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Bending quality of three Brazilian hardwoods modified by different hydrothermal treatments

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TECHNOLOGY OF FOREST PRODUCTS

ABSTRACT

Background: Boiling, steaming, and microwave radiation treatments were used for softening and then manufacturing curved samples using three Brazilian hardwoods. Solid wood samples from *Carya illinoinensis*, *Luehea divaricata* and *Platanus x acerifolia* were characterized for bending quality, bending defects, minimum radius of bending, spring-back and moisture content.

Results: Among the studied woods, *Carya illinoinensis* wood was the most suitable to conform curved samples since 96.7% of these wood samples reached a perfect curved shape. On the other hand, *Luehea divaricata* wood presented the worst behaviour, which was marked by only 36.7% of samples achieving a perfect shape. In general, the studied hydrothermal treatments and exposure times induced similar softening behaviours.

Conclusion: In this sense, microwave heating seems to be a good option when compared to the other pathways, since it is known that this method require low consumptions of both time and energy.

Keywords: Mechanical properties, wood bending, wood softening, microwave treatment, steam bending

HIGHLIGHTS

Boiling, steaming and microwave are appropriated for manufacturing curved samples;
Microwave seems to be the best since it requires less time and energy;
Among the studied woods, *C. illinoinensis* was the most suitable wood to conform curved samples, reaching 96.7% of perfectly shaped curved samples;
L. divaricata presented the worst behaviour, with only 36% of samples with perfect curved shape.

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INTRODUCTION

Hydrothermal treatments can be used to promote chemical and anatomical changes in wood, which may lead to many advantages, such as increases in dimensional stability (Sandberg et al., 2013), colour uniformity (Ayadi et al., 2003), and durability (Boonstra et al., 2007), which are partially attributed to a plasticization mechanism (Rowson Ali et al., 2021). The plasticization of wood also increases its bending capacity and allows the creation of stronger and stiffer curved specimens if compared to those specimens shaped using untreated lumbers (Ozarska and Daian, 2010). Therefore, this mechanism may allow the utilization of certain woods for manufacturing high-value-added products. The plasticization of wood is normally conducted using two main pathways: hydrothermal and chemical processes. Currently, hydrothermal treatments can be considered the most promising treatments for producing curved solid woods by bending processes (De Peres et al., 2016).

A hydrothermal treatment applies heat and moisture to the wood specimen for a certain time, causing some changes in chemical composition and microstructure, which increases the overall wood piece flexibility (Sandberg et al., 2013). The bent shape of wooden specimens remains after the hydrothermal treatment due to cell wall deformations caused by synergistic effects imparted by heat, moisture, and/or external forces. During the hydrothermal treatment, water enters from amorphous regions of the wood cell wall and interacts with cellulose, hemicellulose and lignin, then, these wood compounds are disordered by the heat action and their stable deformed shape remains when the wood is cooled down (Yuhe and Muehl, 1999). The hydrothermal treatments processes can be carried out through different heating methods, such as boiling (Charrier et al., 2002), steaming (Esteves et al., 2007), and microwaving (Ozarska and Daian, 2010), which are applied at variable temperatures (Studhalter et al., 2009), pressures (Rowell et al., 2009), and times (Esteves et al., 2008). The effects of these treatments depend on both the wood species and applied processes, wherein most wood changes are intensified by high temperatures (Biziks et al., 2013).

In boiling operation, the wood fibres are fully impregnated with hot water, causing a lignin plasticization mechanism and, consequently, cell softening (Sandberg et al., 2013). In addition, the hemicelluloses are partially dissolved by the hot water, forming an aqueous extract, that can be converted into valuable chemicals (Gašparík and Barcík, 2013). In steaming, wood pieces are usually placed in a steaming box to introduce moisture at a temperature of around 100 °C. These parts are then bent and kept in the bending device while they are cooled down and acquire their final moisture content (Kuljich et al., 2015). The microwave heating consists of propagating microwaves through water molecules in the wood. Then, the vibratory movement of the active water molecules generates intermolecular friction, vaporizing the moisture inside the wood cell wall, which may collapse due to the high internal pressures developed (Yuhe and Muehl, 1999). Compared to the other treatments, this process has great advantages, such as short plasticizing time, small energy requirement and easy temperature control, which also yields a low cost (de Peres et al. 2016; Yuhe and Muehl, 1999).

These hydrothermal treatments may be adjusted to adequate the wood mechanical properties to perform a good forming or bending. However, the process parameters also must be controlled to avoid the degradation of the lignin–saccharide matrix (Gašparík and Barcík, 2013), since autocatalytic reactions, such as cleavage of acetyl groups, generate carbonic acids and cause hydrolysis of certain amorphous polysaccharides. Besides, hygroscopic polysaccharides (namely hexoses and pentoses) may be converted into less hygroscopic furan-based biopolymers (hydroxymethylfurfural and furfural, respectively), which reduces the equilibrium moisture content by half of that value of an untreated wood. This behaviour facilitates the wood plasticizing since temporarily breaks chemical bonds from the lignocellulosic matrix (Sandberg et al., 2013).

Furthermore, anatomical features of wood are affected by hydrothermal treatments. The greatest effect is related to the fibres, which undergo decreases in cross-sectional area and wall thickness (Biziks et al., 2013). These anatomical changes yield decreases in mechanical properties of the wood, including modulus of elasticity, modulus of rupture, and brittleness (De Peres et al., 2020). These effects are favourable for the manufacture of curved wood samples. Besides the advantages attributed to the wood softening, increase in brittleness, as well as decreases in impact toughness, modulus of rupture and strain of failure are also commonly reported, leading to a high incidence of cracking and splitting (Rowell et al., 2009). Therefore, although the bending process has been used for manufacturing curved wood samples, there is a gap in the literature related to the bending quality of typical hardwoods pre-treated by different hydrothermal processes. Based on that, this work aims to evaluate the bending quality of three Brazilian hardwoods softened by boiling, steaming, and microwave radiation.

MATERIAL AND METHODS

Material selection and sampling

Carya illinoinensis (27-30 years old), *Luehea divaricata* (50-60 years old) and *Platanus x acerifolia* (24-25 years old) adult trees were cut in Santa Maria/Brazil. 5 trees per specie were studied, totalizing 15 trees. Prismatic wood samples with the dimensions of 1 cm × 2 cm × 34 cm (radial × tangential × longitudinal) were cut according to the ASTM D5536 (ASTM, 2010). All clear wood samples were selected without defects, such as knots and cracks, and defects caused by xylophagous organisms. The samples were stored in a climatic chamber (at 65 % RH and 20 °C) until reaching constant mass. Further information from the selected woods can be consulted in previous studies from the research group (de Peres et al., 2016; de Peres et al., 2020)

Hydrothermal treatments

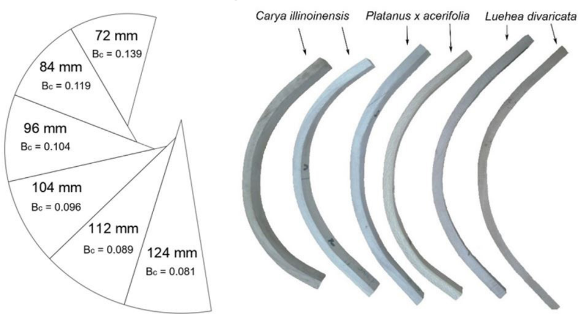
Wood samples were treated by three different softening methods: boiling, steaming, and microwave radiation. The boiling process was carried out at atmospheric pressure using boiling water (~100 °C) and an open tank. The steaming operation was carried out in a plastic box built to allow the entry of steam and the flow out of condensed water, keeping inside the box at atmospheric pressure. Finally, microwave radiation was applied to the saturated (waterlogged) samples, which were previously dried, using a laboratory equipment adjusted for 2.45 GHz frequency and 900 W nominal power. Each hydrothermal treatment was performed for two different exposure times, as shown in Table 1. Ten samples were used for each hydrothermal treatment, totalizing sixty samples per specie. The moisture content in the wood samples was measured before and after each treatment process using the gravimetric method described in ASTM D143 (ASTM, 2014). More information about the hydrothermal treatments can be obtained in previous studies of the group (de Peres et al., 2016; de Peres et al., 2020).

Table 1. Summary of the applied hydrothermal treatments.

Code	Treatment time	Treatment method
B45	45 min	Boiling
B60	60 min	Boiling
S45	45 min	Steaming
S60	60 min	Steaming
M60	60 s	Microwave radiation
M90	90 s	Microwave radiation

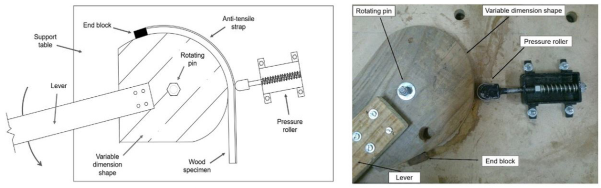
Wood bending and its evaluation

Wood bending was performed after the hydrothermal treatments using a lab-made bending device, which was built based on that proposed by Ozarska and Daian (2010). The device had a variable radius of bending curvature, forming a spiral, which radius decreases progressively from 124 to 72 mm, ensuring that different bending coefficients (Bc) could be applied along to the wood specimen (Figure 1). Bc was determined as specimen thickness (t) divided by radius of curvature (Rc), i.e. $Bc = t/Rc$. A pressure roller endowed with a spring and an anti-traction strap was used to ensure the fixation of the sample (Figure 2). Furthermore, an end block was used to inhibit a longitudinal shear failure.



(a) (b)

Figure 1. Variable dimension shape used in bending process (a) and post-bended wood samples (b).



(a) (b)

Figure 2. Illustrative image (a) and photograph (b) of the built bending device.

Some curved wood specimens presented defects developed due to the bending process, namely crushing, splinting, cross cracking, and perpendicular break (Figure 3). Some small defects in curved wood samples can be fixed by machining and polishing. Based on that, similarly to what was proposed by Ozarska and Daian (2010) and Kuljich et al. (2015), a ranking was created to analyse the bending quality. The defects were classified according to their damage and aesthetic aspect, as shown in Table 2. Those samples that received ranks above 2 were considered as usable samples.

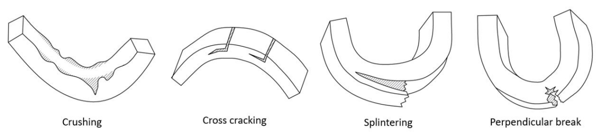


Figure 3. Aesthetical aspects of founded bending defects.

Table 2. Ranking adopted to classify the curved wood specimens. Adapted from Ozarska and Daian (2010).

Ranking	Quality	Description
5	Perfect	Perfect shape; no defects.
4	Good	Small aesthetic defects; Up to two defects by sample.
3	Medium	Moderate aesthetic defects; Up to two defects by sample.
2	Poor	Severe aesthetic defects; Up to two defects by sample.
1	Very poor	Sample collapse; Three or more defects by sample.

The smaller the radius of curvature supported by the sample, the higher the bending quality. Thus, to evaluate this factor, the minimum radius of bending (MRB) of each group was determined as the radius of occurrence of the first defect, i.e. the smallest radius of curvature in which both the inner and outer surfaces are not fractured. Furthermore, when the sample is released from the bending device, there is a loss of its curved shape (LCS), known as spring-back. To determine spring-back (Equation 1), the chord distance between ends of the bent sample was measured for two conditions: when the wood part was fixed in the bending device (D_{fixed}) and 24 h after its release from the bending device, meanwhile the sample was subject to cooling and conditioning in a climatic chamber at 20 °C and 65% relative humidity (D_{cool}).

$$\text{Spring-back} = \frac{D_{cool} - D_{fixed}}{D_{fixed}} \times 100 \quad (1)$$

Where: D_{fixed} is the chord distance between sample ends when the sample was fixed in the bending device and D_{cool} is the chord distance between sample ends 24 h after the bending process.

Statistical analyses

Statistical analyses of the obtained data were performed using Two-Way ANOVA followed by means tests using LSD-Fisher method at a significance level of 5%, in which wood species and hydrothermal treatments were considered as factors. The computational implementation was developed in Python 3.9 programming language.

RESULTS AND DISCUSSION

Bending defects

Figure 4 shows the number of accounted defects considering the 10 samples evaluated per group. The samples from *Carya illinoinensis* wood presented only one defect of splitting and another one of crushing (Figure 4a), whereas the *Luehea divaricata* wood presented 4 types of defects and a total of 80 accounted defects, encompassing all hydrothermal treatments, which indicates that the incidence of bending defects along the bending processes was much more related to wood species than the bending method. Regarding the *Platanus x acerifolia* wood, a total of 35 defects from 3 different types was encountered.

Splitting was the most frequent defect in the studied wood samples (Figure 4) and occurred when the anti-traction strap was removed from the tensile side. Furthermore, cross cracking, perpendicular break, and crushing were also observed. All defects were probably caused by the use of a high bending coefficient, insufficient pressure from the supports or unsuitable adjustments of either the flexion belt or end supports (Burvill et al., 2013). Table 3 shows results obtained by ANOVA for the number of defects per sample. The results indicate that there is no statistical relationship between the type of hydrothermal treatment and the defect incidence, although there was a statistically significant difference between the species.

Table 3. ANOVA table for the number of defects.

Source	Sum of squares	Degrees of freedom	Mean square	F-value
Treatment	3.3	5	0.66	0.62 ^{ns}
Specie	51.1	2	25.55	23.8 *
Interaction				
Treatment x Specie	10.6	10	1.06	0.99 ^{ns}
Residue	173.9	162	1.07	

*Significant at the level of 5% error probability in the LSD-Fisher test; ns = not significant;

According to Cademartori et al. (2013), hydrothermal treatments highly increases wood compressibility, although tensile properties are only slightly affected. This behaviour is undesirable since both compressive strains (the interior face) and tensile strains (exterior face) will occur during a wood bending. The wood species showed a significant effect on the number of defects, as shown in Figure 5. Due to its natural origin and anisotropic nature, wood presents great variability in terms of its oscopic, chemical, mechanical, and anatomical characteristics (Green, 2001).

The ranking of each treatment was determined by the aforementioned visual parameters, wherein all samples ranked 3 or more were considered usable samples. Regardless of the hydrothermal treatment, Table 4 shows that the *Carya illinoinensis* wood obtained the best performance among the studied woods (Figure 3) with 100% of its samples ranked as 3 or more. The *Platanus x acerifolia* wood presented the second-best performance (except for B45), achieving a satisfactory 71.7% of usable samples and more than 50% of samples endowed with a perfect curved shape. The *Luehea divaricata* wood obtained the worst performance since only 61.7% of its samples were considered useable and only 36.7% of its

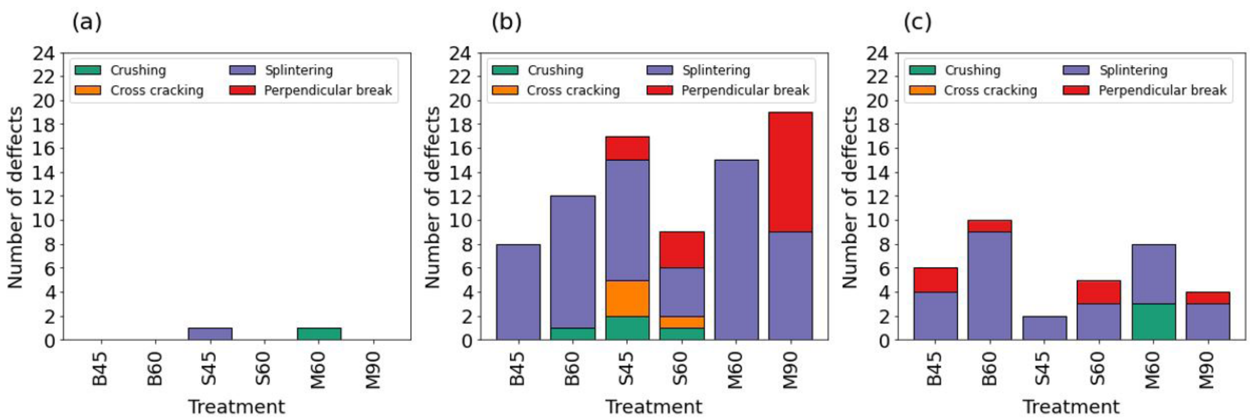


Figure 4. Number of bending defects per group for *Carya illinoinensis* wood (a), *Luehea divaricata* wood (b), and *Platanus x acerifolia* wood (c).

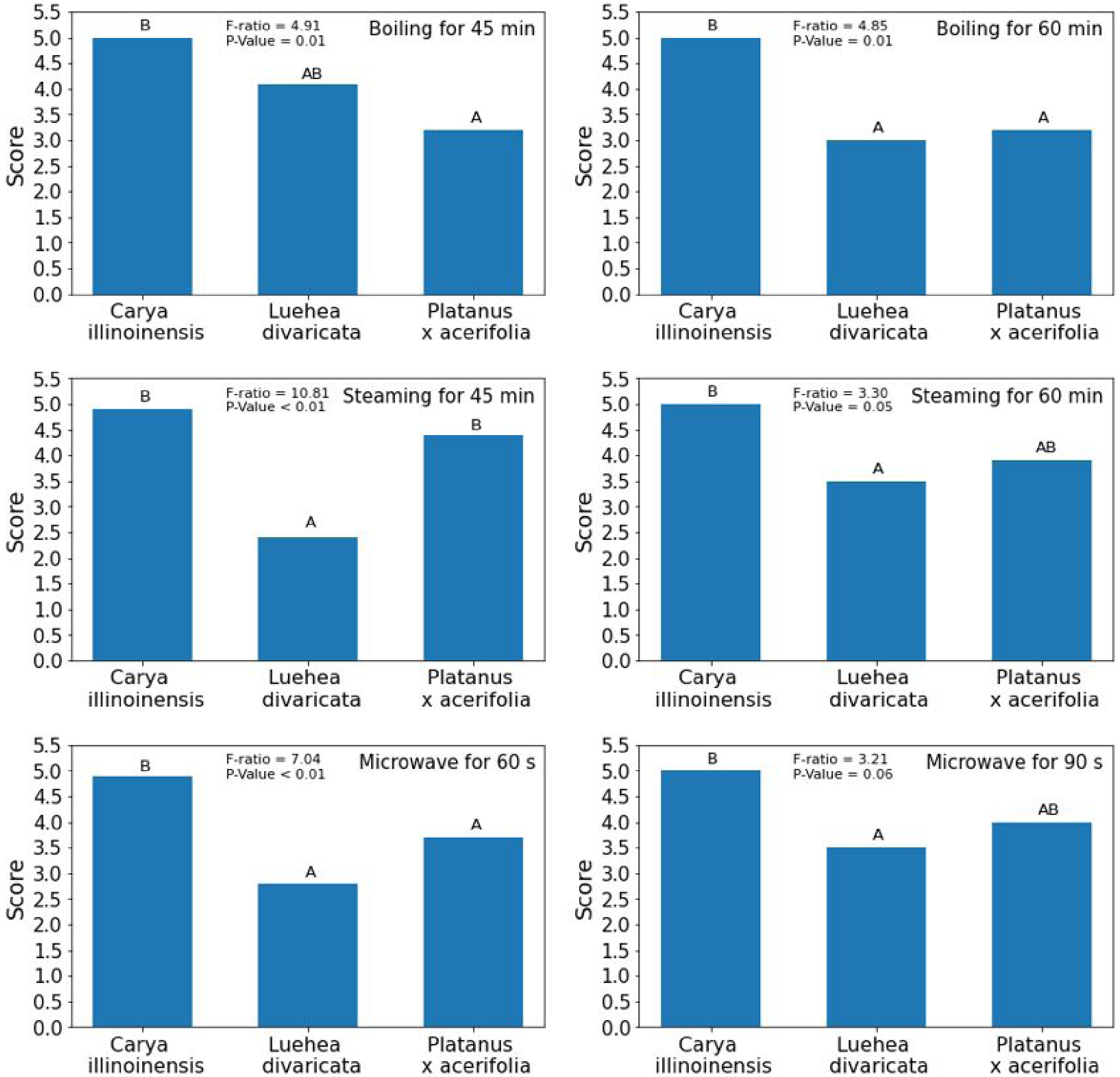


Figure 5. Ranking for bending quality achieved for the studied hydrothermally treated hardwoods.

pieces obtained a perfect bent shape. A high number of defects is detrimental from both economic and environmental standpoints since the higher the number of defects, the higher the cost of repairs and the higher the amount of discarded samples. The percentage of usable samples presented by these species is considerably high when compared with previous studies. In this sense, Kuljich et al. (2015) did not report perfect curved shapes for any of the seven poplar hybrid clones studied by them. On the other hand, Ozarska and Daian (2010) reported only few samples (5-6%) without defects produced using *Acacia melanoxylon* wood and *Eucalyptus saligna* wood. Differences in comparison between the present study and the literature can be attributed to both bending parameters and wood species.

Table 4. Frequency distribution of ranking by each wood species.

Ranking	*Carya illinoinensis*	Luehea divaricate	*Platanus x acerifolia*
5 (%)	96.7	36.7	56.7
4 (%)	3.3	13.3	8.3
3 (%)	-	11.7	6.7
2 (%)	-	11.7	8.3
1 (%)	-	26.7	20.0

Minimum radius of bending (MRB)

Regarding the MRB, no statistical differences were found in a comparison between the hydrothermally treated woods, as shown in Table 5. On the other hand, when considered as a factor, the wood species significantly affected this property. Figure 6 shows the frequency distribution of the radius of curvature for each wood species and Table 6 presents the cumulative frequency of this data. MRB was determined by the radius in which more than 95% of the pieces were satisfactorily bent.

Table 5. ANOVA table for the minimum radius in bending.

Source	Sum of squares	Degrees of freedom	Mean square	F-value
Treatment	8.96	5	1.79	0.76^{ns}
Specie	142.47	2	71.24	30.09*
Treatment x Specie	22.72	10	2.27	0.96^{ns}
Residue	383.50	162	2.36	

*Significant at the level of 5% error probability in the LSD-Fisher test; ns = not significant;

The *Carya illinoinensis* wood reached the best performance, with 98.3% of its samples presenting MRB of 72 mm, which corresponds to the highest bending coefficient. Whereas, the *Luehea divaricata* wood presented the worst performance, with only 38.4% of its samples resisting the higher coefficient of bent. However, sample failures in all the other radius of curvature led to the same MRB if compared to that of the *Platanus x acerifolia* wood (MRB = 124 mm; BC = 0.081).

Both the *Luehea divaricata* wood and *Platanus x acerifolia* wood presented low MRB, these values are similar to the ones obtained by Ozarska and Daian (2010), who studied the bending quality of 8 species pre-treated by microwave radiation. The authors used samples with a thickness of 25 mm and a 260-400 mm radius of curvature range, reaching bending coefficients in the range of 0.063-0.089. The *Carya illinoinensis* wood clearly obtained an exceptional behaviour, resisting a bending coefficient 64% higher than the best one obtained by these authors.

Spring-back

The instantaneous spring-back is a natural reaction of all wood species and occurs due to the release of internal stresses developed during the bending process. Table 7 shows descriptive results on the collected data,

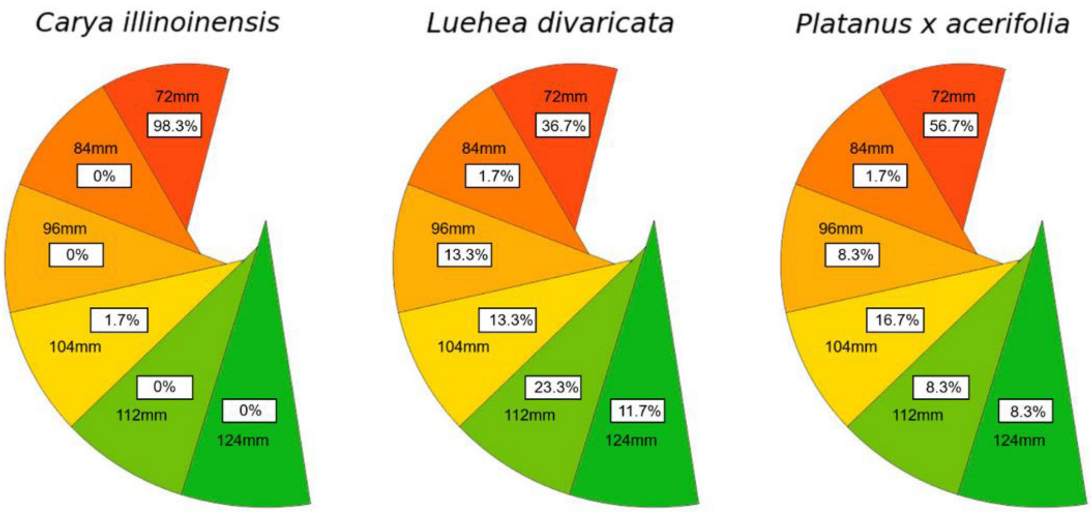


Figure 6. Frequency distribution for the minimum radius of curvatures of the studied woods.

Table 6. Cumulative frequency distribution of minimum radius by species.

Wood species	Cumulative frequency by minimum radius (%)					MRB (mm)	Bending coefficient
	72 – 84 mm	72 – 96 mm	72 – 104 mm	72 – 112 mm	72 – 124 mm		
Carya illinoinensis	98.3	98.3	100	100	100	72	0.139
Luehea divaricate	38.4	51.7	65.0	88.3	100	124	0.081
Platanus x acerifolia	58.4	66.7	83.4	91.7	100	124	0.081

where statistical differences were attributed to both the hydrothermal treatments and wood species. There was no clear trend between spring-back and type of hydrothermal treatment since M90 presented the highest spring-back among the treatments for *Carya illinoinensis*, while it presented the lowest value for the *Platanus x acerifolia* wood. The *Luehea divaricata* wood presented the worst spring-back, i.e. the higher percentage of loss of its circular shape after cooling. Furthermore, among the hydrothermal treatments, M90 provided the worst behaviour in general. The *Carya illinoinensis* wood reached the lowest loss in circular shape, although it presented a high value for M90 (12.07%). The same behaviour was observed for the *Luehea divaricata* wood and, on the other hand, all the hydrothermally treated *Platanus x acerifolia* woods presented low and similar spring-back levels (<6%).

Table 7. Mean values of spring-back by species and hydrothermal treatments.

Species	Treatment	n*	ΔMC (%)	Spring-back (%)
Carya illinoinensis A	B45	10	21.36	3.62 (0.94)** a***
	B60	10	25.26	4.11 (2.80) a
	S45	10	10.65	3.56 (1.32) a
	S60	10	12.75	3.28 (1.09) a
	M60	10	-9.43	4.21 (3.11) a
	M90	10	-14.62	12.07 (6.82) b
Luehea divaricate B	B45	10	34.20	5.66 (2.48) ab
	B60	6	35.96	8.83 (2.19) bc
	S45	6	12.76	5.04 (1.04) ab
	S60	7	15.82	2.68 (1.01) a
	M60	7	-12.65	7.53 (7.06) bc
	M90	8	-22.13	10.51 (3.47) c
Platanus x acerifolia A	B45	6	35.70	3.66 (1.04) b
	B60	8	40.60	4.63 (1.00) c
	S45	9	10.63	5.97 (1.39) d
	S60	8	15.06	4.02 (0.64) bc
	M60	10	-16.21	4.70 (1.05) bc
	M90	10	-26.10	3.11 (0.98) a

* Number of usable samples
** Standard error of the mean
*** Means followed by the same letter are not statistically different

In general, the increases in MC due to the hydrothermal treatments (boiling and steaming) caused increases in plasticity and decreases in spring-back. According to Yuhe and Muehl (1999), the decrease in moisture content of wood negatively affects the instantaneous spring-back, which was seen here for the microwave-treated woods (except for the *Platanus x acerifolia* wood), where the loss of moisture occasioned high levels of spring-back. Unlike other methods, where the softening is performed in a wet environment, there is a release of the imprisoned water in the form of steam in microwave radiation, which decreases its moisture content. The exposure time to microwaves greatly influenced the MC of all woods, in a way that the greater the exposure time, the greater the moisture loss. However, this treatment represents a good alternative for softening wood, since may induce a plasticization mechanism in much less time than the conventional processes.

CONCLUSION

The treatments were efficient for plasticizing the studied hardwoods, although no significant differences were found in the comparison between the treatment in terms of number of defects, quality rank, and minimum radius of curvature. The most prominent variations were attributed to the wood species, the *Carya illinoinensis* wood presented the most suitable bending performance, with a remarkable success rate in bending as 96.7% of its samples obtained a perfect curved shape. Whereas, the *Luehea divaricata* wood presented the worst behaviour of the studied woods, with just 36.7% of perfect samples. The defects that appeared in the wood samples were either due to excessive bending or the accumulation of stresses introduced by the bending device, elucidating that further research may focus on improved bending methods. Finally, the increase in moisture content imparted by the hydrothermal treatments seemed to be a positive factor in the increase found in bending quality. Besides this, compared to the other hydrothermal treatments, microwave radiation can be considered a suitable option to replace traditional methods, since it requires a much lower exposure time and energetic expenses to achieve similar results.

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Authorship contribution

Project Idea: DAG, MLP
Database: MLP
Processing: MLP
Analysis: ABA, RAD
Writing: RAD, ABA
Review: ABA, RAD, DAG

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