Dimensional Stability and Equilibrium Moisture Content of Thermally Modified Hardwoods

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The dimensional stability and equilibrium moisture content (EMC) of thermally modified hardwoods were studied. Lumber of yellow-poplar (*Liriodendron tulipifera*); red oak (*Quercus borealis);* white ash (*Fraxinus americana*), red maple (*Acer rubrum*); hickory (*Carya glabra*), and black cherry (*Prunus serotina*) were modified in industrial thermo-vacuum system. The water absorption rate, EMC, swelling, anti-swelling efficiency, shrinkage, anti-shrinkage efficiency, and anisotropy of the specimens were measured and compared to unmodified wood. The results show that thermal modification significantly decreased water absorption of wood which leads to improved dimensional stability. Specifically, thermally modified wood showed reduced EMC (22% in hickory to 59% in red maple), increased water absorption repellent (14.9% in black cherry to 29.6% in yellow-poplar), increased anti-swelling efficiency (14.2% in hickory to 71.4% in ash), increased anti-shrinkage efficiency (23.5% in red maple to 65.6% in ash), and reduced anisotropy coefficient (4.7% in red oak to 31.9% in black cherry).

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INTRODUCTION

Thermally modified woods (TMW) are extensively being used in different applications such as decking, cladding, flooring, facade battens, and terrace boards (Espinoza *et al.* 2015; Candelier *et al.* 2016), structural elements (Roszyk *et al.* 2020) in exterior applications and humid environments, *etc.* (Shaikhutdinova *et al.* 2017). They are being offered as a sustainable alternative (Bond *et al.* 2023). Thermal modification (TM) consists of exposing wood to high temperatures, which alters the chemical composition of wood irreversibly and impacts its physical and mechanical properties (Tjeerdsma and Militz 2005; Militz and Altgen 2014; Hill *et al.* 2021). Different methods of TM are used in industry, and they differ in medium and schedule.

One of the methods that is currently being used in the market is modification in a vacuum atmosphere. Thermo-vacuum treatment is classified as dry/wet, close/open thermal treatment, and its detailed specification can be found in selected publications (Esteves *et al.* 2008; Allegretti *et al.* 2012; Sandak *et al.* 2015). The dry, open process uses wood with a 12% moisture content, which slowly increases the temperature until it reaches the temperature of 200 to 240 °C in a vacuum atmosphere, and the modification residues are removed from the tank during the process. Different species require different temperatures and different durations of modification. Other methods of wood modification are thoroughly presented in different review papers (Sandberg *et al.* 2017; Zelinka *et al.* 2022).

The main purpose of TM is to acquire wood with better dimensional stability, making it suitable for use in variable moisture conditions, particularly in exterior applications. Modifying wood at high temperatures degrades its main structural components, such as hemicellulose, cellulose, and lignin, as well as extractives. Hemicelluloses and the amorphous region of cellulose degrades, thereby contributing to the increase in the degree of crystallinity of this polymer (Hill *et al.* 2021). In addition, a cross-linkage between the lignin and the polymers occurs because of the thermal degradation of the wood, which is responsible for the decrease in the hygroscopicity and an improvement in their dimensional stability (Vernois 2001; Bekhta and Niemz 2003; Calonego *et al.* 2010). TM changes EMC, which leads to lower dimensional changes when the humidity changes. Significant improvement of the dimensional stability of the wood is due to the reduction of hydrophilic hydroxyl (-OH) groups (Xu *et al.* 2019). Vernois (2001) reported that TMW at 200 °C presents an equilibrium moisture content of 4% to 5% instead of 10% to 12%.

Hardwoods have different chemical and anatomical properties in different plantations (Oladi *et al.* 2013) and more diverse anatomy than softwoods. The Appalachian region in North America is home to several hardwood species. As reported by the *Appalachian Hardwood Species Guide* (2023), yellow poplar (YP) is a fast-growing wood species that represents a significant percentage (35%) of the growth and production in the region. Recently, YP is experiencing high commercial value because of its versatility and use as a substitute for increasingly scarce softwoods in furniture and framing construction. Poplar species contain tension wood that has cells strengthened by a gelatinous (pure cellulose) layer in its structure (Masoumi *et al.* 2023). The oak species are the most important type of hardwood in the region.

One unique feature of Appalachian oak is that fewer sub-species of the oak reach the commercial market, which assures uniform quality for furniture manufacturers, architectural woodworkers, and flooring manufacturers. White Ash (*Fraxinus americana*), which has five common species in the region, is about 3 to 5% of the volume of lumber production. The maples represent about 10% of the standing timber and volume of lumber production, in the Appalachian area only two are important commercially; the sugar maple (*Acer saccharum*) and the red maple (*Acer rubrum*). Hickory, also known as “Appalachian Pecan”, has not been cut in the forest of the Appalachian region in years past, so today it is abundant. Black cherry is 1% of the species in the area, but it is a highly prized wood for furniture. TM can significantly increase the functional value of low-value hardwood species, and as a result, change its position in the market for wood and engineered wood materials.

While the mechanical properties, biological durability, and bonding performance of these species have already been published by Gonzalez *et al.* (2021), Masoumi and Bond (2023), and Masoumi *et al.* (2023), literature in this field lacks data on the physical properties. The goal of this study was to measure the dimensional stability and equilibrium moisture content of thermally modified hardwoods.

EXPERIMENTAL

Sample Preparation

Lumber selected for this study were 1-inch-thick lumber of six different hardwood species of the Appalachian region, yellow poplar (*Liriodendron tulipifera*), red oak (*Quercus borealis (rubra)*), ash (*Fraxinus americana*), red maple (*Acer rubrum*), hickory (*Carya glabra*), and black cherry (*Prunus serotina*).

Thermal modification

Prior to the modification, the lumber was kiln-dried to 6 to 8% MC. Thermal modification of the lumber was performed in an industrial dry-open vessel thermo-vacuum and the maximum modification temperature, and the density of the lumber is presented in Table 1.

Table 1. Modification Temperature and Oven-dried Density for Different Wood Species

Wood Species	YP	Red Oak	Ash	Red Maple	Hickory	Black Cherry
Temperature (°C)	210	195	205	205	205	200
Unmodified Density (g/cm^2)	0.44	0.74	0.74	0.61	0.77	0.56
Modified Density (g/cm^2)	0.37	0.46	0.32	0.45	0.59	0.46

Physical Properties

Unmodified and thermally modified lumber were randomly selected to prepare test specimens. Cubes of each treatment type of every species with dimensions of 1 in × 1 in × 1 in (L × R × T) were cut from the lumber. Physical experiments were conducted based on the ASTM D143-22 standard. Sixty cubes of each treatment type with no sign of split, knot, or irregular grain pattern with appropriate radial, tangential, and longitudinal directions were selected. Specimens were divided into two groups for measuring swelling and shrinkage and labeled with permanent marker indicating their number and species name. Prior to measurement, the specimens were conditioned by placing them in a climatic chamber that was adjusted to 21 °C and 65% relative humidity (RH), which is equal to 12% RH for 20 days until unmodified specimens reached the equilibrium moisture content.

After conditioning, the samples were weighed, and their dimensions were measured using a 0.01-g accuracy balance and a 0.01-mm accuracy digital caliper. Shrinkage samples were placed in an oven at a temperature of 103 ± 2 °C for 24 h, and swelling samples were submerged in distilled water for 20 days. A block was put on the sample's top to ensure complete submersion and saturation. Every day the specimens were checked, and more water was added to the dishes to compensate for evaporation. The dishes were big enough to have enough space for free swelling. Subsequently, samples were left to dry at room temperature for 3 days to avoid cracking and then placed in the oven at a temperature of 103 ± 2 °C for 24 h. After each phase, the weight and dimensions of the samples were measured.

Properties calculation

The density, equilibrium moisture content, shrinkage coefficient, swelling coefficients, anti-swelling efficiency (ASWE), and anti-shrinkage efficiency (ASHE) were calculated using Eqs. 1 through 6. The ASWE is the difference between the swelling of the modified and unmodified wood, and the ASHE is the difference between the shrinkage of the modified and unmodified wood.

$$D = \frac{m}{v} \tag{1}$$

$$EMC = \frac{w_{12} - w_0}{w_0} \times 100 \tag{2}$$

$$SH = \frac{V_w - V_o}{V_o} \tag{3}$$

$$S = \frac{V_w - V_o}{V_o} \times 100 \tag{4}$$

$$ASWE = \frac{S_u - S_m}{S_u} \times 100 \tag{5}$$

$$ASHE = \frac{SH_u - SH_m}{SH_u} \times 100 \tag{6}$$

In Eqs. 1 through 6, D is density (g/cm^2), m and v are dry weight (g) and dry volume (mm^3), respectively; W_{12} is the weight (g) at a temperature of 20 °C and relative humidity of 65%; W_o is oven-dried weight (g); SH is shrinkage (mm); V_w the volume (mm^3) after immersion; V_0 is dry volume (mm^3); S is swelling (mm); u is unmodified, and m is thermally modified. EMC of 1 inch cubes was calculated in 20 days of conditioning when samples reached constant weight. EMC is typically assumed to have been reached when the rate of MC change with time (dM⁄dt) drops below a certain value (Glass *et al.* 2018).

Anisotropy coefficient is the difference in swelling between tangential and radial direction, and water absorption repellency is the difference in water absorption between unmodified and TMW, calculated by Eqs. 7 and 8,

$$AC = \frac{(T_w - T_o)/T_o}{(R_w - R_o)/R_o} \tag{7}$$

$$WAR = \frac{WA_u - WA_m}{WA_u} \times 100 \tag{8}$$

where AC is anisotropy coefficient; T is Tangential (mm); R is Radial (mm); W is wet; O is overdried; WAR is the water absorption repellent; WA is water absorption; u is unmodified; and m is modified.

Statistical Analysis

The statistical analysis (using Jump Pro 16, SAS Company, Cary, NC, USA) was performed according to a completely randomized design and the significance level α = 0.05. The effect of the treatments was checked by the analysis of variance (ANOVA, F-test). The null hypothesis was that the thermal modification had no effect. In cases of statistically significant difference between means (F-test, $p < 0.05$). If needed, the Tukey multiple range test was used to determine whether the differences in means were significantly different. Finally, the data were graphed using MS-Excel (Microsoft Corp., Redmond, WA, USA).

RESULTS AND DISCUSSION

Water Absorption and EMC

The water absorption in 20 days of immersion and EMC values at 20 °C and 65% RH are presented in Table 2 and Fig. 1. The TM significantly (P value= < 0.05) increased water absorption repellent and decreased the EMC of all the species. The YP (29.6%) showed a maximum followed by red oak (22.5%), ash (21.9%), red maple (21.8%), hickory (20.7%), and cherry showed a minimum (14.9%) moisture absorption repellent (MAR). The moisture absorption of wood is through the available hygroscopic site (*i.e.*, hydroxyl groups) of the cellulose, hemicellulose, and lignin contained in the cell wall. The decrease in moisture absorption and EMC or in other words, moisture holding capacity of wood, by TM is explained by the degradation of the hydroxyl content in hemicelluloses and cellulose, which are sites to hold water (Esteves and Pereira 2009; Hill *et al.* 2021). Moreover, the changes in lignin, *i.e.*, self-condensation, polycondensation, and cross-linking with carbohydrates, which seals the cellulose microfibrils, precluding water absorption (Bekhta and Niemz 2003; Hill *et al.* 2021). The maximum reduction in EMC was for red maple, 11.7 to 4.8 and the less reduction was for cherry, 5.8 to 4.1 compared to unmodified and TMW, respectively. The authors' results are consistent with previous studies with the same TM species and other TM species. Ling *et al.* (2016) reported that TM decreased the EMC in *Populus cathayaha* from 7.83% (unmodified) to 4.14% in TMW (220 °C). The large decrease in moisture absorption and EMC between TM and the control clearly demonstrated that thermo-vacuum modification has great influence on degrading hygroscopic sites. In addition, thermo-vacuum treatment in an open vessel system ejects the cell wall residuals during the process, thus reducing the mass of wood more significantly than closed systems. This is as reported by Juizo *et al.* (2018), who modified *Eucalyptus* in a closed system at 180 to 240 °C with a statistically insignificant reduction in mass loss.

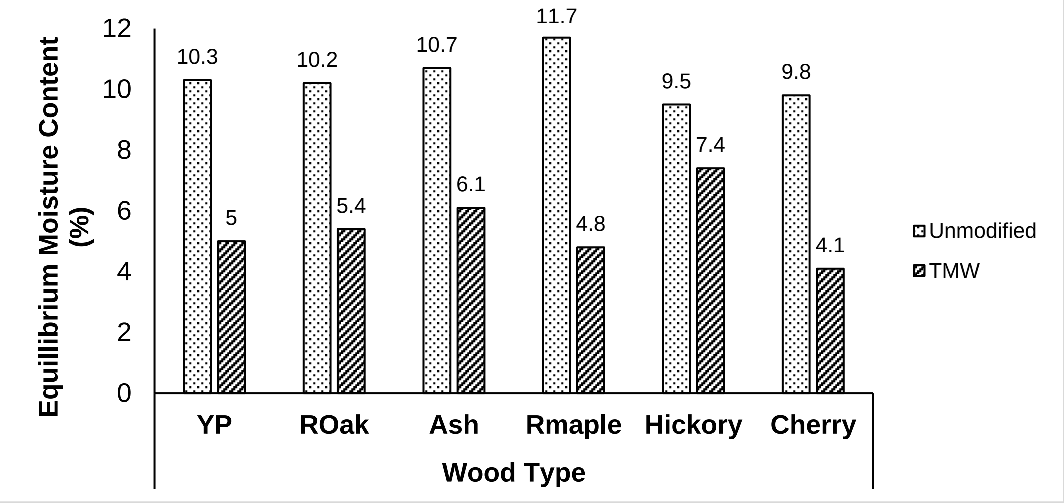

Fig. 1. Equilibrium moisture content of unmodified and TMW

Swelling

The data of radial, tangential, and volumetric swelling coefficient (SC) are shown in Fig. 2. Swelling of TMW for all species were significantly lower (P-value = <0.05) than that of unmodified wood. Compared to the swelling of the unmodified sample, the radial, tangential, and volumetric swelling decreased in all the species. The swelling properties of TMW can also be evaluated using the ASE parameter. The ASE indicates the low potential of TMW to swell when exposed to moisture. Based on the results presented in Table 2, ASE is 39.9 (YP), 18.2 (red oak), 71.4 (ash), 30.3 (red maple), hickory (14.2), and 36.4 (Cherry), respectively. These results are consistent with the findings of Cermak *et al.* (2015). The cited authors reported that TM *Pinus sylvestris* L. had an increased ASWE of 42.7%. The present results also were consistent with the results of Molinski *et al.* (2010), reporting the reduced swelling in ash. The reduction in swelling of TMW is widely reported in previous studies (Menezes *et al.* 2014; Hill *et al.* 2021). The modification by a thermo-vacuum system has a significant effect on the reduction of swelling in all species. Wood swelling is unique to wood species, wood direction, treatment schedule; hardwoods, particularly have more anisotropic structure and have less dimensional stability in the tangential direction than in the radial in swelling (Hill *et al.* 2021). The reduction in swelling is directly related to reduced water absorption rate, as explained in the moisture absorption section, which is mainly due to the decline in swelling of TMW, and is also due to reduction in the amorphous regions of cellulose, adding to crystallinity of cellulose and making it hydrophobic. Moreover, crosslinking in lignin through polycondensation reactions is another reason for reduced swelling. It is believed that cross-linkage between lignin and polymers occurs in thermal modification and is a beneficial change contributing for the decrease in the hygroscopicity and increasing the dimensional stability (Pieria 2020).

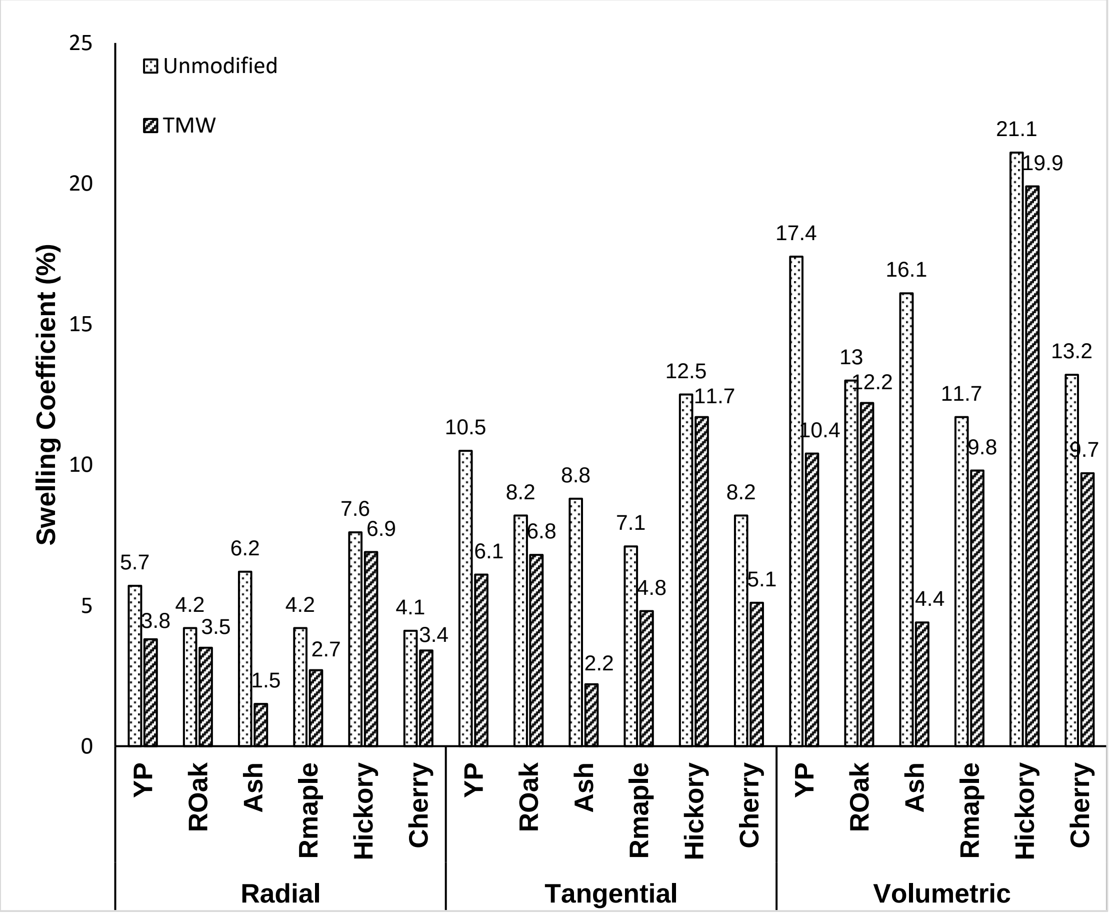

Fig. 2. Radial, tangential, and volumetric swelling coefficient of unmodified and TMW

Table 2. Data of Anisotropy Coefficient, Anti-Swelling Efficient, Water Absorption Repellent

Wood Type	YP		Red Oak		Ash		Red Maple		Hickory		Cherry	
	C	TM	C	TM	C	TM	C	TM	C	TM	C	TM
Anisotropy Coefficient	1.8	1.6	2.1	2	1.5	1.4	1.8	1.7	1.7	1.6	2.2	1.5
Water Absorption Repellent (%)	29.6		22.48		21.9		21.76		20.7		14.94	
Anti Swelling Efficient (%)	39.9		18.7		71.4		30.3		14.2		36.4	
Anti Shrinkage Efficient (%)	41.6		40.9		65.6		23.5		34.2		51.2	

YP: Yellow poplar; C: control; TM: Thermally modified

Shrinkage

Shrinkage coefficients (SHC) of the samples are shown in Fig. 3, and anti-shrinkage coefficient in Table 2. All species showed significantly lower SHC (P value = < 0.0001) than unmodified wood. Anti-shrinkage efficiency is also a good approach to find the difference of shrinkage between unmodified and TMW.

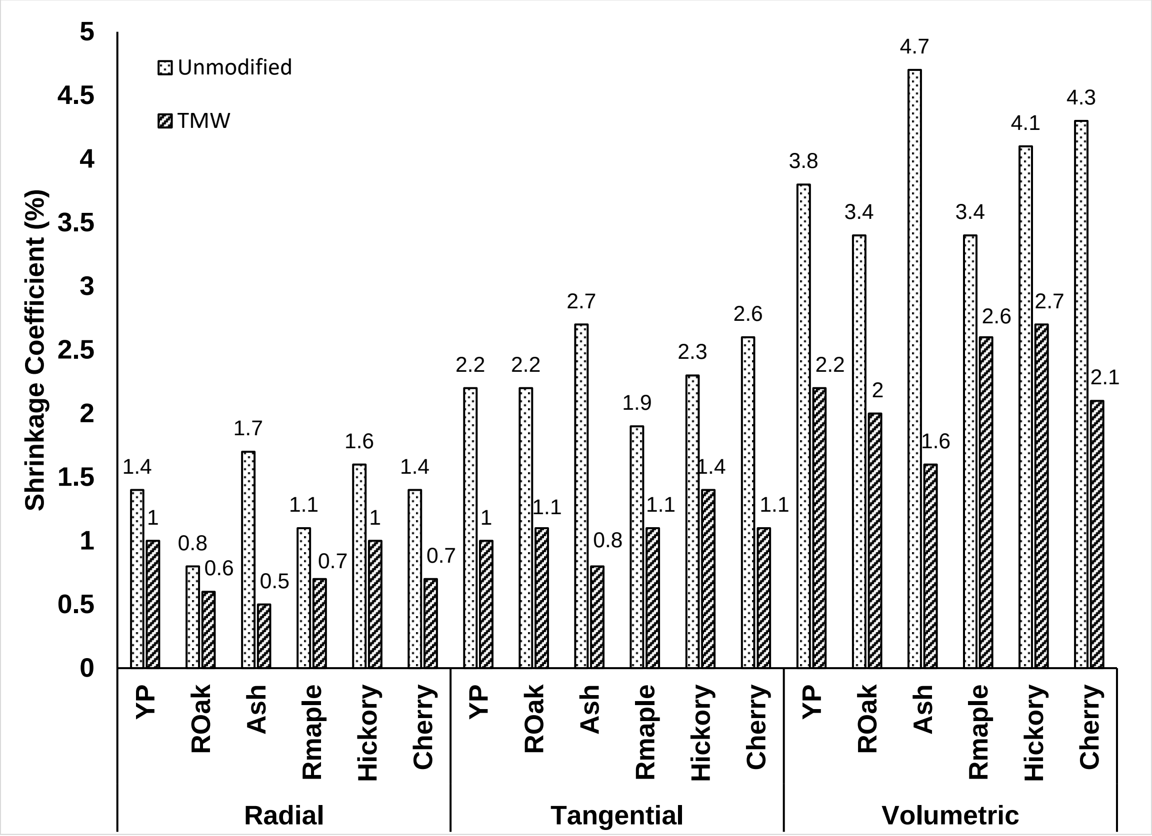

Fig. 3. Radial, tangential, and volumetric shrinkage coefficient of unmodified and TMW

Based on the results of Table 2, the data shows the ASHE of 41.6 for YP, 40.9 for red oak, 65.6 for ash, 23.5 for red maple, 34.2 for hickory and 51.2 for cherry. This result is in accordance with the findings of Cao *et al.* (2023), in which they modified *Eucalyptus* wood at different temperatures (150 to 210 °C) and reported that thermal modification at elevated temperature increases the ASHE. The shrinkage and swelling values of wood increase with higher density because of having thicker cell walls (Tsoumis 1991; Ling *et al.* 2016). The reduction in shrinkage could be attributed to the properties of TMW wood such as lower density and thinner cell wall (Todaro *et al.* 2018). Based on the mechanism of open vessel thermo-vacuum treatment that removes the residual of cell wall in modifying process, the residuals will not build up in lumen or on cell walls. The results is a reduction in density of modified wood, a reduction in the thickness of the cell walls, and lowered shrinkage.

Anisotropy

Based on the results presented in Table 2, TM reduced the anisotropy in all the samples. The anisotropy coefficient was reduced from 1.8 to 1.6 in YP, 2.1 to 2 in red oak, 1.5 to 1.4 in ash, 1.8 to 1.7 in red maple, 1.7 to 1.6 in hickory, and 2.2 to 1.5 in cherry. Previous studies also reported the reduction in anisotropy in TMW. Kurul and Gorgun (2022) modified YP at 180 °C for 3 hours and observed that TM reduces anisotropy, swelling and shrinkage. Hermida *et al.* (2022) modified *Dinizia excelsa* Ducke wood at different temperatures (180 to 215 °C) and reported that the anisotropy of TMW ranged from 1.43 to 1.59. Menezes *et al.* (2014) modified (140, 160, and 180 ºC) *Corymbia citriodora* and *Eucalyptus saligna* woods. They observed that TM reduced the anisotropy coefficient of both species, particularly, with the most expressive values being observed in the higher temperatures. This is close to the schedule of the present study. Consequently, the result is as observed in the findings of this study. In TM, the cell wall undergoes severe degradation, extractives disappear, the amorphous region of cellulose degrades, and its crystallinity increases (Todaro *et al.* 2018), which is possibly the main cause of reduction in anisotropy.

CONCLUSIONS

1. Thermal modification in an open vessel thermo-vacuum system decreased water absorption in all the hardwood species tested.
2. Swelling of all the species significantly decreased, ranging from a 14.2 to 71.4% increase in anti-swelling efficiency.
3. Shrinkage of all the species significantly decreased, ranging from a 23.5 to 65.5% increase in anti-shrinkage efficiency.
4. Anisotropic behavior of all the species decreased due to thermal modification.
5. As a result of reduced water absorption that leads to reduced swelling and shrinkage, dimensional stability of thermally modified wood (TMW) improved in all the hardwood species. The improved dimensional stability is a key property in design of wood products for better utilization in different types of applications and this level of improvement in TMW properties would keep the TM as a green and efficient method for sustainable wood modification.

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