may be explained by two studies: (i) according to Gierlinger *et al.*,²⁹ redness (a^*) and lightness (L^*) indexes

are more correlated with wood extractive content, while the yellowness index is primarily related to the lignin's photochemistry; (ii) on the other hand, Valverde and Moya³⁰ mention that many extractives settle in the radial parenchyma cells, which means that in a radial surface, color will change more than that in a tangential surface. Therefore, L^* and a^* should be expected to change in green condition, since the extractives are exposed and the lignin still has not begun to photodegrade as the drying process has not started, which explains why the differences in b^* are not observed in flat and quarter pattern boards.

The magnitude of the color parameters changed in the different steaming–drying times (Table 4), L^* in particular, followed by b^* and, to a lesser extent, a^* . The steam-drying treatment increased lightness (L^*) significantly in flat and quarter grain green-condition boards (Table 4); however, when the board presents MC above 50%, the effect is the opposite, significantly reducing L^* .

This behavior is reflected in the differential values of luminosity (ΔL^*). ΔL^* values in flat and quarter pattern steam-dried woods were positive for wood in green condition, as opposed to 50% MC wood, where ΔL^* values were negative (Figs. 5(a) and 5(b)). No

Surface Chemical and Color Characterization of Steam-Dried *Tectona grandis* WoodTable 4. Lab system color parameters of *Tectona grandis* wood before and after the different steaming–drying times.

MC	Color parameters	Time	Steaming time (h)						
			0	3	6	9	12	15	18
			Flat pattern						
Green	L^*	Before	40.1A	41.5A	43.4A	43.7A	42.2A	41.2A	42.7A
		After	55.2B	53.9B	54.2B	53.5B	55.7B	55.5B	54.4B
	a^*	Before	11.7A	10.8A	10.4A	11.1A	11.3A	11.5A	11.1A
		After	11.0A	11.2A	10.4A	9.9A	10.3A	9.9B	9.6B
	b^*	Before	29.0A	28.4A	29.1A	28.9A	29.4A	28.3A	29.4A
		After	27.8A	25.4B	27.0B	26.2B	27.9A	26.5B	25.6B
50% MC	L^*	Before	56.5A	53.5A	54.9A	55.1A	55.4A	57.4A	55.8A
		After	51.2B	50.5B	50.4B	51.7B	51.4B	50.0B	50.2B
	a^*	Before	8.1A	8.0A	8.2A	7.8A	8.0A	7.4A	7.3A
		After	9.4B	9.6B	9.1A	9.4B	9.3B	8.4B	8.5A
	b^*	Before	22.9A	23.8A	25.6A	25.7A	27.6A	27.8A	27.7A
		After	23.8A	25.7B	26.3A	28.6B	29.0A	28.1A	28.4A
			Quarter pattern						
Green	L^*	Before	45.4A	46.6A	43.3A	45.2A	46.3A	45.2A	43.6A
		After	54.5B	57.3B	56.6B	57.5B	56.1B	55.1B	53.7B
	a^*	Before	9.1A	9.4A	10.3A	9.8A	9.2A	9.5A	10.4A
		After	8.7A	8.9A	9.4A	9.0A	8.6A	7.8B	7.6B
	b^*	Before	29.9A	29.4A	28.5A	29.1A	29.7A	29.1A	27.1A
		After	27.8B	27.5B	26.5B	28.1A	27.3B	25.3B	24.3B
50% MC	L^*	Before	61.3A	62.7A	62.5A	62.1A	62.5A	62.2A	61.5A
		After	60.2A	60.5B	60.2B	59.5B	57.5B	56.0B	54.4B
	a^*	Before	8.4A	8.1A	9.1A	9.0A	8.7A	8.5A	8.7A
		After	8.8A	8.3A	8.6A	8.8A	8.2A	8.0A	8.2A
	b^*	Before	27.8A	30.9A	27.8A	27.5A	26.8A	29.5A	29.1A
		After	29.2A	28.6A	28.3A	28.4A	26.6A	25.8B	24.5B

Notes: MC: moisture content.

defined behavior was observed in flat pattern among the different times (Fig. 5(a)). The lowest value of ΔL^* in flat pattern boards was observed during the 9 h of steam-drying and in the 3 h of steaming, for green-condition wood and 50% MC, respectively (Fig. 5(a)). For quarter pattern board, the ΔL^* values were not the lowest, after 12 h of steaming of the green-condition wood, while an increase in the steaming time in 50% MC wood was observed (Fig. 5(b)).

Meanwhile, the parameter of redness (a^*) was statistically unaffected in flat and quarter pattern boards in green condition between 0 h and 12 h of steaming–drying. Nevertheless, in the 15- and 18-h steaming treatment, the parameter a^* decreased significantly in both types of grain pattern (Table 4). This is reflected in the fact that the largest differential redness value (Δa^*) was observed at those steaming

times in the two types of grain pattern (Figs. 5(c) and 5(d)). Steam-drying results vary between flat and quarter patterns when the wood has 50% moisture content. For flat grain boards, the redness value increases statistically with any steaming–drying time, while in quarter pattern boards, this color parameter was not statistically affected (Table 4). Again, this behavior may refer to the changes in the differential redness values (Δa^*), which were positive (above one) for flat pattern boards at all steaming times (Fig. 5(c)). In the quarter pattern boards, the same values were negative, without exceeding 0.5 (Fig. 5(d)).

The color parameter b^* decreased statistically after steaming–drying for both grain patterns in green condition, but for the wood with 50% MC, it was only affected statistically in the flat pattern boards that have been subjected to the steam-drying treatment during 3 and 9 h of steaming and in

A. Berrocal et al.

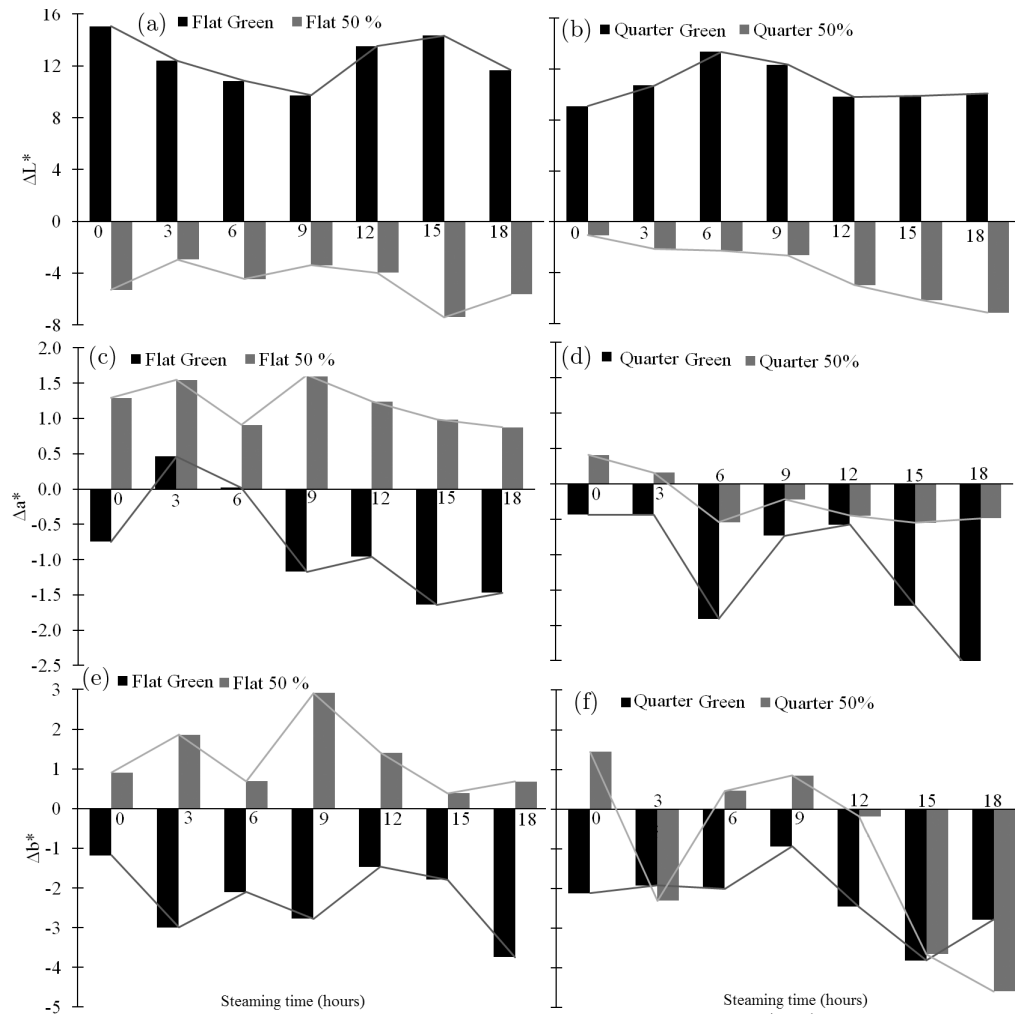


Fig. 5. Color differentials of parameters L^* , a^* and b^* before and after steam-drying *Tectona grandis* flat and quarter pattern boards in two moisture conditions (green and 50% MC).

quarter pattern boards between 15 h and 18 h of steaming (Table 4). The differential change of yellowness (Δb^*) showed that the greatest differentials are observed at 3 and 9 h in flat pattern board, and after 15 h of steaming in quarter pattern boards (Figs. 5(e) and 5(f)).

The behavior of the color parameters is compared to other studies, such as Salas and Moya¹¹ and Basri *et al.*,³¹ which were conducted under similar moisture or grain pattern conditions. The above mentioned authors found that L^* and b^* diminished statistically when the wood is dried, whereas a^* was statistically unaffected. The results were only congruent for 50% MC wood and not for flat pattern (Table 4) or green-condition wood.

In the evaluation of the surface color change (ΔE^*) after the steam-drying treatment, compared

to the teak from natural forest, a minor change was obtained in the flat grain wood between 3 h and 6 h steaming-drying, for both moisture conditions. Furthermore, color change tends to decrease in quarter pattern boards with increasing steaming-drying time (Fig. 6).

The wood color difference index ΔE^* (Eq. (1)) is expressed as a distance between two points in the color coordinate system, with the quadratic addition of each coordinate difference.²⁰ Cui *et al.*³² mentioned that the color's change value (ΔE^*) defined the levels at which color differences are perceived. When the values of ΔE^* rise above 10, color change is very appreciable. Consequently, it is preferable to find a steaming condition with the lowest ΔE^* values relative to teak wood from natural forests⁵ in order to

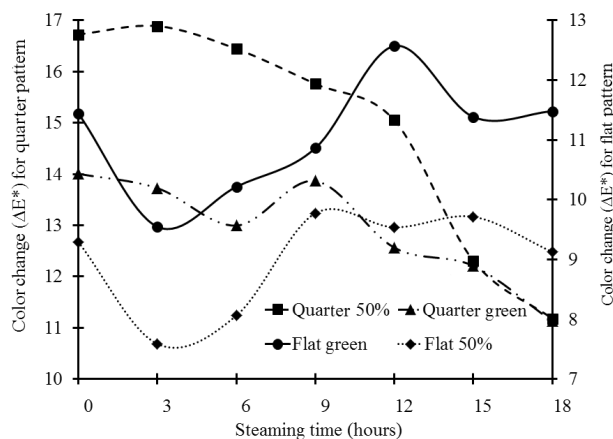


Fig. 6. Color change (ΔE^*) of *Tectona grandis* wood surface of flat and quarter pattern boards, in green condition and 50% MC after different steaming–drying times.

achieve the desirable color. According to the ΔE^* values found with the different steaming times and grain patterns (Fig. 6), the lowest color difference between steamed wood and model teak is achieved after 3 h of the steam-drying of the flat pattern boards at 50% moisture content and 6 h of steam-drying of the green condition wood. Meanwhile, for quarter pattern boards, the best condition is 18 h of the steam-drying (Fig. 5).

Color changes relative to changes in the modifications of the chemical composition of the surface of flat pattern boards, coincide with the diminution of the relative intensity between 1596 and 1733 cm^{-1} (Figs. 3(h)– 3(l)) and the lowest value of the wood color difference index ΔE^* in this type of grain pattern (Fig. 6). As for the quarter pattern board, with the constant change of the ratio of intensity (ratio I_n/I_{1031}), it also coincides with the decrease of the color change with the steaming time (Fig. 6). This means that color changes of the wood probably occur due to the loss of the C=O group linked to the aromatic skeleton of lignin. Specifically, cross-links have been formed between aromatic units in the lignin. However, hemicellulose degradation may also be occurring. Glucose, for example, showed alteration in the signals at 1318 cm^{-1} (Fig. 3(c)) or xylan, indicated by the alteration in the bands at 1453 cm^{-1} (Fig. 3(d)), which favor dissolution of carbohydrates during steaming,³¹ leaving it more exposed to lignin. As a result, redness (a^*) in the wood tends to be higher in the case of flat green wood after 3 h of

steaming (Fig. 3(c)), and the differences in the values of Δa^* in quarter pattern boards after 18 h of steaming are greater.

4. Conclusion

The FTIR bands at 1031, 1053, 1108, 1158, 1231, 1318, 1373, 1419, 1453, 1506, 1558, 1595, 1652, 1717 and 1733 cm^{-1} signals in the range studied (800–1800 cm^{-1}) were found to be present in the surfaces of all the different treatments. On the other hand, the chemical components with signals at 1540, 1554, 1575, 1616, 1635, 1675 and 1695 cm^{-1} were evidenced in the FTIR spectra of the quarter pattern samples of wood not subjected to steaming, therefore, were only present in wood before steaming. Regarding the signals at 1158, 1231, 1373 and 1419 cm^{-1} , it was not possible to observe any trend in this intensity; however, bands at 1053, 1108, 1453, 1506, 1536, 1558, 1595, 1652, 1683, 1700 and 1733 cm^{-1} showed a decrease in the vibration ratio with the steaming time. The only signal where this ratio augmented was at 1318 cm^{-1} probably due to the reduction of the cellulose crystallinity by the steaming–drying process, however further research is required to confirm it.

Different steaming–drying times changed the magnitude of the color parameters, L^* in particular, followed by yellowness (b^*) and then by redness (a^*). The evaluation of the color change of the surface due to steaming–drying, with teak wood from natural forests as the model, shows that flat pattern boards present the lowest change between 3 h and 6 h of steaming–drying in the two moisture conditions, while in quarter pattern boards, color change tends to diminish with the increase of the steaming–drying time.

Color changes relative to changes in the modifications of the chemical composition of the surface of flat pattern boards, coincide with the diminution of the relative intensity between 1596 cm^{-1} and 1733 cm^{-1} (Fig. 1) and the lowest value of the wood color difference index ΔE^* in this type of grain pattern (Fig. 5). As for the quarter pattern boards, the constant change of the ratio of intensity (ratio I_n/I_{1031}) also coincides with the decrease of the color change with the steaming time. This means that color changes of the wood probably occur due to the loss of the C=O group linked to the aromatic skeleton of lignin.

A. Berrocal et al.

Acknowledgments

The authors wish to thank the *Vicerrectoría de Investigación y Extensión* at the *Instituto Tecnológico de Costa Rica (ITCR)*, and we thank Life Forestry Costa Rica S.A. for providing a sample of their 11-year-old teak trees from their plantations for the present study.

References

1. FAO, *Planted Forests and Trees, Global Planted Forests Thematic Study: Results and analysis*, Working Paper No. 38, FAO, Rome (2006).
2. R. Moya, B. Bond and H. Quesada, *Wood Sci. Technol.* **48** (2014) 411.
3. R. Moya and J. Calvo, *Ann. For. Sci.* **69** (2012) 947.
4. P. K. Thulasidas, K. M. Bhat and T. Okuma, *J. Trop. For. Sci.* **18** (2006) 51.
5. R. Moya and B. Marin, *New Forests* **42** (2011) 3296.
6. K. M. Bhat, *Wood News* **9** (1999) 48.
7. R. Moya and A. Berrocal, *Ann. For. Sci.* **67** (2010) 107.
8. S. Fehér, S. Komán, Z. Börcsök and R. Taschner, *BioResources* **9** (2014) 3456.
9. Y. Cao, J. Jiang, J. Lu, R. Huang, J. Jiang and Y. Wu, *BioResources* **7** (2012) 2809.
10. A. Berrocal and P. Agüero, *Kurú Rev. For.* **7** (2006) 1.
11. C. Salas and R. Moya, *Drying Technol.* **32** (2014) 301.
12. J. Guo, K. Song, L. Salmén, Y. Yin, *Carbohydrate Polym.* **115** (2015) 207.
13. X. N. Huang, D. Kocaeffe, Y. Kocaeffe, Y. Boluk and C. Krause, *Appl. Surf. Sci.* **264** (2013) 117.
14. F. Lionetto, R. Sole, D. Cannoletta, G. Vasapollo and A. Maffezzoli, *Materials* **5** (2012) 1910.
15. J. S. Stevanic and L. Salmén, *Holzforschung* **63** (2009) 497.
16. D. Varga and M. E. Van der Zee, *Holz Roh. Werk.* **66** (2008) 11.
17. L. Calienno, C. Pelosi, R. Picchio, G. Agresti, U. Santamaria, F. Balletti and A. L. Monaco, *Stud. in Conserv.* **60** (2015) 131.
18. M. Y. Li, S. C. Cheng, D. Li, S. N. Wang, A. M. Huang and S. Q. Sun, *Chin. Chem. Lett.* **26** (2015) 221.
19. R. S. Hunter and R. W. Harold, *HunterLab: The Measurement of Appearance* (John Wiley and Sons, New York, 1995).
20. American Society for Testing and Materials (ASTM), ASTM D2244-11: Standard Practice for calculation of Color Tolerances and Color differences from instrumentally measured color coordinates, ASTM, West Conshohocken, PA (2014).
21. A. K. Moore and N. L. Owen, *Appl. Spectrosc. Rev.* **36** (2001) 65.
22. N. H. Okoye, A. N. Eboatu, J. S. Fabiyi, P. I. Udeozo, R. U. Arinzea and S. C. Odinma, *Am.-Eurasian J. Sci. Res.* **9** (2014) 45.
23. G. Bonifazi, L. Calienno, G. Capobianco, A. Lo Monaco, C. Pelosi, R. Picchio and S. Serranti, *Polym. Degrad. Stab.* **113** (2015) 10.
24. M. Åkerholm and L. Salmén, *Polymer* **42** (2001) 963.
25. L. P. Ramos, *Quím Nova* **26** (2003) 863.
26. G. Garrote, H. Domínguez and J. C. Parajó, *Holz Roh. Werk.* **59** (2001) 53.
27. B. Dos Santos, V. Sâmia, G. I. Bolzon and J. L. Monteiro de Matos, *Cerne* **20** (2014) 337.
28. M. Nakamura, Y. Miyake and T. Nakano, *J. Wood Sci.* **58** (2012) 505.
29. N. Gierlinger, D. Jacques, M. Grabner, R. Wimmer, M. Schwanninger, P. Rozenberg and L. E. Pâques, *Trees* **18** (2004) 102.
30. J. C. Valverde and R. Moya, *Color Res. Appl.* **39** (2014) 519.
31. E. Basri, D. Rohadi, T. Priadi and I. Wahyudi, *J. Ilmu Tek. Kayu Tropis* **2** (2004) 57.
32. W. Cui, P. Kamdem and T. Rypstra, *Wood Fiber Sci.* **36** (2004) 291.